



16th International Symposium on Essential Oils
16. Internationale Arbeitstagung über Ätherische Öle

Holzminden/Neuhaus (Germany)
September 18.-21., 1985



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Scientific Committee: A. Baerheim Svendsen (Leiden), E.-J. Brunke (Holzminden)
R. Hopp (Holzminden, K.-H. Kubeczka (Würzburg)

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Hammerschmidt, Dr. C.-H. Kappey, Dr. F.-H. Köster,
Dr. L. Tumbrink (all Holzminden)

C o n t e n t s

- A. Programme
- B. Abstracts of the Lectures
- C. Abstracts of the Posters
- D. List of Participants

P r o g r a m m e

Tuesday, September 17th, 1985

17.00 h - Arrival and check-in (Haus des Gastes, 1. floor, Symposium office)

18.00 h - Get together party
(Haus des Gastes)

-22.00 h

Wednesday, September 18th, 1985

- 9.00 h - Opening Session in Honour to Otto Wallach
(Lecture Hall, Chairman: E.-J. Brunke)
- F. Bohlmann (Techn. Univ. Berlin, Germany)
New Types of Sesquiterpenes
- 10.15 h - Coffee break
- 10.45 h - 2. Morning Session (Lecture Hall, Chairman: R. Hopp)
- I. Klimes and D. Lamparsky: (Givaudan AG, Switzerland)
Analytical results concerning the essential oil of *Artemisia pallens* (Wall.)
- K.-H. Kubeczka (Univ. Würzburg, Germany)
New Constituents from the Essential oils of *Pimpinella* species
- P. Weyerstahl (Techn. Univ. Berlin, Germany)
Isolation and Synthesis of Compounds from the Essential oil of *Helichrysum italicum*.
- M. Köpsel (Haarmann & Reimer, Holzminden, Germany)
An unusual compound isolated from *Mentha piperita* oil.
- 12.30 h - Lunch break
- 14.00 h - 1. Afternoon session (Lecture Hall, Chairman: K.-H. Kubeczka)
- H. Wolf (Techn. Univ. Braunschweig, Germany)
Sesquiterpene syntheses via π -Cyclization
- E.-J. Brunke and L. Tumbrink (DRAGOCO GmbH, Holzminden, Germany)
First Total Synthesis of Spiro-santalol, a Constituent of East Indian Sandalwood oil
- G. Buchbauer and H. Spreitzer (Univ. Wien, Austria)
Synthesis of Fragrance Compounds within the Bicyclo[2.2.2]octane Series
- 16.00 h - Coffee break
- 16.30 h - 2. Afternoon session (Lecture Hall, Chairman: P. Weyerstahl)
- H.M.R. Hoffmann (Univ. Hannover, Germany)
Synthesis of Tetramethylimonene Derivatives
- K. Kieslich (Ges. f. Biotechnologische Forschung, Braunschweig, Germany)
- 18.00 h Microbial Transformations of Terpenoids

Thursday, September 19th, 1985

- 8.30 h - 1. Morning Session (Lecture Hall, Chairman: D. Lamparsky)
- F.B. Whitfield and J.H. Last (CSIRO, Australia)
The Flavour of Passionfruit (A Review)
 - F.P. van Lier, L.M. van der Linde and A.J.A. van der Weerd
(Naarden International, Netherlands)
The isolation and synthesis of the character impact compound in
Calamus oil
 - P. Werkhoff and R. Hopp (Haarmann & Reimer, Holzminden, Germany)
Isolation and Gaschromatographic Separation of Menthol and Menthone
Enantiomers from Natural Peppermint Oils
- 10.30 h - Coffee break
- 11.00 h - 2. Morning Session (Lecture Hall, Chairman: F. Bohlmann)
- D. Joulain (Robertet et Cie, France)
Study of the Fragrance given off by certain Springtime Flowers
 - R. Kaiser (Givaudan AG, Switzerland)
New natural products of structural and olfactory interest identified
in "Fig leaf absolute" (*Ficus carica*)
 - T.H. Moxham (Univ. of Bath, Great Britain)
The commercial exploitation of lichens for the perfume industry
- 12.30 h - Lunch break
- 14.00 h - Poster Session
(Entrance Hall, 1. Floor)
- 15.30 h - Departure to Bevern
- 16.00 h - (Castle of Bevern) Chamber Concerto
- 17.00 h - Departure to Corvey
- 17.15 h - Guided Visit to Castle and Monastery Corvey
- 19.00 h - (Schloßrestaurant Corvey) Symposium Dinner
- 23.00 h - Departure to Neuhaus

Friday, September 20th, 1985

- 8.30 h - 1. Morning Session (Lecture Hall, Chairman: W. Bruhn)
- W. Herres (Bruker GmbH, Karlsruhe, Germany) and K.-H. Kubeczka (Univ. Würzburg, Germany)
HRGC-FTIR-Investigations on volatile Terpenes
- S. Ebel, W. Garbade, W. Mück and A. Werner-Busse (Univ. Würzburg, Ger.)
Selective Detection in HPLC-Analysis of Essential Oils
- W. Günther, F. Schlegelmilch and J. Wohland (WGA, Düsseldorf, Germany)
The Calculation of Column Polarities
- 10.30 h - Coffee break
- 11.00 h - 2. Morning Session (Lecture Hall, Chairman: F.B. Whitfield)
- U. Kobold, O. Vostrowski and H.-J. Bestmann (Univ. Erlangen-Nürnberg, Germany)
Reaction Gas Chromatography in the Structure Elucidation of Terpenoids. Insecticides of Plant Origin in *C. Balsamita*
- G. Lange (Univ. Würzburg, Germany)
The Use of High Resolution Mass Spectrometry in GC/MS-coupling for Analysing Complex Mixtures of Volatiles
- W. Schultze, G. Lange and G. Heinrich (Univ. Würzburg, Germany)
Direct analysis of Plant Material by Mass Spectrometry
- 12.30 h - Lunch break
- 14.00 h - 1. Afternoon Session (Lecture Hall, Chairman: A. Baerheim-Svendsen)
- O. Ekundayo (Univ. Ibadan, Nigeria)
Essential Oil Composition of Some Nigerian Medicinal Plants
- M.H. Boelens and R.J. Sindren (Bordas SA, Sevilla, Spain):
The chemical composition of Laurel Leaf oil (*Laurus nobilis* L.) obtained by Steam Distillation and Hydrodiffusion
- E.H. Graven (Univ. Fort Hare, South Africa)
Native Southern African Aromatic Plants - a New Vehicle for Rural Development
- 16.00 h - Coffee break
- 16.30 h - 2. Afternoon Session (Lecture Hall, Chairman: J.J.C. Scheffer)
- C. Zdero (Techn. Univ. Berlin, Germany)
New Terpenoids from Mexican *Stevia* species

- 17⁰⁰ - H.L. de Pooter and N.M. Schamp (Univ. Gent, Belgium)
Comparison of the Volatiles of some *Satureja* and *Calamintha* species
- 17³⁰ - E. Stahl (Univ. Hamburg, Germany)
Chemical Composition and Variation of the Essential Oil from the Norwegian *Thymus praecox ssp. arcticus* and *Thymus pulegioides*
- 18⁰⁰ - E.-J. Brunke and F.-J. Hammerschmidt (DRAGOCO GmbH, Holzminden, Germany)
Volatile Constituents of *Achillea wilhelmsii* C. Koch (Syn. *A. santolina* auct. mult.) with different regional origin
- 18.30 h

Saturday, September 21st, 1985

- 8.30 h - 1. Morning Session (Lecture Hall, Chairman: E. Stahl)
- A.M. Bosabalidis and T. Tsekos (Univ. Thessaloniki, Greece)
Ultrastructure of the essential oil secretion in the oil glands of the fruit peel of Mandarin (*Citrus deliciosa* Ten.)
 - K.H. Kubeczka (Univ Würzburg, Germany)
Morphology and Chemistry of the essential oil secretion of *Dictamnus albus*
 - J. Reichling, H. Becker, R. Martin, G. Burckhardt (Univ. Heidelberg, Germany)
Comparative Study on the Production and Accumulation of Essential Oil in the Whole Plant and in the Cell-culture of *Pimpinella anisum*
- 10.30 h - Coffee break
- 11.00 h - 2. Morning Session (Lecture Hall, Chairman: H. Schilcher)
- A.M. Janssen, J.J.C. Scheffer and A. Baerheim Svendsen (Univ. Leiden, Netherlands)
Antimicrobial screening of essential oils
 - K. Knobloch, H. Weigand and N. Weis (Univ. Erlangen-Nürnberg, Germany)
On Biochemical Actions of Terpenoids
- 12.00 h - Final discussion and closing session
- 12.30 h

Poster Presentation

- P1 E.A. Aboutabl, A.A. El-Azzouny and F.-J. Hammerschmidt
The Essential Oil of *Nigella sativa* L. Seeds
- P2 R. Bos, H. Hendriks and C. Mastenbroek
Variations in the Composition of the Essential Oil in Individual Plants of a Strain of *Valeriana officinalis* L. s.l.
- P3 M.M. Carmo and S. Frazao
The Essential Oil of Portuguese Pine Needles. First Results.
- P4 K. Grzunov, J. Mastelić and N. Ruzić
Isolation and Identification of Terpenylglucosides in the Leaves of Dalmatian *Salvia officinalis*
- P5 F.-J. Hammerschmidt, E.-J. Brunke and F.-H. Köster
Essential Oil of *Cedrela odorata* L. (Meliaceae) from Brazil - Revised List of Constituents
- P6 H.-P. Hanssen, A. Klingenberg, E. Sprecher
Screening for Volatile Terpenes in Yeasts
- P7 E. Héthelyi, B. Dános and P. Tétényi
GC/MS Analysis of Essential Oils of some *Tagetes* Species
- P8 T. Hirvi, I. Salovaara, H. Oksanen and E. Honkanen
Volatile Constituents of Coriander Fruit Isolated by Different Methods and Cultivated at Different Localities
- P9 R. Huopalahti
Stability at 3,6-Dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran in Aqueous Buffer Solutions
- P10 R. Ikan, B. Crammer, V. Weinstein, Z. Goldschmidt, and H. Spiegelstein
Synthesis of γ - and δ -Lactones and their Odorous and Plant-growth Regulating Activity
- P11 J. Karlsen
Studies on the Essential Oil of *Zanthoxylon armatum* of Nepalese Origin. A Suitable Oil for Industrial Production
- P12 F. Moattar, H. Sansam-Sheriat
Qualitative and Quantitative Determination of Essential Oils of *Eucalyptus* Species in Iran
- P13 O. Motl, J. Lenfeld, A. Trka
Anti-inflammatory Efficacy of Terpenes from *Conyza canadensis*
- P14 O. Motl, V.P. Papageorgiou, G. Ochir, N. Argyriadou and H. Dunkel
The Essential Oil of *Heracleum dissectum*
- P15 N. Ntezurubanza, J.J.C. Scheffer, A. Looman, and A. Baerheim Svendsen
Composition of the Essential Oil of *Ocimum canum* Grown in Rwanda
- P16 U. Ravid and E. Putievsky
Carvacrol and Thymol Chemotypes of Wild East Mediterranean Labiate Herbs.

- P17 E. Sarer, J.J.C. Scheffer, A. Looman, A. Baerheim Svendsen
The Essential Oils of Three *Origanum* Species grown in Turkey
- P18 J.J.C. Scheffer, R. Hiltunen, M. von Schantz and A. Baerheim Svendsen
Analysis of the Volatiles from Fruits of *Heracleum* Species by HSGC -
an Interlaboratory Study
- P19 F. Schlegelmilch, W. Günther, M. Lux
Adsorbents for the Dynamical Headspace
- P20 G. Schmaus, K.-H. Kubeczka, and V. Formacek
Structural Elucidation of an Irregular Sesquiterpene Alcohol from
Peucedanum palustre (L.) Moench
- P21 S. Schmidt, R. Bos
Volatiles of *Orthosiphon stamineus* Bent.
- P22 R. Schubert and H.J. Hübschmann
The Ion Trap Detector: The Techniques and it's Application
- P23 H.C. Srivastava and P.G. Karmakar
Germination of *Jasminum Grandiflorum* Linn. Seeds - Effect of Sunlight,
Shade and Darkness
- P24 H.C. Srivastava and P.G. Karmakar
Floral Biology of Jasmines
- P25 E. Stahl und V. Sinnwell
A Volatile Diterpenoid from *Ammi visnaga* (L.) Lam. Fruits
- P26 H. Vuorela, R. Hiltunen, J. Pohjola and I. Laakso
Application of Headspace Gas Chromatography in Essential Oil Analysis.
VII. Hydrodistillation Compared with Headspace
- P27 J. Wohland, W. Günther, F. Schlegelmilch
The Thermal Conductivity Detector in Capillary GC

New Types of Sesquiterpenes

F. Bohlmann
Department of Organic Chemistry
University of Berlin
Straße des 17. Juni 135
D-1000 Berlin 12

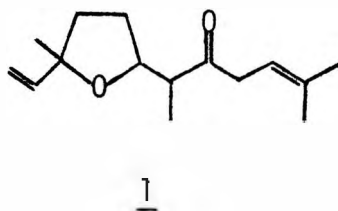
- Abstract is not available -

ANALYTICAL RESULTS CONCERNING THE ESSENTIAL OIL OF *Artemisia pallens* (Wall.)

