

FAST AND EASY GC/MS IDENTIFICATION OF MYRRH RESINS

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Extracts prepared from *Commiphora molmol* resins were analyzed by GC-MS. Twenty-two terpenoid compounds were identified in the hexane extract of the resin. Among them, 2-acetoxifuranodiene (9.80%), furanoeudesma-1,3-diene (8.97%), isofuranogermacrene (6.71%), epicurzerenone (3.64%), 2-methoxyfuranodiene (2.97%), and lindestrane (2.74%) were the main compounds from the first myrrh resin (Tamar Ltd.), and furanoeudesma-1,3-diene (20.59%), isofuranogermacrene (17.94%), 2-acetoxifuranodiene (8.80%), 2-methoxyfuranodiene (7.33%), and lindestrane (6.24%) from the second myrrh resin (Pamir Ltd.).

INTRODUCTION

Commiphora molmol has been used throughout history in incense and as a perfume. Since Biblical times it has been used for the treatment of wounds. The first attempts to identify content compounds were almost 100 years ago. Many compounds identified in *Commiphora* were first found in other sources: (-elemene [1], (-elemene [2–4], (-elemene [5], (-copaene [6–8], (-bourbonene [9], (-selinene [10], germacrene B [11], and germacrene D [12]. Even compounds typical of *Commiphora*, lindestrane [13] and isofuranogermacrene (isogermacrene, curzerene) [14, 15], were previously isolated from *Lindera strychnifolia*.

EXPERIMENTAL

The aim of our study was to identify the main content compounds of a laboratory prepared ethanol extract from *Commiphora molmol* bought from two different suppliers as a certified resins.

Plant material and methods

Commiphora molmol resins bought from Tamar Ltd. and Pamir Ltd. (Israel) were extracted with ethyl alcohol. The prepared extracts were used for analysis.

GC-MS analysis

For qualitative analysis the samples were analyzed by GC-MS in a Hewlett-Packard G1800A GCD system which has a HP-5971 gas chromatograph with electron ionization

detector. An ultralow-bleed 5% phenylpoly(dimethylsiloxane) capillary column (30 m × 0.25 mm i.d. × 0.25 (m film thickness) (SPB-5, Supelco Inc.) was used. For analysis the following method was applied: initial temperature 100(C, initial time 2 min, then temperature programmed from 100 to 280(C at 10(C/min (inlet 250(C; detector 280(C; splitless injection/purge time 1.0 min), helium gas (1 ml/min), solvent delay 4.00 min.

Libraries

For identification of the compounds the NIST/EPA/NIH and Wiley 7th Mass Spectral Libraries and comparison with published spectral data and isolated pure compounds were used. Relative percentage amounts were calculated from GC-MS by the computer.

Results and discussion

All compounds identified in the myrrh resin extracts were previously identified in *Commiphora*. In opoponax oil (also called bisabol myrrh), obtained by steam distillation of the natural oleo-gum-resin from the tree *Commiphora*

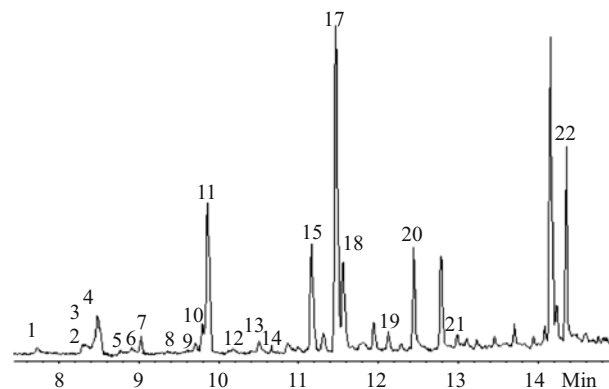


Fig. 1. Gas chromatogram of the hexane extract of *Commiphora molmol* (for peak identification see Table 1).

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TABLE 1. Content compounds identified from two different myrrh resins (see Fig. 1)

Compound	Retention		Percentage	
	time	indices	(Tamar)	(Pamir)
1 δ -elemene	7.76	1340	0.14	0.69
2 α -copaene	8.32	1371	0.33	0.16
3 β -bourbonene	8.46	1379	0.16	0.31
4 β -elemene	8.51	1381	2.01	3.89
5 α -bergamotene	8.79	1394	0.11	nd
6 (+)-aromadendrene	8.94	1402	0.29	nd
7 γ -elemene	9.07	1408	0.86	1.97
8 α -humulene	9.38	1423	0.11	0.06
9 ϵ -macrene D	9.73	1439	0.33	0.17
10 β -selinene	9.83	1444	0.71	0.32
11 isofuranogermacrene	9.95	1449	6.71	17.94
12 δ -cadinene	10.21	1460	0.40	nd
13 elemol	10.54	1474	0.57	nd
14 germacrene B	10.70	1480	0.41	0.36
15 epicurzerenone	11.28	1503	3.64	nd
16 furanoeudesma-1,4-diene	11.45	1509	0.74	nd
17 furanoeudesma-1,3-diene	11.64	1516	8.99	20.59
18 lindenene	11.71	1520	2.74	6.24
19 2-methoxyfuranodiene, isomer I	12.19	1537	0.90	1.17
20 2-methoxyfuranodiene, isomer II	12.53	1549	2.97	7.33
21 2-hydroxyfuranodiene	13.07	1567	1.54	nd
22 2-acetoxymethoxyfuranodiene	14.34	1607	9.80	8.80

