

The analysis of some South African essential oils
by
comprehensive two-dimensional gas chromatography

THE ANALYSIS OF SOME SOUTH AFRICAN
ESSENTIAL OILS
BY
COMPREHENSIVE TWO-DIMENSIONAL GAS
CHROMATOGRAPHY
(GC×GC)

Submitted in partial fulfilment of the requirements for the degree of

Master of Science, Chemistry

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in the Faculty of Natural and Agricultural Sciences

University of Pretoria

Pretoria

July 2003

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228 401 012.148
11/2003/11/2003/11/2003

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Summary

By virtue of their fragrance and therapeutic nature, essential oils have gained great application in the cosmetic and pharmaceutical industries. These oils, as products consumed by the public for the general health and cosmetics, should be of high quality and unadulterated.

In this project comprehensive two-dimensional gas chromatography (GC×GC) was used for the qualitative analysis of some South African essential oils. The main purpose of the project was to evaluate GC×GC for identification of essential oil constituents and for fingerprinting the different essential oils for quality control purposes. Essential oils of the same kind but of different origin were compared and the quantitative variation between their components was studied.

Gas chromatography–time-of-flight mass spectrometry (GC-TOFMS) as well as comprehensive two-dimensional gas chromatography–time-of-flight mass spectrometry (GC×GC-TOFMS) was used to help identify component peaks separated in the GC×GC instrument.

Once certain precautions are taken and the delicate modulator is correctly optimised, the GC×GC results are reproducible and easy to interpret. Overall, the GC×GC technique utilised showed good promise for quality control of essential oils.

Gaschromatografie-vlugtydmasaspektrometrie (GC-TOFMS) sowel as GC×GC-TOFMS is gebruik vir die vorduidige identifikasie van komponent-peuke wat deur die GC×GC instrument geskei is.

Indien sekere voorsoorgemaatredke getref word en die delikate modulator korrek geoptimeer word, is die GC×GC resultate herhaalbaar en maklik om te interpreteer. Die GC×GC tegniek hou groot belofte in vir die kwaliteitskontrolle van essensiële olië.

Opsomming

Die kosmetiese en farmaseutiese industrieë is baie afhanklik van essensiële olies vanweë hulle reuk en terapeutiese eienskappe. Synde produkte wat deur die publiek gekoop word om skoonheids- en algemene gesondheidsredes, moet hierdie olies eg (onvervals) en van 'n hoë gehalte wees.

In hierdie projek is omvattend-tweedimensionele gaschromatografie (GC×GC) gebruik vir die kwalitatiewe analise van sommige Suid-Afrikaanse essensiële olies. Die hoofdoel van die projek was om die tegniek GC×GC te evalueer vir die identifikasie van essensiële-olie-komponente en vir die vingerafdruk-kartering van verskillende essensiële olies met kwaliteitskontrole as oogmerk. Essensiële olies van dieselfde spesie maar van verskillende oorsprong is vergelyk en die kwantitatiewe variasie tussen hul komponente is bestudeer.

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Optimizing

My earnest appreciation and gratitude goes to all the persons who have been there for me when their indispensable help was needed. To mention a few: my supervisor Prof. E. Rohwer for his excellent guidance and encouragement throughout the project, my co-supervisor Dr. E. Harden for her great contribution in all aspects of the project.

Abbreviations

Dr. S. Bauemeister for her meticulous reading of the manuscript and constructive comments. Mr. Anthony J. Hasset for helping with the GC-TOFMS runs and processing the data. The help from Mr. Alexander Whaley and Mr. Amose Mbele in the language aspect of the writing process is also greatly appreciated.

References

My gratitude also extends to Ms. K. Swanepoel (Dept. of Agri., Conser. and Envir., Lowveld College of Agriculture, Nelspruit), for providing all the essential oil samples, to Leco (Leco (PTY) LTD, USA) for analysing two of the essential oil samples using their GC×GC-TOFMS equipment and Dr. Peter Gorst-Allman from Leco South Africa for processing the data.

Last but not least, the funding from the office of the Eritrean Human Resource Development (EHRD) is sincerely acknowledged.

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- b) Extraction of Essential Oils Using Steam Distillation
- c) Extraction of Essential Oils Using Hydro-Diffusion
- d) Extraction of Essential Oils By Water and Steam Distillation
- e) Extraction of Essential Oils by Cohabitation
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m	Metric number of components in a sample
µm	Micro-metre
min	Minute
ms or msec	Milli-seconds
N	Theoretical plate number
n	Peak capacity
p	Number of single component peaks in a chromatogram
PPG	Polypropylene glycol
PEO	Polystyrene glycol
R	Resolution
%RSD	Percentage relative standard deviation
RTL	Retention time locking
s	Number of visible component peaks in a chromatogram

Abbreviations

Abbreviations

2D	Two-dimensional
CGC	Capillary gas chromatography
cm	Centimetre
D1	First dimension
D2	Second dimension
d_f	Stationary phase film thickness
FID	Flame ionisation detector
GC	Gas chromatography
GC-GC	Heart-cutting gas chromatography
GC×GC	Comprehensive two-dimensional gas chromatography
GC×GC-TOFMS	Comprehensive gas chromatography-time-of-flight mass spectrometry
GC-MS	Gas chromatography-mass spectrometry
GC-TOFMS	Gas chromatography-time-of-flight mass spectrometry
HMW	High molecular weight
I.D.	Internal diameter
kPa	Kilo-Pascal
L	Column length
LC-GC	Liquid chromatography-gas chromatography
LMCS	Longitudinally Modulated Cryogenic System
m	Metre; number of components in a sample
μm	Micro-metre
min	Minute
ms or msec	Milli-seconds
N	Theoretical plate number
n_c	Peak capacity
p	Number of single component peaks in a chromatogram
PPG	Polypropylene glycol
PEG	Polyethylene glycol
R	Resolution
%RSD	Percentage relative standard deviation
RTL	Retention time locking
s	Number of visible component peaks in a chromatogram

sec	Second
SFC	Supercritical fluid chromatography
SFC-GC	Heart-cutting supercritical fluid chromatography-gas chromatography
SFC×GC	Comprehensive supercritical fluid chromatography-gas chromatography
SFE	Supercritical fluid extraction
STD or σ	Standard deviation
TLC	Thin layer chromatography
TDM	On-column thermal desorption modulator
t_m	Dead time
t_R	Retention time
u	Peak widths at base
w_b	Peak width at half height
w_h	Average linear gas velocity

Herbal plants and their essential oils have been used for various purposes since antiquity. The ancient civilisations of Egypt, Rome, Greece, China, and others were known for their use of essential oils as herbal medicines, perfumes, for massaging, as additions to bathing water, and essences were used as offerings to gods and goddesses (e.g. burning of Frankincense in temples) [2].

In modern times, essential oils have gained numerous commercial and industrial applications. They are extensively used as ingredients in the perfumery, pharmaceutical and flavouring industries. The world trade and consumption in essential oils involves huge amounts of essential oils and money. For example, in 1994 alone, US\$ 9 billion worth of flavour and fragrance materials of natural origin were consumed worldwide [3]. Their pleasant fragrance and therapeutic nature have also gained essential oils a wide application in the field of aromatherapy, such as their use as massage oils to relieve different ailments and anxieties or merely as beauty accessories (skin toners). Consequently, many essential oil studies are undertaken which include plant cultivation to increase oil yield, improving oil extraction