I. Klimes and D. Lamparsky

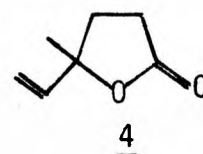
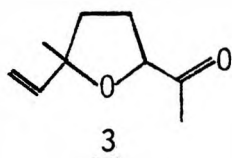
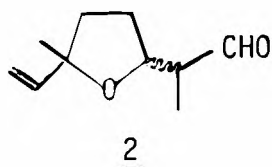
GIVAUDAN Research Comp. Ltd., CH-8600 Dübendorf-Zurich, Switzerland

We already discussed earlier in this year ("Topics in Flavour Research", Freising-Weihenstephan, April 1985) some analytical results of the oil concerning sesquiterpenic compounds related to the main component, davanone 1.



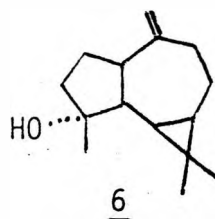
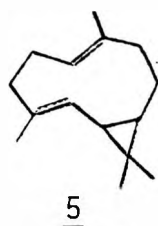
It is now interesting to see that even in the lower-boiling part of davana oil the same structural feature, a methyl-vinyl-substituted tetrahydrofuran ring so typical for the mentioned series of sesquiterpenoids, continues to play a prominent role within the minor constituents.

For instance, the occurrence of compounds 2 - 4 is somewhat indicative for oxidative degradation processes responsible for the formation of such substances from the starting nerolidyl skeleton in the plant.



Enzymatic reduction seems to be the next step furnishing the corresponding alcohols which may be dehydrated to further derivatives.

The group of sesquiterpene hydrocarbons comprises a great variety of skeletons with bicyclogermacrene 5 as the main representative. The presence of further constituents with a cyclopropane ring in their molecules, i.e. aromadendrene, β -maaliene or spathulenol 6 shows a second preference by enzymic systems in *Artemisia pallens* to transform the acyclic farnesene skeleton.



New constituents from the essential oils of Pimpinella species

K.-H.Kubeczka

Department of Pharmaceutical Biology, University of Würzburg,
Mittlerer Dallenbergweg 64, D-8700 Würzburg (FRG)

As part of systematic investigations of the essential oils from Apiaceae, especially of the genus Pimpinella, the volatile constituents from several Pimpinella species have been analysed. All the root oils investigated contain considerable amounts of phenylpropane and sesquiterpene derivatives, whereas monoterpenes occur only in trace amounts. The phenylpropanoids found show the unusual substitution pattern 2-hydroxy-5-methoxy, which has not been found elsewhere up till now.

In addition to these phenylpropanoids, all plants of the genus Pimpinella were shown to contain terpenoid C_{12} -hydrocarbons. In view of the extremely restricted occurrence of these compounds in the plant kingdom, especially within part of the family Apiaceae, the possible taxonomic value of these substances with regard to the definition of the genus Pimpinella will be discussed.

Isolation and Synthesis of Compounds from the Essential Oil of *Helichrysum italicum*

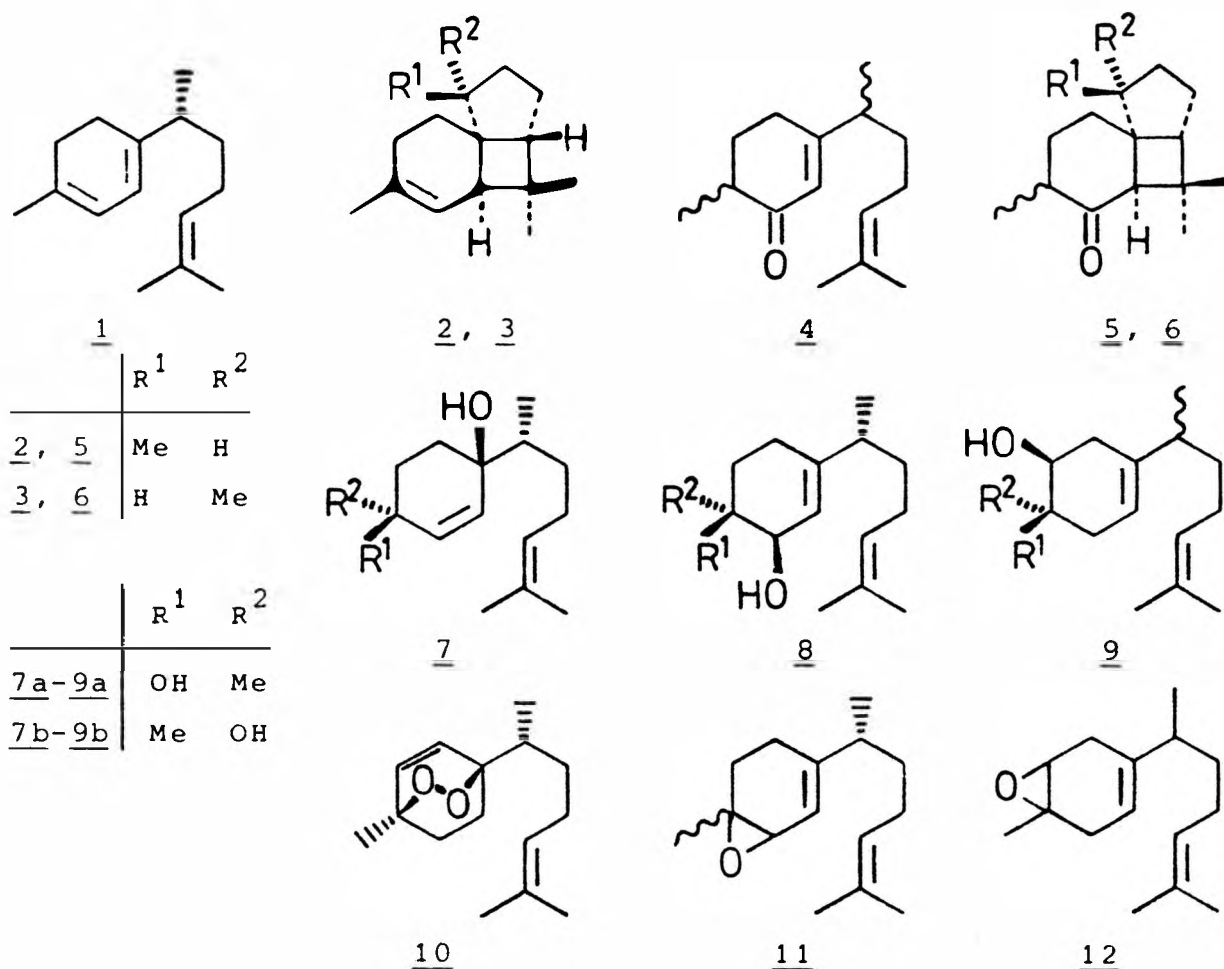
Peter Weyerstahl, Jürgen Leimner, Helga Marschall, Norbert Meier, Stefan Scholz and Marlies Weirauch

Institut für Organische Chemie, Technische Universität Berlin
 Straße des 17. Juni 135, D-1000 Berlin 12, West Germany

Helichrysum oil is widely used in perfumery. Our previous investigation of the sesquiterpene hydrocarbon fraction has shown γ -curcumene (1) to be the main compound. Two other sesquiterpenes with a novel skeleton, italicene (2) and isoitalicene (3) could be found in relatively high amount, and their structure has been elucidated¹⁾.

The synthesis of 2 and 3, formally [2+2]cycloaddition products of 1, was elaborated starting from *m*-bromoanisole via enone 4 and its photocyclization products 5 and 6.

From the polar sesquiterpene fraction some diols (7, 8) were isolated whose origin should also be γ -curcumene (1). While 7a was readily obtained from 1 via the endoperoxide 10, the selective epoxidation to give 11 is difficult. Therefore, extensive studies of the regioselectivity of the epoxidation of 1 as well as of β -curcumene to yield 12 were attempted. Hydrolysis of 11 resp. 12 under various conditions furnished the diols 7b, 8a,b, and 9a,b.



1) J. Leimner, H. Marschall, N. Meier and P. Weyerstahl, Chem. Lett. 1984, 1769.

An unusual compound isolated from Mentha piperita oil

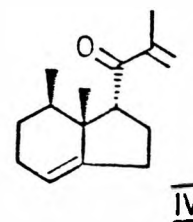
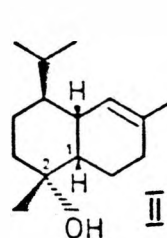
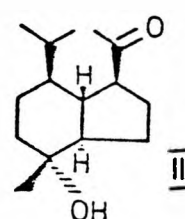
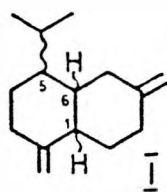
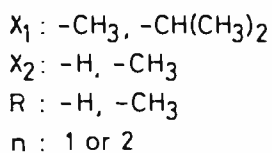
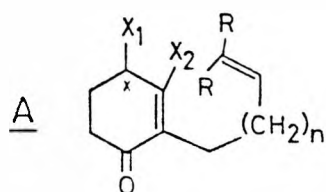
Manfred Köpsel, Alfred Krempel and Horst Surburg
Haarmann & Reimer GmbH
Research Laboratories
D-3450 Holzminden, Germany

In the course of our investigations on trace components of the essential oil of *Mentha piperita*, we isolated an unknown compound, the EI mass spectrum of which could not be interpreted. By means of CI and high resolution mass spectra, the molecular formula $C_{12}H_{20}O_3$ was determined. After isolation by preparative gas chromatography, the infrared and NMR spectra were recorded, from which an ortho-ester structure (I) was proposed. In order to verify this, compound (I) was synthesized starting from isopulegol which can be seen as the precursor of (I) in *Mentha piperita*. The spectra of the synthetic material and those of the natural one proved to be identical.

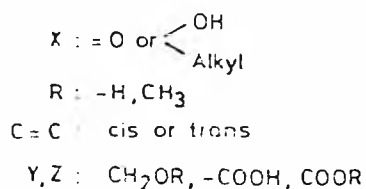
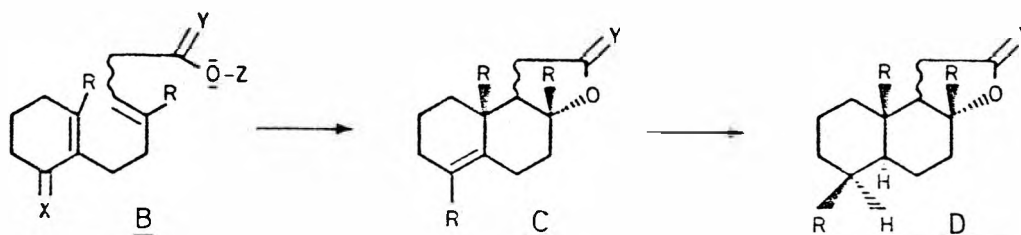
Sesquiterpene Syntheses via π -Cyclization

H. Wolf, Technische Universität Braunschweig.

1. Cyclohexenone derivatives of general formula A were regio- and stereoselectively cyclized (CH_3COOH / $(\text{CH}_3\text{CO})_2\text{O}$ / HClO_4) to the corresponding bicyclic enol acetates. These are suitable intermediates for the synthesis of several sesquiterpenes as ϵ -dienes of the cadalane series: (I), torreyol (II), oplopanone (III) and chiloscypnone (IV).



2. Allylic alcohols and some enones of general formula B with oxygen containing functional groups as "terminators" were stereoselectively cyclized to tricyclic ethers or lactones (C) with different alkyl substitution pattern. The saturated compounds D represent analogs of some ambergris components.



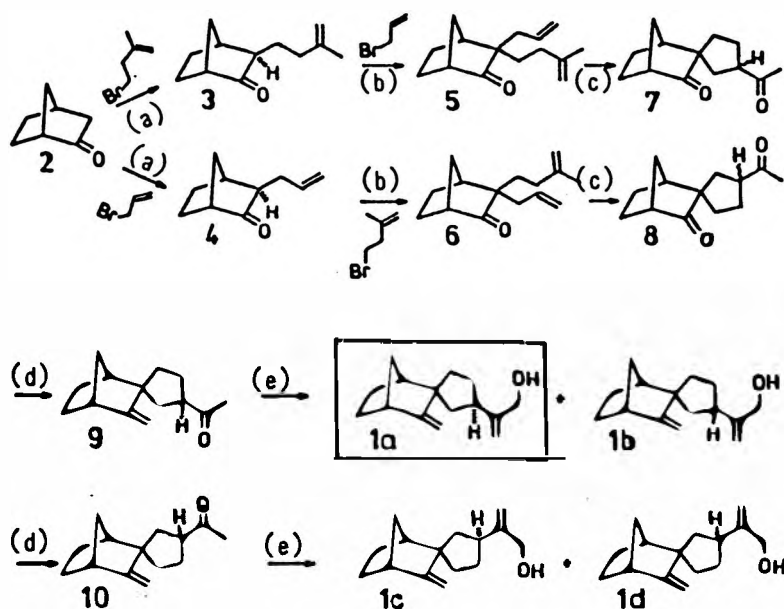
First Total Synthesis of Spirosantalol, a Sesquiterpene Alcohol from East Indian Sandalwood Oil

Ernst-Joachim Brunke and Ludwig Tumbrink
DRAGOCO, Research Laboratories
D-3450 Holzminden, West Germany

During the analytical work on the constituents of East Indian Sandalwood Oil, which has been performed in our working group five years ago, the Spirosantalol (1a) has been isolated for the first time. Its structure has been elucidated by spectroscopical methods. For the demonstration of the constitution of this new sesquiterpene skeleton, the total synthesis has been performed.

The synthesis started with norbornanone and resulted stereoselectively (see scheme) in the pure spirosantalols 1a, 1b, 1c and 1d, respectively.

The relative configuration of the epimers 1a/1b and 1c/1d, respectively, resulted from ^1H - and ^{13}C -NMR-spectroscopy. The structure of the natural spiro-santalol has been determined as 1a by comparison with the synthesized isomers 1a-d.