erythraea glabrescens Engler, sesquiterpenes, β -elemene, γ -elemene, δ -elemene, and a tricyclic sesquiterpene, α -copaene, were identified [16]. Lindenene, β -bourbonene, isofuranogermacrene, and germacrene D were isolated from the hexane extract of the essential oil of *Commiphora abyssinica* (Berg) Engler with the help of column and preparative chromatography [17]. Column chromatography showed the main compound of the nonpolar fraction of the hexane extract of myrrh, the resin of *Commiphora molmol* Engler, as a new compound, furanoeudesma-1,3-diene [18,19]. From the fractionated essential oil of myrrh, *Commiphora molmol*, several compounds were isolated and identified by column chromatography. One of them was 2-methoxyfuranodiene [20]. From a sample of *Commiphora holtziana* resin collected in Kenya, β -selinene was found (see Scheme 1).

The major compounds which we identified in myrrh resin supplied by Tamar Ltd. were 2-acetoxymethoxyfuranodiene (9.80%), furanoeudesma-1,3-diene (8.97%), isofuranoger-

macrene (6.71%), and epicurzerenone (3.643%), followed by 2-methoxyfuranodiene (2.97%) and lindenene (2.74%). The other main compound in the sample was β -elemene (2.01%) and 2-hydroxyfuranodiene (1.54%). GC/MS identified 44.34% of all the separated compounds identified in myrrh resin (Table 1).

Furanoeudesma-1,3-diene (20.59%), isofuranogermacrene (17.94%), 2-acetoxymethoxyfuranodiene (8.80%), 2-methoxyfuranodiene (7.33%), and lindenene (6.24%) were found as major compounds in the second extract (Pamir Ltd.) (see Table 1).

We have found that the method that we used for analysis is suitable for the fast investigation of extracts from the *Commiphora* samples.

REFERENCES

- G. L. K. Hunter and G. L. Parks, *J. Food Sci.*, **29**, 25–26 (1964).
- D. Kim and H. S. Kim, *J. Org. Chem.*, **52**, 4633–4534 (1987).
- T. Wakamatsu, H. Hara, K. Taira, and Y. Ban, *Heterocycles*, **26**, 1203–1206 (1987).
- M. Kato, H. Kurihara, and A. Yoshikoshi, *J. Chem. Soc. Perkin Trans.*, **1**, 11, 2740–2743 (1979).
- J. H. Gough and M. D. Sutherland, *Aust. J. Chem.*, **17**, 1270–1281 (1964).
- V. H. Kapadia, B. A. Nagasampagi, V. G. Naik, and S. Dev, *Tetrahedron Lett.*, **28**, 1933–1939 (1963).
- P. de Mayo, R. E. Williams, G. Buchi, and S. H. Fearhel, *Tetrahedron*, **21**, 619–627 (1965).
- V. H. Kapadia, B. A. Nagasampagi, V. G. Naik, and S. Dev, *Tetrahedron*, **21**, 607–618 (1965).
- J. Krepinsky, Z. Samek, F. Sorm, et al., *Tetrahedron Suppl.*, **8**, 53–70 (1966).
- W. Cocker and T. B. H. McMurry, *Tetrahedron*, **8**, 181–204 (1960).
- K. Nishimura, N. Shinoda, and Y. Hirose, *Tetrahedron Lett.*, **10**, 3097–3100 (1969).
- K. Yoshihara, Y. Ohta, T. Sakai, and Y. Hirose, *Tetrahedron Lett.*, **27**, 2263–2264 (1969).
- K. Takeda, H. Minato, M. Miyawaki and M. Ishikawa, *Tetrahedron*, **20**, 2655–2663 (1964).
- H. Ishii, T. Tozjo, M. Nakamura, and K. Takeda, *Tetrahedron*, **24**, 625–631 (1968).
- H. Hikino, K. Agatsuma, and T. Takemoto, *Tetrahedron Lett.*, **9**, 2855–2858 (1968).
- J. A. Wenninger and R. L. Yates, *J. Assoc. Off. Anal. Chem.*, **52**, 1155–1161 (1969).
- C. H. Brieskorn and P. Noble, *Planta Med.*, **44**, 87–90 (1982).
- C. H. Brieskorn and P. Noble, *Phytochemistry*, **22**, 187–189 (1983).
- R. A. Wilson and B. D. Mookherjee, *Characterization of Aromatic Donating Components of Myrrh*, 9th Int. Congr. of Essential Oils, Singapore (1983), No. 400.
- C. H. Brieskorn and P. Noble, *Phytochemistry*, **22**, 1207–1211 (1983).
- G. J. Provan, A. I. Gray, and P. G. Waterman, *Flavour Fragrance J.*, **2**, 109–113 (1987).