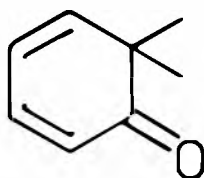
- a) NaNH_2 b) LDA c) $\text{O}_3/(\text{CH}_2)_2\text{S}$; NaOH; $\text{H}_2/\text{Raney-Ni}$
d) $(\text{CH}_3\text{O})_3\text{CH}$; MeLi; SOCl_2/Pyr . e) $(\text{CH}_3)_2\text{S}=\text{CH}_2$; LDA

Synthesis of Fragrance Compounds within the Bicyclo [2.2.2]octane Series

by Gerhard Buchbauer and Helmut Spreitzer

Institute of Pharmaceutical Chemistry, University of Vienna,
A-1090 Vienna, Währingerstrasse 10, Austria

6,6-Dimethylcyclohexadienone (1), easily obtained by standard procedures, serves as a key substance for the preparation of 3,3-disubstituted bicyclo [2.2.2]octane derivatives. Such "homo norbornane" compounds are of interest not only for fragrance chemists but also for pharmaceutical chemists studying structure-activity-relationships. That was the reason why we were in need of an easy and economic access to homocamphenilone, the "bridge-homologue" of the natural monoterpene ketone camphenilone. 1 not only furnishes this homologated ketone in 3 steps with good yields but serves also as the starting material for a 7-step synthesis of a new analogue of norpatchoulenol which exerts a weak but characteristic odour of patchouli.



1

Synthesis of Tetramethyl Monoterpenes

H.M.R.Hoffmann,
Department of Organic Chemistry,
University of Hannover, Schneiderberg 1 B
D-3000 Hannover

3,3,5,5-Tetramethyl-limonene has been prepared biomimetically from inexpensive mesityl oxide.^{1,2} Regioselective and stereoselective functionalization of the various carbon atoms yields inter al. the tetramethylated analogues of carvone, the cis and trans carveols,² α - and β -terpineols, sobrerol, perilla alcohol, Δ^1 -p-menthene and $\Delta^{1,7}, \Delta^8$ -menthadiene.³⁻⁶

¹ H.M.R.Hoffmann, H.Vathke-Ernst, Chem.Ber. 114 (1981) 1182.

² R.J.Giguere, G.von Ilseman, H.M.R.Hoffmann, J.Org.Chem. 47 (1982) 4948.

³ H.M.R.Hoffmann, D.Pauluth, Liebigs Ann.Chem. 1985, 396.

⁴ D.Pauluth, H.M.R.Hoffmann, ibid., 1985, 403.

⁵ D.Pauluth, H.M.R.Hoffmann, ibid., 1985, 756.

⁶ H.M.R.Hoffmann, A.Köver, D.Pauluth, J.C.S.Chem.Comm. 1985, 812.

MICROBIAL TRANSFORMATIONS OF TERPENOIDS

K. Kieslich, W.R. Abraham, B. Stumpf, P. Washausen

Modification of terpenoids for the preparation of new fragrances and flavours or for their chemical intermediates requires reactions with high regio-, stereo- and enantioselectivity. Biotransformations are a valuable support of the chemical synthesis or variation of these natural products or chemical prepared synthones.

The present state in this field is reviewed by examples of biotransformations of citronellal, patchoulol, ionones and ketoisophorone a.o. with different degree for practical application. Newer results of own investigations of substrates like limonene and similar monocyclic monoterpenoids, several acyclic structures and sesquiterpenoids like α -cedrene, cedrol, longipinene, β -caryophyllene, α -humulene and humulene epoxides are described.

The Flavour of Passionfruit - a Review

Frank B. Whitfield and John H. Last
CSIRO, Division of Food Research,
P.O. Box 52,
North Ryde, N.S.W. 2113, Australia.

Abstract

In recent years there has been renewed interest in the flavour of the passionfruit (genera Passiflora); species of which were first investigated in some detail in 1972. These new studies have led to the identification, for the first time, of a number of compounds believed to be important in the flavour of these fruits. A total of 287 volatile compounds have now been identified in the two main commercial varieties, the purple passionfruit P. edulis (Sims) and the yellow fruit P. edulis f. flavicarpa (Degener), and their hybrid cultivars. Other studies have led to a better understanding of the biosynthesis of several key esters, and have demonstrated the role of non-volatile precursor compounds in the production of the monoterpenoids and possible production of the C₁₃ norterpenoids.

In addition to reviewing the chemistry of passionfruit flavour, the paper will outline the variety of passionfruit raw materials available for study, the techniques used for the isolation of volatile compounds and the effects that these variables have on the composition of volatile extracts. Furthermore, as a result of the identification of new compounds and a newly gained knowledge of the formation of important flavour components, areas for future research including plant breeding, horticultural aspects and juice processing will be discussed.

The Isolation and Synthesis of the Character Impact Compound in
Calamus Oil

F.P. van Lier, L.M. van der Linde and A.J.A. van der Weerd
Naarden International, Organic Research Department
P.O. Box 2, NL-1400 CA Bussum

- Abstract is not available -

ISOLATION AND GAS CHROMATOGRAPHIC SEPARATION OF MENTHOL
AND MENTHONE ENANTIOMERS FROM NATURAL PEPPERMINT OILS

P. Werkhoff and R. Hopp

Haarmann + Reimer GmbH, Holzminden, FRG

The four diastereomeric "menthols", neomenthol, menthol, isomenthol, and neoisomenthol and the two diastereomeric "menthones", menthone and isomenthone have been isolated from natural peppermint oils (*mentha arvensis* and *mentha piperita*) by preparative gas chromatography just as by preparative-scale high performance liquid chromatography. The four menthols were derivatized by reaction with isopropyl isocyanate to form the corresponding isopropyl urethanes and the chiral ketones were converted into their oxime derivatives. Subsequently gas chromatographic enantiomer separation of monoterpene alcohols and monoterpene ketones on fused silica capillary columns coated with XE-60-S-valine-S- α -phenylethylamide was performed. Configurations of menthol and menthone enantiomers were assigned by optically pure reference substances.

Another alternative to this approach is complexation gas chromatography. For this reason complexation gas chromatography with Ni(II)-bis-[3-heptafluorobutyryl-(1R)-camphorate] \leftarrow as a chiral stationary phase was additionally used to resolve racemic menthols and menthones quantitatively into their enantiomers without derivatization.

From both methods we can draw the conclusion that the biogenesis of menthol and menthone isomers proceeds obviously strictly enantioselective.

The determination of enantiomeric excess ee showed ee-values $\geq 99\%$.

Bearing in mind the profound impact that stereochemical factors can have on biological activity or sensory properties, the separation of diastereomers and enantiomers will increase and become more and more important.

STUDY OF THE FRAGRANCE GIVEN OFF BY CERTAIN SPRINGTIME FLOWERS

Daniel Joulain
Research Laboratories, P. Robertet et Cie,
B.P 100, F-06333 Grasse-Cedex

Abstract : For several years now, the systematic head-space analysis of natural products from plant origin has become a routine technique in order to gain knowledge of the composition of their odoriferous emissions. These natural products may be for food use (fruits, beverages etc...), or in the case of flowers, may be raw materials for the perfumery industry.

Some flowers which bloom during springtime are processed in the industry as hexanic extracts, further treated with ethanol to yield "absolutes" : *Spartium junceum* L. (broom), *Narcissus poeticus* L. (narcissus), and to a lesser extent *Lonicera caprifolium* L. (honeysuckle).

In the Grasse region, a certain number of plants - including trees - produce during springtime very fragrant flowers which cannot be used in the industry for essentially economical reasons, although some of them have been used for a long time before being abandoned : *Syringa vulgaris* L. (lilac), *Pittosporum tobira* Ait., *Robinia pseudoacacia* L. (false acacia), *Wistaria sinensis* D.C., *Lilium candidum* L. (white lily), *Olea europaea* L. (olive tree), *Philadelphus coronarius* L. (seringa), *Coronilla emerus* L.

The head-spaces from these flowers have been separated within 30 minutes after they have been collected, using a rapid technique consisting of cryogenic trapping under vacuum. In each case, 1 to 10 ppm of a mixture of volatiles was obtained, and then analyzed by GC/MS.

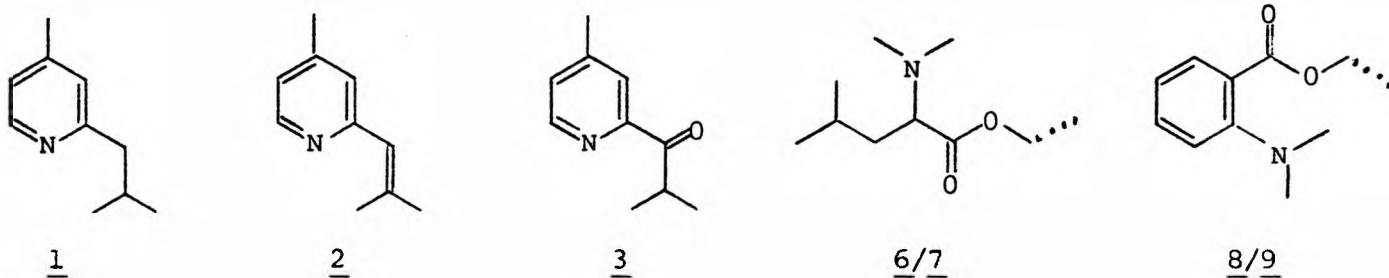
A number of previously unreported substances have been positively identified in these flowers ; some of them may be considered as new natural products.

NEW NATURAL PRODUCTS OF STRUCTURAL AND OLFACTORY
INTEREST IDENTIFIED IN FIG LEAF ABSOLUTE
(*Ficus carica* L.)

R. Kaiser
GIVAUDAN Research Company Ltd.
CH-8600 Dübendorf - Zürich

SUMMARY

In 1980, Fig Leaf Absolute was reported by "RIFM" to have a potential for inducing sensitization as well as phototoxic reactions in human skin. This prompted us to investigate the chemical composition and olfactory concept of this comparatively rare perfume material used for decades as speciality in fine fragrances. Among 200 constituents identified, a series of structurally and olfactorily interesting trace constituents not yet known as natural products attracted special attention.



Discussed in more detail are the 2,4-disubstituted pyridines 1-3, the N,N-dimethylated leucine esters 6 and 7 as well as the N,N-dimethylated anthranilates 8 and 9. The pyridines 1-3 could also be identified in other essential oils or absolutes, respectively.

The Commercial Exploitation of Lichens in the Perfume Industry.

T. H. MOXHAM,

DEPARTMENT OF PLANT SCIENCES, SCHOOL OF BIOLOGICAL SCIENCES
UNIVERSITY OF BATH, CLAVERTON DOWN, BATH, AVON, BA2 7AY, UK.

The lichens Evernia prunastri (oakmoss) and Pseudevernia furfuracea (treemoss) are gathered from forests in Mediterranean areas (and also North India) for use in the manufacture of perfumes and cosmetics. These lichens do also grow in temperate regions where their growth is much slower, but where they have been shown to be susceptible to atmospheric pollution, which retards their growth even further. In the forests where they are collected commercially these lichens regenerate very quickly and are reharvestable after one to five years depending on the areas where they are harvested. These forests, at present, do not appear to be affected by industrial pollution, and if the collection of lichens is 'managed' as it is now, supplies of the lichen should be able to be maintained in the foreseeable future.

Oakmoss is collected from the trunks of the trees, but treemoss is mainly gathered from twigs and branches where it hangs in clusters. Although distinction is made between 'cedarmoss' - collected in Morocco - and 'treemoss' - gathered mainly from pines in Southern France - the species of lichen is the same, viz. Pseudevernia furfuracea, and the differences in fragrance properties are most probably directly attributable to the wood component. In a random sample of treemoss ready for processing, the lichen was stripped from the twigs and the proportion by weight was found to be 44% lichen, : 56% wood, however, an average wood component would probably be between 35 - 55% by weight. Commercial oakmoss has a much purer lichen component, although there are usually several other species of fruticose lichens as well as some small quantities of foliose lichens.

The lichens, which are gathered dry, are packed into large jute sacks and are taken by lorry to the baling plant where they are prepared for shipment to the processing factories. The bales will store well so that the lichens can be processed during periods when there is no seasonally available material.

Finally, passing mention is made of the other commercial uses of lichens.

HRGC-FTIR investigations on volatile terpenes

W.Herres¹, K.-H.Kubeczka² and W.Schultze²

Essential oils typically represent complex mixtures of mostly terpenoid compounds. Analytical techniques directly working on those mixtures must provide both separation and on-line identification. This is the field of the so-called hyphenated techniques chromatography molecular spectroscopy. HRGC-FTIR has already proved its potential in different fields of application. The informations extracted from HRGC-FTIR are often complementary to those from GC-MS.

The authors report some investigations in the field of volatile terpenes among the general points discussed are: Gram-Schmidt (total IR) versus FID response, vapour phase versus condensed spectra. Considerations are given concerning optimized generation of IR spectral window chromatograms for terpene hydrocarbons with special attention on sesquiterpenes.

Results will be presented and discussed from experiments of isolated compounds, copaiba balsam and parsley leave volatiles.

¹Bruker Analytische Messtechnik GmbH, Wikingenstrasse 13, D-7500 Karlsruhe 21 (FRG)

²Department of Pharmaceutical Biology, University of Würzburg, Mittlerer Dallenbergweg 64, D-8700 Würzburg (FRG)

Selective Detection in HPLC-Analysis
of Essential Oils

S. Ebel, W. Garbade, W. Mück and A. Werner-Busse

Institut für Pharmazie und Lebensmittelchemie
Am Hubland, D-8700 Würzburg

Although GC-analysis and GC/MS- or GC/IR-coupling techniques are the most recommended methods in analysis of essential oils HPLC will have some advantages if there are specific methods of detection. Naturally occurring essential oils contain a number of components with chiral carbon atoms and will occur in most cases only as one enantiomer. Therefore HPLC with a polarimetric detector contrary to other detection methods differentiates between synthetic racemic products and natural enantiomers. In order to have a better signal to noise ratio smoothing algorithms have to be used. Examples are given for essential oils of Mentha and Carum.

Another selective detection is based on photodiode array detectors. Complex chromatograms can be convoluted with the UV-spectrum of the substance to be determined or using convolution procedures with orthogonal polynomials. While most peaks are suppressed or lead to negative signals, the substance to be determined will give amplified signals. Quantitative analysis are possible using the same technique.

The Calculation of Column Polarities

W. Günther, F. Schlegelmilch, J. Wohland,
WGA, Düsseldorf and FH Niederrhein, Krefeld.

Since the basic works of Kovats and especially Rohrschneider the yesteryears choice of columns for gaschromatography is too ineffective, because of its timewasting empirical trying. Nowadays, in fact of using the topic works of these both authors, there is a possibility of a strong scientific calculating of the exact needed column polarity.

In case of Rohrschneider, there is the opportunity to calculate the individual polarity of the components as well as the polarity of the stationary liquid phase.

That means at least, that in practice the needed polarity of the stationary liquid phase should be precalculated before the practical work is started.

In this communication lecture the possibility of calculation of certain compounds, the calculation of different stationary liquid phases as well as the calculation molecular structure of the stationary liquid phase is shown.

Reaction gas chromatography for structure elucidation of terpenoid alcohols. Sesquiterpene alcohols present in the insecticidal essential oil of C. balsamita.

Chrysanthemum

U. Kobold, O. Vostrowsky and H.J. Bestmann
Organic Chemistry Institute, University Erlangen-Nürnberg, Henkestr. 42, D-8520 Erlangen, FRG.

Several monoterpene alcohols were dehydrated catalytically using alumina as the catalyst in the injection port of a gas chromatograph. The reaction products were identified by gas chromatography and mass spectrometry. The relative yields of products were correlated with formation enthalpies $\Delta H_{\text{gas}}^{\text{f}}$ derived from force field calculations.

The method allows the identification of unknown alcohols on a ng or μg scale by the determination of the olefins generated by the dehydration reaction.

The applicability of this technique was demonstrated by the identification of sesquiterpene alcohols present in the essential oil of Chrysanthemum balsamita. This oil showed insecticidal activity on several species of aphids.

The use of high resolution mass spectrometry in GC/MS-coupling for analysing complex mixtures of volatiles

Gerda Lange

Institute of Organic Chemistry, University

Wulf Schultze

Department of Pharmaceutical Biology, University

D-8700 Würzburg (FRG)

Volatile substances biosynthesized by biological systems often appear in the form of very complex mixtures. Sometimes their total amount is too small to carry out any isolation of individual compounds, e.g. this applies to distillates of some plant tissue cultures.

In such cases structural information can only be gathered by data obtained from coupling techniques like GC/MS or GC/FTIR.

In this paper we want to give some examples illustrating the possibilities, advantages and limitations of HRMS (high resolution mass spectrometry) in combination with capillary gc. First, some experimental parameters of this technique are discussed, e.g.

- choice of the reference compound and its introduction into the mass spectrometer
- selection of reference peaks and their influence on accurate mass calculation
- interrelationship between resolution, sensitivity and scan-rate

Furthermore, two selected examples shall demonstrate that reliable HRMS data can even be gained from very small gc peaks being partially overlapped by another compound.

Finally, analysing a distillate of plant tissue cultures for two particular groups of compounds (2,4-dienals and alkyl-furanes) by means of mass chromatography, the advantage (enhanced selectivity) of carrying out this technique in HRMS mode (in contrast to LRMS) becomes evident.

Direct analysis of plant material by mass spectrometry

Wulf Schultze

Department of Pharmaceutical Biology, University

Gerda Lange

Institute of Organic Chemistry, University

D-8700 Würzburg (FRG)

Georg Heinrich

Institute of Plant Physiology, University

A-8010 Graz, Austria

A quick and reliable identification of one (or more) characteristic compound(s) of a very small piece of plant material is of interest, when investigations are carried out concerning

- a) the transport and distribution of a particular component within one single plant
- b) the detection of chemotypes
- c) the analysis of dried medicinal plants (drugs)

This paper only deals with the last subject and shows that many plant drugs (i.e. small pieces of them) can be introduced directly into a mass spectrometer and get analysed without any previous preparations.

Analysis was done in normal EI mode (70 eV), the application of special methods such as CA was not necessary. The requirements, advantages and limitations of this type of ms measurement are discussed. Several selected drugs representing different groups of secondary compounds (essential oils, coumarins, phthalides, alkaloids) were investigated in this way.

The results demonstrate that this method is an important tool for

- detecting an adulteration of drugs
- checking, whether particular components of the drug became destroyed during storage
- investigating several parts of a dried plant with regard to differences in their composition of secondary products.

Essential Oil Composition of Some Nigerian Medicinal Plants

Olusegun Ekundayo

Department of Chemistry,
University of Ibadan,
Ibadan. Nigeria.

Abstract

Biological and pharmaceutical importance of essential oils is well established. However, the composition of only a few essential oils from Nigerian medicinal plants have been investigated so far. In this paper, a survey of the existing data in the literature is compiled and the more recent results of the analytical studies of some essential oils are also reviewed.

THE CHEMICAL COMPOSITION OF LAUREL LEAF OIL,
OBTAINED BY STEAMDISTILLATION AND HYDRODIFFUSION.

Mans H. Boelens and Rafael Jimenez Sindreu

DESTILACIONES BORDAS CHINCHURRETA S.A.

Apartado 11, Seville, SPAIN.

The chemical composition of the essential oil of the leaves of *Laurus Nobilis* L., which was obtained by steamdistillation and by hydrodiffusion, has been studied.

About 30 constituents have been quantitatively detected in these oils. These constituents can be subdivided into the following groups of chemical compounds: monoterpenes, 1,8-cineole, monoterpenoid alcohols, monoterpenoid esters, sesquiterpenes and benzenoids. There exist striking differences between the concentration of the constituents in the steamdistilled and the hydrodiffusion oil.

	steamdistilled	hydrodiffusion
monoterpenes HC	26%	16%
1,8-cineole	43%	28%
monoterpenoid alcohols	17%	27% ₁₇
monoterpenoid esters	9%	16%
sesquiterpenes HC	1%	1%
benzenoids	4%	12%

Possible explanations for these differences will be discussed, namely hydrolysis/dehydration (alcohols/esters - monoterpenes), solubility in water (1,8-cineole), more extensive extraction during hydrodiffusion (benzenoids).

The organoleptic quality of the hydrodiffusion oil is superior to that of the steamdistilled oil, possibly due to the higher concentration of the benzenoids.

NATIVE SOUTHERN AFRICAN AROMATIC PLANTS - A NEW VEHICLE FOR
RURAL DEVELOPMENT

E.H. GRAVEN, J.B. GARDNER & C.L.C. TUTT

Agricultural and Rural Development Research Institute,
University of Fort Hare, Alice, Ciskei, via Republic of South Africa

Southern Africa is richly endowed with aromatic plants some of which appear to have potential as essential oil crops. In Ciskei it has been found that given the right economic incentive, rural people will enthusiastically collect Tagetes minuta (Tagetes), Artemisia afra (S.A. Armoise) and Pteronia incana (Pteronia) and that these plants could form the basis of an essential oil industry. The fact that these plants occur as weeds in grazing and cultivated fields, appears to have additional merit for the establishment of such an industry.

Using the Ciskei Essential Oils project (CENTOIL PROJECT) which is located at the University of Fort Hare as a basis for discussion, the paper chronicles problems encountered in the production, processing and marketing of oils from the above plants and discusses the development of an essential oil industry for the benefit of a rural community. Reference is also made to the research concerning Eriocephalus punctulatus (Erocephalee), a highly scented mountain shrub from which is produced an attractive oil which seems to have considerable market potential.

New Terpenoids from Mexican Stevia Species

C. Zdero

Department of Organic Chemistry University
of Berlin

Straße des 17. Juni 135

D-1000 Berlin 12

- Abstract is not available -

Comparison of the Volatiles of some Satureja and
Calamintha species

H.L. De Pooter and N.M. Schamp

Laboratory of Organic Chemistry,
Faculty of Agricultural Sciences, State University of Gent,
Coupure links 653, B-9000 Gent, Belgium

The first difficulty one encounters when working with some Satureja and Calamintha species, is the identification of the plant material on hand. The multitude of names, frequently of different genera, under which some species are known, illustrates the problems in classifying Satureja and Calamintha.

Adzet and Passet [1] proposed to help differentiate between these genera on the basis of the oil composition, in that Satureja should contain large amounts of phenolics and aromatics, and Calamintha would be characterized by derivatives of p-menthane, oxygenated on C-3.

In order to test the applicability of this proposal, the following plants with different fragrances, and named Satureja and/or Calamintha, were studied : C. nepeta (\equiv S. calamintha; mint-like odour), C. nepeta ssp glandulosa (musty), C. sylvatica (flowery mint-like), Clinopodium vulgare (\equiv Calamintha clinopodium; almost odourless).

The essential oil of C. nepeta was found to contain iso-menthone and pulegone [1]; C. nepeta ssp glandulosa was rich in piperitone oxide and piperitenone oxide; C. sylvatica contained a large amount of neomenthol. This tends to confirm the proposal of Adzet and Passet.

On the other hand, in Clinopodium vulgare oil no p-menthane derivatives were detected, the major components being β -caryophyllene and germacrene-D. In this case the classification Clinopodium would seem more indicated than Calamintha.

[1] Adzet, T. and Passet, J. (1972). Riv. Ital. EPPOS 54, 482.

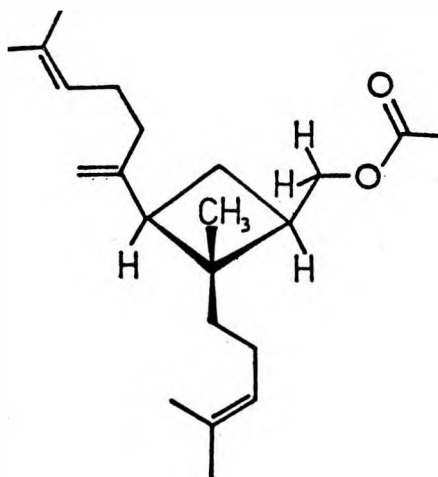
A Volatile Diterpenoid from *Ammi visnaga* (L.) Lam. Fruits

Elisabeth Stahl⁺) and V. Sinnwell⁺⁺)

⁺) Lehrstuhl für Pharmakognosie, Universität Hamburg,
Bundesstr. 43, D-2000 Hamburg 13

⁺⁺) Institut für Organische Chemie, Universität Hamburg,
Martin-Luther-King-Platz 6, D-2000 Hamburg 13

The fruits of *Ammi visnaga* (Apiaceae) are of pharmaceutical importance because of their content of pyranocoumarins and furanochromones. In addition they contain 0,03 % of an essential oil, which has recently been the subject of our chemical investigation. Besides some common monoterpenoids and aliphatic esters, a diterpenoid ($C_{22}H_{36}O_2$, m.w. 332) has been isolated, which was proved to be an acetic ester of a diterpene alcohol. By means of modern nmr spectroscopy, the structure of this diterpene ester could be established.



It can be considered as a cyclisation product of the two monoterpenoids myrcene and geraniol.

Volatile Constituents of *Achillea wilhelmsii* C. Koch (Syn. *A. santolina* auct. mult.) with different regional origin

E.-J. Brunke and F.-J. Hammerschmidt
DRAGOCO Gerberding & Co. GmbH, Research Laboratories
D-3450 Holzminden, Germany

Achillea wilhelmsii C. Koch (Syn. *A. santolina* auct. mult.) (Compositae) is an annual herb growing on dry sandy places, sometimes covering the ground. Its distribution is from North Africa, Turkey, southern parts of the Soviet Union to the Iran. The plant has been used as popular medicine in Egypt and other islamic countries.

We have detected the occurrence of different geographic races of *A. wilhelmsii*. The essential oil, obtained from plant material growing wild in Egypt, contained as main constituents:

α -pinene (1.0 %), camphene (2.0 %), 1,8-cineole (13.1 %), γ -terpinene (1.6 %), p-cymene (3.5 %), α -thujone (1.4 %), trans-sabinene hydrate (1.0 %), camphor (14.1 %), terpinenol-4 (6.5 %), fragranol acetate (23.4 %), and fragranol (8.5 %).

The plant material, collected in Anatolia (Turkey) contained as main constituents: α -pinene (2.7 %), camphene (7.6 %), 1,8-cineole (21.4 %), γ -terpinene (1.2 %), p-cymene (2.0 %), artemisia alcohol (1.4 %), camphor (36.1 %), terpinenol-4 (3.0 %), borneol (4.0 %), and thymol (2.6 %), β -eudesmol (1.5 %) and caryophyllenols (1.8 %).

The main difference between the regional races of *A. wilhelmsii* is the exclusive occurrence of fragranol and its esters in the plant material from Egypt (fragranol type) and the occurrence of borneol and thymol, as well as the absence of fragranol and its derivatives in the plant material from Turkey (borneol/thymol type).

In addition, more than 60 minor components have been identified in each of the two essential oils and could be used for further differentiation between the two chemotypes.

ULTRASTRUCTURE OF THE ESSENTIAL OIL SECRETION IN THE OIL GLANDS OF
THE FRUIT PEEL OF MANDARIN (CITRUS DELICIOSA TEN.)

A.M. BOSABALIDIS and I. TSEKOS

Department of Botany, University of Thessaloniki,
Thessaloniki 54006, Greece

Abstract

The exocarp (fruit peel) of mandarin possesses numerous secretory structures producing an essential oil. Each of them consists of a globular/oval glandular body, in which an inner cavity is progressively formed. Opening of this cavity starts from a single cell located in the central region of the glandular body. This cell exhibits typical features of lysis and finally disintegrates. Disintegration phenomena gradually extend to the neighbouring cells resulting in the enlargement of the cavity, in which the essential oil will later accumulate.

The secretion process begins with an impressive increase in the number of plastids in the secretory cells. In the plastid matrix a membranous tubular network develops, in the area of which numerous oil droplets initiate. The mentioned morphological characteristics of the plastids observed during the stage of secretion strongly suggest that these organelles constitute the intracellular sites of the essential oil biogenesis. Uptake of the exudate from the plastids is performed by the endoplasmic reticulum elements, which further align along the walls of the secretory cells. Fusion of the endoplasmic reticulum membranes with the plasmalemma results in the release of the essential oil within the apoplast (channel network), through which it is transported to the central cavity of the gland.

Morphology and Chemistry of the Essential Oil Secretion of Dictamnus
albus

K.-H. Kubeczka
Department of Pharmazeutical Biology
University of Würzburg
Mittlerer Dallenbergweg 64
D-8700 Würzburg

- Abstract is not available -

COMPARATIVE STUDY ON THE PRODUCTION AND ACCUMULATION OF
ESSENTIAL OIL IN THE WHOLE PLANT AND IN TISSUE CULTURES OF
PIMPINELLA ANISUM L.

J. REICHLING, G. BURKHARDT, R. MARTIN, H. BECKER

Institut für Pharmazeutische Biologie der Universität
Heidelberg, Im Neuenheimer Feld 364, 6900 Heidelberg 1, FRG

A comparative study on the essential oil composition of the whole plant, callus- and suspension cultures of *Pimpinella anisum* L. was carried out.

For the first time the whole organism and the tissue cultures were both observed to be similar in their constituents e.g. pseudoisoeugenol-2-(2-methylbutyrate), epoxy-pseudoisoeugenol-2-(2-methylbutyrate) and β -bisabolene. Anethole, the main component of the 'fruit-oil', could occasionally be detected in the essential oil of the tissue cultures. Divergent from the whole plant, the callus culture contained myristicine.

In addition to β -bisabolene we could identify the following sesquiterpenoids in the essential oil of fruit and shoots:

Fruit-oil: δ -elemene, α -copaene, ar-curcumene, α -himachalene, β -himachalene and γ -himachalene.

Shoot-oil: δ -elemene, ar-curcumene, γ -himachalene, sesquiphellandrene, δ -cadinene, α -humulene, caryophyllene, β -farnesene, germacrene-D, bergamotene, geijerene and pregeijerene.

Additionally the diterpenoid phytadiene I was isolated.

Antimicrobial screening of essential oils

A.M. Janssen, J.J.C. Scheffer and A. Baerheim Svendsen

Division of Pharmacognosy, Center for Bio-Pharmaceutical Sciences,
Leiden University, Gorlaeus Laboratories, P.O. Box 9502,
NL-2300 RA Leiden.

For essential oils a wide variety of biological activities has been described, for example effects on the central nervous system, spasmolytic, insecticide, insect attractant or repellent and antimicrobial effects (1). The antimicrobial activities have gained much attention. This might be caused partly by a high antimicrobial potential of essential oils, but also by the fact that antimicrobial activities can be measured in simple assay techniques. The techniques used can be divided roughly into agar overlay techniques (also called disc diffusion technique) and dilution techniques. The present communication will deal with various aspects that can be of importance when the agar overlay technique is used in case of essential oils: e.g. amounts applied onto a disc, activities of monoterpene hydrocarbons, changes in the composition of oils during application, vapour phase activities and others.

(1) H. Schilder, Dtsch. Apoth.-Ztg. 124 (1984) 1433-1442.

On Biochemical Actions of Terpenoids

Karl KNOBLOCH, Hildegunde WEIGAND and Norbert WEIS
Institut für Botanik und Pharmazeutische Biologie, Universität
Erlangen-Nürnberg, Schlossgarten 4, D-8520 Erlangen, Fed.Rep.of Germany

Essential oils of rather wide origin are known to reveal antimicrobial actions. This is especially due to defined terpenoids in a medium where they may be emulsified.

Being lipid soluble, essential oils will dissolve in biological membranes such as mitochondria, chloroplasts or bacterial cytoplasmic and intracytoplasmic membrane systems.

Bacterial metabolic pathways can be overlooked more clearly compared to higher organized structures. Among the purple bacteria, one of the most ancient orders of form of life on the earth, the Rhodospirillaceae are of special interest. They grow either aerobically in the dark or anaerobically in the light using organic substrates. Depending on the environmental oxygen content, they develop the enzymatic respiratory system within the cytoplasmic membrane or the light-harvesting and photosynthetic active intracytoplasmic membrane structures.

Cell-free membrane preparations of these organisms, e.g., *Rhodopseudomonas sphaeroides* or *Rhodospirillum rubrum*, are used frequently to study energy metabolism pathways.

We found that essential oils or terpenoids will dissolve within the biological membranes thus interfering with respiratory or photosynthetic electron transport, concomitantly occurring proton translocation and coupled phosphorylation steps. Known chemicals at rather low concentrations may function as inhibitors or uncouplers at specific sites within the molecular biological arrangements.

Using membrane preparations from *R. sphaeroides* and NADH as the oxidizable substrate, we observed that, e.g., *p-cymene* at a 5 mM conc. inhibits both, respiratory electron flow and oxydative phosphorylation to about 25%, and that proton translocation is decreased by 15%. *Carvacrol* is even more effective and at a 5 mM level totally diminishes all energy transforming steps. Even 0.2 mM *carvacrol* will inhibit electron flow and coupled phosphorylation up to 50%. Under the same conditions, *menthone* (5 mM), e.g., causes 80% inhibition. 5 mM *linalyl-acetate* reveals a more pronounced uncoupling effect on the generation of ATP (80%) than on the electron transport (60% inhibition), whereas under its influence proton translocation is continuously increasing with time.

In search of the action of terpenoids on cell metabolism, bacterial membranes appear to be very useful.

The Essential Oil of Nigella sativa L. Seeds

E.A. Aboutabl*, A.A. El-Azzouny** and F.-J. Hammerschmidt

* Pharmacognosy Dept., Faculty of Pharmacy, Cairo University, Egypt

** Pharmaceutical Sciences Laboratories, National Research Center,
Dokki, Cairo, Egypt

*** DRAGOCO Research Lab., D-3450 Holzminden, Germany

Nigella sativa L. (Ranunculaceae) is indigenous and grows well in different localities of Egypt. The seeds of this plant were used by ancient Egyptians and Arabs in the treatment of several diseases including asthma, cough, bladder and kidney calculi. In addition they are used as carminative and as well as the essential oil as flavouring agent for food and beverages.

Literature on the constituents of the volatile oil of N. sativa L. seeds is rather conflicting (1-4). Therefore, the essential oil obtained by steam distillation was analysed by capillary gas chromatography (60 m DB Wax, cross-linked Carbowax) coupled to mass spectrometry.

More than sixty components were separated and identified. The oil contains ~46 % monoterpene hydrocarbons, mainly p-cymene (~32 %) and α -pinene (~9 %), and 25 % of carbonyl compounds, principally thymoquinone (24.5 %). Phenols (~1.7 %), alcohols (~0.9 %) and esters (~16 %) are also present. These are mainly the ethyl esters of certain saturated and unsaturated fatty acids. Eight disulphides in very small amounts proved to be present in the oil.

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Unterschiede in der Zusammensetzung des ätherischen Oles von einzelnen Pflanzen aus einem Klon von *Valeriana officinalis* L. s.l.

R. Bos⁺, F. M. S. v Putten⁺, H. Hendriks⁺ und C. Mastenbroek^{*}

+ Labor für Pharmazeutische Chemie und Pharmakognosie, Ant. Deusinglaan 2,
NL-9713 AW Groningen

* V. N. K., P. O. Box 1, NL-8080 AA Elburg

Die Züchtung von *Valeriana officinalis* zielt auf:

1. Erhöhung des Wurzelertrages mittels Auslese auf dickere Wurzel in der Annahme das diese sich zu einem grösseren Teil mechanisch ernten lassen.
2. Verbesserung des Gehaltes an wirksamen Bestandteilen wie z.B. ätherisches Öl, Valerenan-derivate(Valerensäure) und Valepotriate.

Anfangs wurden Einzelpflanzen auf Valepotriatgehalt, aber ab 1984 auch auf Valerensäuregehalt ausgelesen.

Der Klon 79.60 zeigte sich reich an Öl, Valepotriate und Valerensäure. Zwei verschiedene Nachkommenschaft wurden einzelpflanzenweise untersucht.

- a. die Nachkommenschaft nach Bestäubung durch andere Ölreiche-Klone (85.01 bis 85.25)
- b. die Nachkommenschaft von a. nach Bestäubung durch nichtselektiertes Material (85.26 bis 85.76).

Der ätherische Ölgehalt ist für die unter a. genannten Pflanzen höher (von 0.4 bis 1.1%) als die unter b. genannten Pflanzen (von 0.1 bis 0.7%).

Von den unter a. genannten Pflanzen ist Valerenal in 11 von 21 Öle der Hauptkomponent.

In den meisten Fällen ist ein hohe Ölgehalt mit einem hohen Valerensäuregehalt verbunden. Zusätzlich kann man die ätherischen Öle unterscheiden nach den Haupt-Substanzen wie z.B. ein Valerenal-Typ, ein Valeranon-Typ, ein Sesquiterpenalkohol-Typ und ein Ketovaleranon-Typ.

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TITLE - The Essential oil of portuguese pine needles. First results.by CARMO,
M.M., and FRAZÃO , S.

(Serviço dos Produtos Naturais - DTIQ - LNETI)

Estrada das Palmeiras - 2745 QUELUZ DE BAIXO - PORTUGAL

ABSTRACT

In order to have a knowledge of the characteristics of the essential oil from portuguese pine needles we carried on a few distillations at different seasons of the year.

We have studied samples of essential oil from Pinus Pinaster Aiton and from Pinus Sylvestris L.

The results of their characteristics are compared with the ones of the oils from different geographical origines.

The chromatograms obtained by GLC on packed and cappillary columns are given , and the main components are identified.

IDENTIFICATION OF TERPENE ALCOHOLS IN TERPENE
 β -D-GLUCOSIDES IN THE LEAVES OF DALMATIAN
SALVIA OFFICINALIS

* K.Grzunov, J.Mastelić, N.Ružić
Laboratory of Organic Chemistry,
Faculty of Technology, Split
Yugoslavia

Terpene alcohols of terpene glucosides were determined in fresh *Salvia officinalis*.

The extraction was done with water and glucosidase inactivated by heating the extract.

After Pb-acetate precipitation and extraction with alcohol and acetone the final purification was carried out by preparative chromatography.

Extracted terpenylglucosides were subjected to enzyme hydrolyses under the pentan layer.

Aglycone identification was done by TLC and GCh with pure alcohols like standards.

These investigation suggested that terpene alcohols in β -D-terpene-glucosides isolated from Dalmatian sage were thujol, menthol and thymol.

Essential Oil of CEDRELA ODORATA L. (MELIACEAE) from Brazil

Revised List of Constituents

E.-J. Brunke, F.-J. Hammerschmidt, F.-H. Köster
DRAGOCO Research Laboratories
D-3450 Holzminden, West Germany

Cedrela odorata L. (Meliaceae) is a large tree, indigenous to the West Indies, Central and South America. The essential oil, mainly produced in Brazil by steam distillation of the wood, is used by man in popular medicine, as wood preservative and in perfumery.

O. Motl and A. Trka¹⁾ identified in a first analysis of the essential oil four sesquiterpene alcohols (epi-cubenol, T-muurolol, torreyol, juniper camphor) and additional fourteen sesquiterpene hydrocarbons.

In our analysis we used the combination GLC/MS and authentic reference substances. The results of Motl and Trka could be confirmed extensively. In addition a number of sesquiterpene hydrocarbons and alcohols were identified, mainly

- β -cubebene 3,2 %; calarene 3,4 %; β -farnesene 1,8 %; α -humulene 2,1 %; β -curcumene 6,8 %
- nerolidol, E 8,0 %; cubenol 0,96 %; β -bisabolol 0,53 %, T-cadinol 0,80 %; α -cadinol 1,5 %; 2Z,6E-farnesol 0,11 %; 2E,6E-farnesol 0,31 %.

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SCREENING FOR VOLATILE TERPENES IN YEASTS

Andreas Klingenberg, Ewald Sprecher, and Hans-Peter Hanssen

Lehrstuhl für Pharmakognosie der Universität, Bundesstr. 43,
D-2000 Hamburg 13, Federal Republic of Germany

23 yeast strains belonging to 17 species have been screened for the occurrence of volatile terpenes. After cultivation on a defined liquid culture medium containing glucose (5 %), yeast extract (0.25 %), and vitamins (thiamine and biotin, each 0.04 %), volatiles -obtained by steam distillation and analysed by means of GC/MS coupling- were determined five to six times during each culture period.

Besides trace components belonging to various chemical classes, many strains accumulated predominantly short chain alcohols and esters, and phenyl compounds (benzyl alcohol and phenylethyl alcohol derivatives) in considerable amounts (mg/l-range).

Additionally, almost all strains produced terpenoid compounds, but in most cases only acyclic sesquiterpenes (farnesol, nerolidol, and their derivatives) could be traced. In this study, only strains of the ascosporegenous genus *Ambrosiozyma* were found to accumulate monoterpenes (mainly linalool, geraniol, citronellol) in considerable amounts (up to 60-100 mg/l).

Terpene production by yeast strains is discussed considering generic differentiation and morphology, taxonomy and individual species and strain ecology.

GC/MS analysis of essential oils of some *Tagetes* species

É.Héthelyi, B.Dános and P.Tétényi

Research Institute for Medicinal Plants, Budakalász,
Hungary

The *Tagetes* genus has been studied in our Institute for 3 years. This collection is increased by seed exchange from botanical gardens, controlled during growing, and their morphological-phenological-histological and chemical characteristics are described.

In this paper the chemical composition of 5 flowering species is shown. The determinations were carried out by GC/MS method.

The oil of *Tagetes minuta* L. contained 38 % β -ocimene, 10 % dihydrotagetone, 10,7 % tagetone, 5 % Z-ocimenone and 26 % E-ocimenone as components. The *T.tenuifolia* Cav. species contains the same components but in a different proportion: 25 % tagetone and 33 % E-ocimenone was determined in the oil. The species *T.natula* L. contained 2 % myrcene, 14 % limonene, 12 % β -ocimene, 7 % tagetone, 13,4 % Z-, and 13,4 % E-ocimenone and instead of dihydrotagetone 12 % β -caryophyllene. *T.erecta* L. and *T.lucica* Cav. contained an essential oil of very different composition from the previous species that is myrcene, limonene, β -ocimene, β -caryophyllene and two unknown components, respectively 15 % piperitone in the oil of *T.erecta* was detected.

VOLATILE CONSTITUENTS OF CORIANDER FRUIT CULTIVATED AT DIFFERENT LOCALITIES AND ISOLATED BY DIFFERENT METHODS

Timo Hirvi^{1, 3} Irma Salovaara¹ Hannu Oksanen² & Erkki Honkanen¹

Technical Research Centre of Finland

1) Food Research Laboratory, Biologinkuja 1

2) Chemical Laboratory, Biologinkuja 7

SF-02150 ESPOO, FINLAND

Coriander (*Coriandrum sativum* L.) is an annual herb originating in the Middle East. At the present time coriander fruit is produced principally in India, Morocco, Italy and Eastern Europe. The plant is also in an experimental cultivation in Finland, because it has been claimed that the climatic conditions prevailing in Northern Europe yield to the herb plants a stronger aroma than that of herbs grown elsewhere.

The present work is a part of a wider study concerning the cultivation of the herb plants in Finland. In this study the effect of different factors on the quality and aroma content of herb plants are examined. The variables include variety, latitude of the place of growth, use of fertilizers, time of harvesting and storage time.

In this preliminary study three coriander varieties were grown at two different localities in the years 1983 and 1984. The oil was isolated by steam distillation. The effect of the variety and latitude on the aroma content is discussed.

The effect of the isolation method was studied using one commercial coriander fruit sample. The oil was isolated both from intact and ground fruits by three methods: steam distillation, CO₂ extraction and extraction with pentane : diethyl ether mixture. The volatile constituents were analysed using gas chromatography and mass spectrometry. For comparison, the volatiles of three different commercial coriander oil extracts were also studied. In all, about 40 compounds were identified and quantified. The influence of the isolation method on the amount of the volatiles is discussed.

3) The present address Soil Analysis Service Ltd

(Viljavuuspalvelu Oy)

Vellikellontie 4, SF-00410 HELSINKI, FINLAND

Stability of 3,6-dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran in aqueous buffer solutions

Rainer Huopalahti

Department of Chemistry and Biochemistry, Laboratory of Food Chemistry, University of Turku, SF-20500 Turku, Finland

In recent studies 3,6-dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran has been identified as one of the major aroma components in the dill herb, exhibiting a pleasant odour similar to the fresh dill herb.

Although 3,6-dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran can be synthesized, detailed information on its stability is, however, lacking. Information of the stability of the benzofuranoid is important for its possible use in the flavour industry. Therefore, in the present work the decomposition of the benzofuranoid in aqueous buffer solutions in the pH range 2-8 at temperature between 5 and 100°C was studied.

3,6-Dimethyl-2,3,3a,4,5,7a-hexahydrobenzofuran was isolated from flowering dill by extraction. The extract was fractionated by vacuum distillation and preparative gas chromatography. The purity of the benzofuranoid was confirmed by GC using the fused silica capillary column coated with the optically active stationary phase.

The benzofuranoid turned out to be a rather stable compound having its maximal stability at pH 4. Its shelf-life at room temperature and at pH 4 was a few months, at 5°C almost a year. The decomposition followed first-order kinetics.

Studies on the Essential Oil of Zanthoxylon armatum of Nepalese Origin
A Suitable Oil for Industrial Production

J. Karlsen

Institute of Pharmacy, University of Oslo

P.O. Box 1068, N-0316 Oslo 3

- Abstract is not available -

Qualitative and Quantitative Determination of Essential Oils of
Eucalyptus Species in Iran

F. Moattar

Department of Pharmacognosie, Faculty of Pharmacy

University of Isfahan, Iran

- Abstract is not available -

Antiinflammatory efficacy of terpenes from *Conyza canadensis*

J. Lenfeld, O. Motl⁺ and A. Trka⁺

Department of Pharmacology, Faculty of Medicine of the
Palacký University, 771 00 Olomouc, Czechoslovakia

⁺Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, 166 10 Prague 6, Czechoslovakia

Conyza canadensis (L.) Cronq. syn. *Erigeron canadensis* (Asteraceae) is an annual occurring practically in all continents as a weed. An essential oil for predominantly medical purposes used to be prepared in small amounts from aerial parts of the plants, and it was called *Oleum erigerontis*.

The essential oil and mainly the light petroleum and ethanol extracts have an antiinflammatory effect. The light petroleum extract was fractionated by column chromatography (alumina, silica gel, argentated silica) and also submitted to pharmacodynamic screening (carrageenine and formalin oedema of rats).

The most strongly antiinflammatory fraction contained 8 sesquiterpenic hydrocarbons. In this mixture the following compounds have been identified by means of GLC/MS: β -santalene, β -himachalene, cuparene, α -curcumene, γ -cadinene. When tested alone, β -himachalene also had an antiinflammatory effect.

Composition of the essential oil from *Heracleum dissectum*

V.P. Papageorgiou⁺, G. Ochir⁺⁺, O. Motl⁺⁺, N. Argyriadou^x
and H. Dunkel^{xx}

⁺Laboratory of Organic Chemistry, College of Engineering,
University of Thessaloniki, Greece

⁺⁺Institute of Organic Chemistry and Biochemistry, Czecho-
slovak Academy of Sciences, 166 10 Prague 6, Czechoslovakia

^xVIORYL S.A., Kifissia, Athens, Greece

^{xx}NATEC Institute, 2000 Hamburg 50, West Germany

The perennial plant *Heracleum dissectum* Ledeb. (Umbelliferae) occurs mainly in East Asia where it is used as an edible plant (for preparation of salads) and recently also as fodder. The aerial part affords 0.1% of an essential oil on steam distillation.

The essential oil was analysed by means of computerized GLC/MS, 64 components were detected. The main components (in %) are: α -pinene (22.2), myrcene (10.9), kessane (8.8), humulene (8.3), β -phellandrene (5.5), kessanyl acetate (3.2), limonene (2.8), linalool (2.4), β -cis-ocimene (2.3), kessyl acetate (2).

Composition of the essential oil of Ocimum canum grown in Rwanda

L. Ntezurubanza^{1,2}, J.J.C. Scheffer², A. Looman² and
A. Baerheim Svendsen²

¹CURPHAMETRA, B.P. 117, Butare, Rwanda (permanent address)

²Division of Pharmacognosy, Center for Bio-Pharmaceutical Sciences,
Leiden University, Gorlaeus Laboratories, P.O. Box 9502,
NL-2300 RA Leiden

Ocimum canum Sims. occurs wild in tropical Africa as well as in other parts of the world. The species grows wild also in Rwanda (Central Africa), where it is used in traditional medicine and for aromatic purposes. In the literature much confusion exists about O. canum. The names O. canum, O. americanum and also O. basilicum and O. kilimandscharicum have been interchanged, particularly for camphor-containing plants. Besides 'camphor-type', also 'methyl cinnamate-type', 'citral-type' and 'linalool-type' essential oils of O. canum have been reported. Because of the confusion about O. canum and since a taxonomic study on Ocimum species growing in Rwanda is performed at the moment, we were interested to investigate whether different chemical varieties of O. canum should be present in Rwanda.

Leaves and flowers of O. canum specimens growing wild in eastern and southern Rwanda (at Kibungo and Butare respectively) were subjected to hydrodistillation. The essential oil samples were analyzed by combined LSC and GLC, and by GC-MS. Comparison with authentic samples and mass spectrometry were used for identification of the essential oil components.

All essential oil samples were characterized by a high content of linalool (60-90%). Neither camphor nor citral and methyl cinnamate could be detected. The samples with the lowest content of linalool (ca. 60%) contained relatively large amounts of sesuiterpene hydrocarbons such as bergamotene (tentatively identified; ca. 10%) and β -caryophyllene (ca. 5%). All monoterpene hydrocarbons were present in minor amounts, limonene (ca. 1%) being the most important one. Oct-1-en-3-ol (ca. 2.5%) and 3-octanol (ca. 1.5%) were the only oxygen-containing components, besides linalool, which amounted to more than 1% in most of the oil samples.

Carvacrol and thymol chemotypes of wild east Mediterranean Labiatae herbs

U. Ravid and E. Putievsky

Department of Medicinal and Spice Crops

Agricultural Research Organization Neve Ya'ar
Experiment Station, P.O. Haifa, 31999, Israel.

Abstract

The essential oils of five phenolic herbs growing wild in the east mediterranean basin were studied. Carvacrol and thymol chemotypes were found in Thymbra spicata, Satureja thymbra, Majorana syriaca, Coridothymus capitatus and Origanum vulgare. The major components in the oils were identified by GLC and GC - MS.

The essential oils of three *Origanum* species grown in Turkey

E. Şarer¹, J.J.C. Scheffer², A. Looman² and A. Baerheim Svendsen²

¹Faculty of Pharmacy, Ankara University, Tandoğan - Ankara, Turkey

²Division of Pharmacognosy, Center for Bio-Pharmaceutical Sciences, Leiden University, Gorlaeus Laboratories, P.O. Box 9502, NL-2300 RA Leiden

Origanum species have been used in medicine and as spices ever since antiquity, mainly because of their content of essential oils. In previous studies we investigated the essential oil of *O. majorana* grown in southern Turkey (1) and we studied its antimicrobial activity. For the present study we collected flowering parts of *O. onites* L., *O. syriacum* L. var. *bevanii* (Holmes) Ietswaart and *O. vulgare* L. ssp. *hirtum* (Link) Ietswaart. The plant material was identified by Ietswaart who made a taxonomic revision of the genus *Origanum* (2).

About 1 kg of dried material of each specimen was collected. The flowering parts were subjected to hydrodistillation for 3 h, which yielded 2-3% of essential oil. The oils were analyzed by GC using 50 m fused silica capillary columns.

The analysis of the three oil samples showed that they were characterized by a high content of carvacrol (60-75%). Other oxygen-containing components were only present in minor amounts, except linalool that amounted to 5% in *O. onites* oil. Two monoterpene hydrocarbons were found in relatively large amounts: γ -terpinene (3-9%) and p-cymene (5-13%). Further analytical data and the results of an antimicrobial screening carried out by means of the agar overlay technique as described elsewhere (3) will be presented.

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Analysis of the volatiles from fruits of *Heracleum* species
by HSGC - an interlaboratory study

J.J.C. Scheffer¹, R. Hiltunen², M. von Schantz² and
A. Baerheim Svendsen¹

¹Division of Pharmacognosy, Center for Bio-Pharmaceutical Sciences,
University of Leiden, Gorlaeus Laboratories, P.O. Box 9502,
NL-2300 RA Leiden

²Division of Pharmacognosy, School of Pharmacy, University of
Helsinki, Fabianinkatu 35, SF- 00170 Helsinki, Finland

Fruits of different *Heracleum* species as well as the essential oils isolated by hydrodistillation from some of the fruits were analyzed by headspace gas chromatography (HSGC). The study was carried out in two laboratories and the results obtained were compared.

The following material was used: fruits of *Heracleum persicum* collected in Iran, and the essential oil from these fruits; fruits of *H. platytaenium*, various samples collected in Turkey, and the essential oil from one of the samples; fruits of *H. laciniatum* (Tromsø palm), various samples collected in Norway; fruits of *H. sibiricum* collected in Norway.

Gas chromatographs Dani HR 3800 and Packard 428 equipped with headspace samplers Dani HSS 3850 and Dani HSS 3950 respectively were used. Columns: glass (29 m x 0.32 mm i.d.) coated with Carbowax 20M; fused silica (51 m x 0.33 mm i.d.) coated with CP-Wax 57 cb. Identification of components was based on comparison of retention times with those of authentic samples identified by MS (1). One sample of *H. laciniatum* fruits was analyzed successively five times in order to determine the variations of the material and the HSGC method. The coefficients of variation were fairly low.

The composition of the volatiles from *H. platytaenium* and *H. sibiricum* fruits was very much alike, octyl acetate being the main component. The results obtained for the volatile components of *H. laciniatum* and *H. persicum* fruits were like they were obtained for one species grown under different environmental conditions. Further results will be given during the presentation.

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A. Baerheim Svendsen, *Planta Med.* 50 (1984) 56-60.

Adsorbents for the Dynamical Headspace

W. Günther, Monika Lux, F. Schlegelmilch,
WGA, Düsseldorf, Dr. A. Kuntze, Düsseldorf
and FH Niederrhein, Krefeld.

In the "Dynamical Headspace" there is in normal cases - especially in analysis of essential oils - a need for certain adsorbents.

Dynamical Headspace, that means: desorption, concentrating (adsorbing), introduction to GC.

The adsorption is normally done at lower temperatures, mostly liquid-nitrogen trapped. Afterwards the trapped components are to be heated up quickestly. That means, there are adsorbents needed which withstand temperature ranges between -150° and $+200-300^{\circ}\text{C}$.

Here are shown the most used adsorbents in this field. The main work is done in the preconditioning for these adsorption traps.

It is shown, that there is no one of the common adsorbents, being offered which is usable without any - sometimes very timewasting - preconditionings.

Structural elucidation of an irregular sesquiterpene alcohol from *Peucedanum palustre* (L.) Moench

K.-H.Kubeczka¹, G.Schmaus¹ and V.Formacek²

Peucedanum palustre (L.) Moench (Apiaceae), the so-called Milk-Parsley, is a perennial plant, growing at wet places (swampy meadows, peat bogs, alder forests, and banks of lakes) in most of Europe, extending eastwards to Central Asia. Formerly the root was used in medicine and in Slavic countries because of its pungent taste as a substitute of ginger. In this paper the structure elucidation of a polar main constituent of the essential root oil will be described. The plant material was collected at several places in Germany, Belgium and Finland. After steam distillation of the chopped fresh roots and fractionation of the obtained oil by CC, the main component of the root oil was isolated from a polar fraction as a viscous liquid.

By means of spectroscopic methods, especially by MS, ¹H NMR and ¹³C NMR, the structure of the unknown substance was elucidated. It was proved to be the irregular sesquiterpene alcohol sesquilavandulol. This is the first report of the natural occurrence of this compound, which has been synthesized in 1944 (1). The structure determination was confirmed by synthesis and comparison of spectral data.

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¹ Department of Pharmaceutical Biology, University of Würzburg, Mittlerer Dallenbergweg 64, D-8700 Würzburg (FRG)

² Bruker Analytische Messtechnik GmbH, Silberstreifen, D-7512 Rheinstetten-Fo (FRG)

Volatiles of *Orthosiphon stamineus* Benth.

ST. SCHMIDT ¹, R. BOS ²

1 Institut für Pharmazeutische Biologie und Phytochemie
Westfälische Wilhelms-Universität Münster, Hittorffstraße 56
D-4400 Münster

2 Laboratorium voor Farmacognosie, Rijksuniversiteit Groningen
Antonius Deusinglaan 2, NL-9713 AW Groningen

The dried leaf of *Orthosiphon stamineus* Benth. is well known as a drug with diuretic properties. Nevertheless only few details have been reported about its constituents, e. g. flavonoids, saponins or essential oils (1).

In order to study the composition of *Orthosiphon* essential oils commercial leaf drug and fresh greenhouse grown leaves and stems were treated by water steam distillation. The obtained oils were investigated by means of GC and GC-MS.

More than 60 different components could be identified. The oils mainly consisted of large amounts of sesquiterpene hydrocarbons, esp. β -Caryophyllene, β -Elemene and Humulene. Caryophyllene oxide and 2-Octen-3-ol were found to be the main oxygenated structures.

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THE ION TRAP DETECTOR:
THE TECHNIQUES AND ITS APPLICATION
R. Schubert, H.J. Hübschmann,
Finnigan MAT GmbH,
Barkhausenstr. 2, D-2800 Bremen

The Finnigan MAT ION TRAP DETECTOR is a sophisticated GC detector which uses a new mass separation technology. It allows the chemist to separate analytical samples by mass as well as by retention time. Positive identification of the eluting compounds is achieved by supplementing GC retention time data with the mass spectra obtained with the ITD.

This positive identification is further facilitated by the extensive library search capabilities of the system.

Set-up of the ITD as well as data acquisition and processing are handled by an IBM Personal Computer XT. The ITD is interfaced to any capillary GC by means of a flexible transfer line. The applications software programs guide the user to respond with on-screen prompts.

The ion trap is conceived as a three dimensional quadrupole utilizing electrodes with hyperbolic inner surfaces. Conventional mass spectrometers form ions in a source region and then focus and inject the ion beam into the mass analyzer. Lens voltages in the source must be adjusted by a skilled operator or a computer. In the ion trap, ionization and mass analysis occur in the same place. As a consequence the ION TRAP DETECTOR is much simpler to operate. The sample enters the central cavity from the heated transfer line. A filament produces electrons which are pulsed into the cavity by changing the polarity of a control electrode. Electron impact ionization results and a variety of ions unique to the sample's molecular structure are produced and trapped. The ion trap is then scanned by increasing the RF amplitude on the ring electrode. This ejects ions from the central cavity into the electron multiplier.

The application software programs guide the user to respond with on-screen prompts. This is shown by the analysis of mugwort oil (*Artemisia vulgaris*). The data demonstrate the rapid response of the ITD as required for capillary GC detection of complex samples. The data obtained from the ITD can be displayed in a variety of formats. The powerful library search program provides rapid searching of large spectral data bases. Versatile peak detection algorithms enables rapid detection of even coeluting compounds.

GERMINATION OF JASMINUM GRANDIFLORUM Linn. SEEDS -
EFFECT OF SUNLIGHT, SHADE AND DARKNESS

H.C.Srivastava and P.G.Karmakar
Indian Institute of Horticultural Research, Bangalore

Jasminum grandiflorum Linn is considered to be the chief source of commercial perfume in Europe, Mediterranean countries and also in some other parts of the world. At Indian Institute of Horticultural Research, Bangalore, an effort is being made to develop its superior variety with high yield of jasmine essential oil. As part of the programme, during germination of Jasminum grandiflorum Linn. seeds some interesting observations have been recorded. The seeds when sown and exposed to sunlight completely failed to germinate. But on sowing the seeds in shade and in nearly dark condition nearly sixty per cent seeds germinated in about ten days. Thus the results indicated inhibiting effect of sunlight and favourable effect of shady condition on germination of Jasminum grandiflorum L. seeds.

FLORAL BIOLOGY OF JASMINES

H.C.Srivastava and P.G.Karmakar
Indian Institute of Horticultural Research, Bangalore

Jasmines have played a major role in Indian agriculture since remote times owing to their extensive cultivation for flowers which are exceedingly accepted by people for use in social functions, personal decoration and perfume industry. Despite it, jasmines have not received attention of breeders and geneticists. The available information on floral biology of jasmines being meagre investigation on this aspects were undertaken on Jasminum grandiflorum Linn strain Pink Pin and Pink Thrum, Jasminum auriculatum Vahl. var Parimullai, Jasminum sambac Ait. var Gundumalli, and Jasminum calophyllum Wall as an auxiliary to improvement programme at Indian Institute of Horticultural Research, Bangalore. Blooming period varied from 3 to 4.5 months. In all these germplasms maximum number of floral buds opened during 6 to 7 p.m. Anthers of J.calophyllum Wall dehiscence during 4 and 5 p.m. whereas in rest of the three jasmines dehiscence was maximum during 3 to 4 p.m. Pollen grains of all the five different types were almost spherical. Largest (61.2 micron) pollen grains were those of J.grandiflorum Linn strain Pink Thrum. Smallest (43.2 micron) pollen grains were recorded in case of J.calophyllum Wall. Pollen fertility as determined by acetocarmine staining test ranged from 13.50 to 71.24 per cent. Under natural pollination seed setting was almost negligible in J.grandiflorum Linn (both the strains) & J.sambac, Ait var. Gundumalli, 30 per cent in J.calophyllum Wall and 50 per cent in case of J.auriculatum Vahl. var Parimullai.

Chemical Composition and Variation of the Essential Oil from
the Norwegian *Thymus praecox* ssp. *arcticus* and *Thymus pulegioides*

Elisabeth Stahl

Lehrstuhl für Pharmakognosie , Universität Hamburg
Bundesstr. 43, D-2000 Hamburg 13

In the summer 1984, *T. praecox* ssp. *arcticus*, previously analysed in Iceland and Greenland, was collected in the surroundings of Trondheim, its only occurrence in Norway. The composition of the essential oil containing linalyl acetate as main component (70%) and several other terpenes, namely nerolidol, hedycaryol, and T-cadinol, did not differ from that of the Icelandic and Greenland plants. In addition to the seven chemotypes then found in Iceland, a further chemotype was very widespread in Norway, containing T-cadinol as the only sesquiterpene alcohol. Generally, in Norway the T-cadinol types predominate over the nerolidol types.

The essential oil of *T. pulegioides*, which was obtained from plants collected at different places in the Oslo district, consists mainly (75%) of metabolites of the terpene phenol biochemical pathway, namely thymol, carvacrol, δ -terpinene, p-cymene, methyl thymol and methyl carvacrol. Additionally some common monoterpenoids, and the sesquiterpenoids β -caryophyllene, germacrene D and β -bisabolene could be detected. Concerning the chemical variation of this species, two chemotypes, a carvacrol type (75% of the plants) and a thymol type (25% of the plants), could be found growing side by side. Thus, in comparison to *T. praecox* ssp. *arcticus*, *T. pulegioides* shows a remarkably lower genetic variation.

APPLICATION OF HEADSPACE GAS CHROMATOGRAPHY IN ESSENTIAL OIL ANALYSIS VII HYDRODESTILLATION COMPARED WITH HEADSPACE.

Vuorela, H., Hiltunen, R., Pohjola, J. and Laakso, I.

Division of Pharmacognosy, School of Pharmacy, University of Helsinki
SF-00170 Helsinki

Headspace gas chromatography (HSGC) is a very useful and time-saving method for evaluating the quality of aromatic and medicinal plants where volatile compounds are present. The evaluation of these drugs has usually been done by means of conventional hydrodistillation or extraction techniques. The aim of the study was to compare the qualitative and quantitative aspects of HSGC and the conventional hydrodistillation-GC-techniques.

The HSGC analyses were carried out on a DAMI HR 3800 GC coupled to a DAMI HSS 3850 using OV-351 (25 m) fused silica capillary column under the following conditions: volume of sample, vials 20 ml; pressurisation, N_2 , 2,8 bar, 10 sec; volume of sample loop 1 ml; 30 mg samples: Anisi fructus, Anisi stellati fructus, Carvi fructus, Foeniculi fructus (2), Juniperii fructus, Matricariae flos, Menthae pip. folium (2), Rosmarin folium. The concentration calibrations were made using the standard addition technique.

Under these experimental conditions all samples except Matricaria flower heads showed similar patterns of essential oil constituents in HSGC and in distilled oil analysed by GC. The coefficient of correlation for the amounts of compounds in the essential oil for both methods were 0.98 ± 0.03 ($n = 10$) and the slope of the regression curve 0.98 ± 0.23 ($n = 10$).

Quantitative determinations of the total oil amount for flower and leaf samples by HSGC gave values appr. 10 % lower than the volumetric determinations; the correlation between the two methods was good, $r = 1.00$ ($n = 4$). However, the fruit samples were difficult to analyse quantitatively by HSGC due to the small sample size and the difficulty in preparing the sample. The essential oil content of fruits determined by HSGC was about 50 % less than the content obtained by the volumetric determinations, the correlation being poor, $r = 0.17$ ($n = 6$).

The HSGC technique is a reliable and efficient method for qualitative screening of plant materials. It is also well suited for the quantitative determinations of essential oil in leaf and flower materials.

The Thermal Conductivity Detector in Capillary Gaschromatography

W. Günther, F. Schlegelmilch, J. Wohland,
WGA, Düsseldorf and FH Niederrhein, Krefeld.

Because of its wide range linearity, its high sensitivity and low price the FID becomes the most used detector nowadays.

If the components to be analyzed are known, that means the response factors are known, quantitative determination is possible when using a FID.

In practice, that means complex mixtures like essential oils, there is no possibility to determine the response factors of all different substance classes, there is absolutely no way to get quantitative exact results. If there is only one unknown component, there is no possibility to run the 100%-method. It is this the field for the TCD.

Here is discussed the sensitivity of TCDs, the different constructions for the use in Capillary Gaschromatography and mainly the practical connections between capillary columns and the detecting systems.

A Comparison of FID and TCD is shown.

16th International Congress on Essential Oils Neuhaus/Holzminden

- Aboutabl, Prof. Dr. E.
Pharmacognosy Department, Cairo University
Kasr-el-Aini Street, 11562 Cairo, Egypt
- Baerheim-Svendsen, Prof. Dr. A.
Gorlaeus Laboratories, Leiden University
P.O. Box 9502, NL-2300 RA Leiden
- Becker, F.
Sixtus-Werke
Urteibachstr. 3, D-8162 Schliersee
- Boelens, M.
Destilaciones Bordas Chinchurreta S.A.
Carretera Carmona 30, 41008 Seville, Spain
- Bohlmann, Prof. Dr.F.
Institut fuer Organische Chemie der Universitaet, IOC Sekr. C 3
Strasse des 17. Juni 135, D-1000 Berlin 12
- Bonefeld, M.
Steinfurter Str. 128a, D-4400 Muenster
- Bos, R.
Ant. Deusinglaan 2, NL-9713 AW Groningen
- Bosabalidis, Dr. A.M.
Department of Botany, University
GR-54006 Thessaioniki
- Brandauer
Aromachemie, Erich Ziegler GmbH
D-8551 Aufsess
- Buchbauer, Dr. G.
Institut fuer pharmazeutische Chemie der Universitaet
Waehringer Str. 10, A-1090 Wien
- Burghart, Dr. J.
Landesuntersuchungsamt fuer das Gesundheitswesen
Ernsberger Str. 3, D-8000 Muenchen 60
- Carle, Dr. R.
c/o Chemiewerke Homburg
Daimler Str. 25, D-6000 Frankfurt 1
- Carmo, Dr. M.M.
Division of Natural Products
Estrada das Palmeiras, P-2745 Queluz de Baixo
- Duprey, R.
c/o Bush Boake Allen Ltd.
Blackhorse Lane, Walthamston London E17 5QP
- Ebel, Prof. Dr. S.
Institut fuer Pharmazeutische Chemie der Universitaet
Am Hubland, D-8700 Wuerzburg
- Ekundayo, Dr. O.
Chemistry Department, University of Ibadan
Ibadan, Nigeria
- Emberger, Dr. R.
Haarmann & Reimer GmbH
Postfach 138, D-3450 Holzminden

- Formacek, Dr. U.
Bruker Analytische Messtechnik GmbH
Silberstreifen, D-7512 Rheinstetten 4
- Frazao, S.
Division of Natural Products
Estrada das Palmeiras, P-2745 Queiuz de Baixo
- Garnero, Dr. J.
P. Robertet et Cie.
37, Avenue Sidi Brahim, BP 100, F-Grasse Cedex
- Gold, Dr. M.
Merz u. Co.
Eckenheimer Landstr. 100, D-6000 Frankfurt
- Graven, Prof. Dr. E.H.
35 Albert Road, King Williams Town 5600, South Africa, 5
- Grzunov, Dr. K.
Laboratory for Organic Chemistry, Faculty of Technology University
Split, Yugoslavia
- Guenther, W.
WGA
Senta-Weg 16, D-4000 Duesseldorf 30
- Hanssen, Dr. H.-P.
Lehrstuhl fuer Pharmacognosie der Universitaet
Bundesstr. 43, D-2000 Hamburg 13
- Hartschen, R.
c/o Phoenix Laboratorium GmbH
Benzstr. 10, Postfach 20, D-7031 Bondorf
- Hass, Dr. W.
BAT Cigarettenfabriken GmbH
Bahrenfelder Chaussee 139, D-2000 Hamburg 50
- Herres, Dr. W.
Bruker Analytische Messtechnik GmbH
Wikingerstr. 64, D-7500 Karlsruhe 21
- Hethelyi, E.
Research Institute for Medical Plants
2011 Budakalasz, Jozsef A. u. 68. Pf. 11, Hungary
- Hildebrandt, B.
Bionorica GmbH
Peterstr. 33-39, D-8500 Nuernberg 30
- Hiltunen, Prof. Dr. R.
Revontulentie 4E 94, SF-00740 Helsinki 74
- Hirvi, Dr. T.
Technical Research Centre of Finland, Food Research Laboratory
Biologinkuja 1, SF-02150 Espoo
- Hoffmann, Prof. Dr. H.M.R.
Institut fuer Organische Chemie der Universitaet
Schneiderberg 1B, D-3000 Hannover 1
- Hopp, Dr. R.
Haarmann & Reimer
Postfach 138, D-3450 Holzminden
- Hubbert, M.
Institut fuer Pharmazeutische Biologie und Phytochemie der Univ.
Hittdorfstr. 56, D-4400 Muenster
- Huopalathi, Dr. R.
Department of Biochemistry, Laboratory of Food Chemistry, Univ.
SF-20500 Turku
- Huovinen, K.
Helsingin Yvopisto, Farmasian laitos
Fabianinkatu 35, SF-00170 Helsinki
- Ikan, Prof. Dr. R.
Department of Organic Chemistry, Hebrew University
Jerusalem 91904, Israel

- Janssen, A.M.
Gorlaeus Laboratories, Leiden University
P.O. Box 9502, NL-2300 RA Leiden
- Joulain, D.
P. Robertet, Research Laboratories
B.P. 100, F-06333 Grasse Cedex
- Kaiser, R.
Givaudan Research Company Ltd.
Ueberlandstr. 138, CH-8600 Duebendorf
- Karl, Dr. C.
c/o Weleda AG
Postfach 1309/20, D-7070 Schwaebisch Gmuend
- Karlsen, Dr. J.
Institute of Pharmacy, University of Oslo
P.O. Box 1068, N-0316 Oslo 3
- Karner, K.
c/o Curt Georgi GmbH
Talstr. 35-37, D-7030 Boeblingen
- Keller, Dr. K.
Bottroper Weg 10, D-1000 Berlin 27
- Kieslich, Prof. Dr. K.
GBF, Gesellschaft fuer Biotechnologische Forschung mbH
Mascheroder Weg 1, D-3300 Braunschweig
- Klingenberg, A.
Institut fuer Angewandte Botanik, Abt. Pharmakognosie
Bundesstr. 43, D-2000 Hamburg 13
- Kloosterman, J.
Rietzangerstraat 159, NL-3815 ED Amersfoort
- Knobloch, Prof. Dr. K.
Institut fuer Botanik und Pharmazeutische Biologie der Universitaet
Schlossgarten 4, D-8520 Erlangen
- Kobold, U.
Institut fuer Organische Chemie II der Universitaet
Henkestr. 42, D-8520 Erlangen
- Koepsel, Dr. M.
Haarmann & Reimer
Postfach 138, D-3450 Holzminden
- Kortlaender, F.
Baeckerstr. 34a, D-8000 Muenchen 60
- Kubeczka, Prof. Dr. K.-H.
Institut fuer Pharmazeutische Biologie der Universitaet
Mittlerer Dalienbergweg 64, D-8700 Wuerzburg
- Kugler, Dr. E.
Coca-Cola GmbH
Max-Keith-Str. 66, D-4300 Essen
- Kutter, L.
Institut fuer Pharmazeutische Biologie und Phytochemie der Univ.
Hittdorfstr. 45, D-4400 Muenster
- Laakso, J.
Hilapellont. HE 13, SF-00390 Helsinki
- Lamparsky, Dr. D.
Givaudan Research Company Ltd.
Ueberlandstr. 138, CH-8600 Duebendorf
- Lange, Dr. G.
Organische Chemie der Universitaet, Massenspektrometrie
Am Hubland, D-8700 Wuerzburg
- Liddle, Dr. P.A.P.
Martini & Rossi
BP 50, 10 Avenue Michelet, F-93401 Saint-Ouen Cedex

- Martin, J.
Ets. Chevallier
37, Avenue Sidi Brahim, F-06332 Grasse Cedex
- Moattar, Prof. Dr. F.
Department of Pharmacognosie, Faculty of Pharmacy
University of Isfahan, Isfahan - Iran
- Motl, Dr. O.
Institut fuer Organische Chemie und Biochemie der Universitaet
Flemingovo nam. 2, 166 10 Praha 6, Tschechoslowakei
- Moxham, Dr. T.H.
Department of Plant Sciences, University of Bath
Claverton Down, Bath, Avon BA2 7AY, Great Britain
- Mueller, Dr. H.-P.
Dr. Ivo Deiglmayr, Chemische Fabrik Nachf. GmbH & Co.
Baumschulenweg 20, D-2900 Oldenburg
- Muenzing, Dr. H.-P.
DROM, Dr. O. Martens Nachfolger KG
Postfach 1141, D-8021 Baierbrunn
- Ntezurubanza, L.
Gorlaeus Laboratories, University of Leiden
P.O. Box 9502, NL-2300 RA Leiden
- Oberdieck, R.
Raps & Co. Gewuerzwerk
Adalbert-Raps-Str. 1, D-8650 Kulmbach
- Pfeiffer, Dr. W.
c/o Raps & Co. Gewuerzwerk
Adalbert-Raps-Str. 1, D-8650 Kulmbach
- Pohjola, J.
Helsingin Yliopisto, Farmasian laitos
Fabianinkatu 35, SF-00170 Helsinki
- De Pooter, Dr. H.L.
Laboratories of Organic Chemistry, Faculty of Agricultural Sciences
Coupure Links 653, B-9000 Gent
- Protzen, K.-D.
c/o Paul Kaders GmbH
Zippelhaus 5, D-2000 Hamburg 11
- Provatoroff, Dr. N.
Regentesselaan 12, NL-1405 EL Bussum
- Putievsky, Dr. E.
Agricultural Research Organization
Newe Ya'ar, Post Haifa 31-999, Israel
- Ramalho, P.
DRAGOCO Perfumes & Aromas Ltda
Av. Interlagos, 5986, Interlagos, 04777 Sao Paulo, Brasilien
- Rapp, Prof. Dr. A.
Bundesforschungsanstalt fuer Rebenzuechtung
Geilweilerhof, D-6741 Siebeldingen
- Ravid, Dr. U.
Agricultural and Research Organization, Regional Experiment Station
Newe-Ya'ar, Post Haifa, 31999, Israel
- Reichling, Dr. J.
Institut fuer Pharmazeutische Biologie der Universitaet
Im Neuenheimer Feld 364, D-6900 Heidelberg
- Rudolph, P.
Correnstr. 62a, D-4400 Muenster
- Seppaenen, T.
Hilapelout 4E13, SF- 00390 Helsinki
- Sarer, Dr. E.
Faculty of Pharmacy, Ankara University
Tandogan-Ankara, Tuerkei

- v. Schantz, Prof. Dr. M.
Universitaet Helsinki, Pharmazeutisches Institut
Fabianinkatu 35, SF-00170 Helsinki
- Scheffer, Prof. Dr. J.J.C.
Gorlaeus Laboratories, Leiden University
PO Box 9502, NL-2300 RA Leiden
- Schilcher, Prof. Dr. H.
Institut fuer Pharmakognosie und Phytochemie der Universitaet
Koenigin-Luise-Str. 2-4, D-1000 Berlin 33
- Schlegelmilch, F.
FHN
Frankenring 20, D-4150 Krefd
- Schmaus, G.
Institut fuer Pharmazeutische Biologie der Universitaet
Mittlerer Dallenbergweg 64, D-8700 Wuerzburg
- Schmidt, Dr. S.
Am Rautenschemm 9, D-5778 Meschede
- Schneider, Dr. E.
c/o Fa. Salus-Haus
Bahnhofstr. 24, D-8206 Bruckmuehl/Mangfall
- Schubert, Dr. R.
c/o Finnigan MAT GmbH
Barkhausenstr. 2, D-2800 Bremen
- Schultze, Dr. W.
Institut fuer Pharmazeutische Biologie der Universitaet
Mittlerer Dallenbergweg 64, D-8700 Wuerzburg
- Stahl, Prof. Dr. E.
Lehrstuhl fuer Pharmakognosie der Universitaet
Bundesstr. 43, D-2000 Hamburg 13
- Surburg, Dr. H.
Haarmann & Reimer
Postfach 138, D-3450 Holzminden
- Urmeter, E.-M.
c/o Curt Georgi GmbH
Talstr. 35-37, D-7030 Boeblingen
- Verzar-Petri, Prof. Dr. G.
Institut of Pharmacognosy, Semmelweis Medical University
Uelloei ut 26 III, H-1085 Budapest VIII
- Vostrowsky, Dr. O.
Institut fuer Organische Chemie der Universitaet
Henkestr. 42, D-8520 Erlangen
- Vuorela, H.
Helsingin Yliopisto, Farmasian laitos
Fabianinkatu 35, SF-00170 Helsinki
- v.d. Weerd, Dr. A.J.A.
Naarden International, Organic Research Department
PO Box 2, NL-1400 CA Bussum
- Wells, Dr. R.
44 Woolmer Wax, Bordon, Hants. GU35 9QF, Great Britain
- Werkhoff, Dr. P.
Haarmann & Reimer
Postfach 138, D-3450 Holzminden
- Weyerstahl, Prof. Dr. P.
Institut fuer Organische Chemie, Skr. TC 2, Technische Universitaet
Strasse des 17. Juni 122-128, D-1000 Berlin 12
- Whitfield, Dr. F.B.
CSIRO, Division of Food Research
PO Box 52, North Ryde, NSW 2113, Australia
- Wilcken, S.
TAD - Pharmazeutisches Werk GmbH
Postfach 720, D-2190 Cuxhaven 1

Wohland, J.

WGA

Senta-Weg 16, D-4000 Duessedorf 30

Wolf, Prof. Dr. H.

Institut fuer Organische Chemie der Universitaet

Schleinitzstr. 2, D-3300 Braunschweig

Zdero, C.

Institut fuer Organische Chemie der Universitaet, IOC Sekr. C 3

Strasse des 17. Juni 135, D-1000 Berlin 12

Ziegler, E.

Aromachemie, E. Ziegler GmbH

D-8551 Aufsess ueber Forchheim

Ziegler, G.

Aromachemie, E. Ziegler GmbH

D-8551 Aufsess ueber Forchheim

Supplement

- Aye, Dr. W.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Bruhn, Dr. W.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Brunke, Dr. E.-J.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Fahlbusch, Dr. K.-G.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Gruber, H.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Hall, Rüdiger
Haarmann & Reimer
Postfach 138, D-3450 Holzminden
- Hammerschmidt, Dr. F.-J.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Hoppe, K.-D.
Paul Kaders GmbH
Zippelhaus 5, D-2000 Hamburg 11
- Kaphey, Dr. C.-H.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Köster, Dr. F.-H.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Krause, C.
Simmarstigen 10A, SF-00330 Helsinki
- Menzel, Dr.
Fa. Pfizer
Postfach 4949, D-7500 Karlsruhe-1
- Moesdarsono, Dr. M.
Jalan Ganesha 10, Bandung-40163, Indonesia
- Rojahn, W.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden
- Tumbrink, Dr. L.
DRAGOCO Gerberding & Co. GmbH
D-3450 Holzminden

