Phytochemistry, 1971, Vol. 10, pp. 1951 to 1953. Pergamon Press. Printed in England

LABIATAE

COMPOSITION OF THE ESSENTIAL OIL OF AUSTRIAN *MENTHA PULEGIUM*

J. H. ZWAVING and D. SMITH

Laboratory of Pharmacognosy, State University, Ant. Deusinglaan 2, Groningen, The Netherlands

(*Received* 7 *December* 1970)

Abstract—The qualitative and quantitative composition of the essential oil of *Mentha pulegium* L., collected in Austria, was investigated. The main components found were limonene (11%), octyl-3-acetate (0·8%), octanol-3 (1%), menthone (8%), isomenthone (7%) and piperitone (70%). Pulegone, which is usually found in oils of *Mentha pulegium* in quantities varying from 10–90%, was not present in the oil investigated.

INTRODUCTION

DURING investigation into the composition of the essential oils from different species of *Mentha*, one of us collected plants of *M. pulegium* in Austria near Lange Lacke, south-east of Lake Neusiedl (Burgenland). *M. pulegium* L. (= *Pulegium vulgare* Miller) is one of the two European representatives of the subgenus Pulegium (Miller) Lam. et DC.[1] According to Briquet[2] *M. pulegium* belongs to the sectio Eupulegia of the subgenus. Unlike the other species of *Mentha*, *M. pulegium* hybridises rarely with other species, only two reports being found in literature.[3,4]

An extensive investigation into the composition of the essential oil of *Mentha pulegium* was recently made by Hefendehl,[5] and there are several previous reports on this subject.[6–15] In all these investigations it was found that the essential oil of *Mentha pulegium* from different parts of the world always contains pulegone in quantities varying from 10–90%.

[1] G. HEGI, *Illustr. Flora Mitteleuropa*, Bd. V, 4, p. 2339, J. F. Lehmanns Verlag, München (1906).
[2] J. I. BRIQUET, in *Die Natürlichen Pflanzenfamilien* (edited by A. ENGLER and K. PRANTL), IV Teil, Abt. 3a, p. 316, Springer-Verlag, Berlin (1900).
[3] F. ELZE, *Chem. Z.* **43**, 740 (1919); E. GILDEMEISTER and FR. HOFFMANN, *Die Ätherische Öle*, Bd. VII, p. 382, Akademie Verlag, Berlin (1960).
[4] M. J. MURRAY, cited in Ref. 5.
[5] F. W. HEFENDEHL, *Phytochem.* **9**, 1985 (1970).
[6] Y. R. NAVES, *Helv. Chim. Acta* **26**, 162, 172, 1034 (1943).
[7] A. FERNANDES COSTA and J. CARDOSO DO VALE, *Noticias Farm.* **18**, 106 (1952); *Chem. Abstr.* **47**, 6094 (1953).
[8] A. HENRIQUE DE SOUZA, *Rev. Brasil. Farm.* **31**, 257 (1950); *Chem. Abstr.* **47**, 7739 (1953).
[9] R. H. REITSMA, *J. Am. Pharm. Assoc. Sci. Ed.* **47**, 267 (1958).
[10] N. L. GURVICH, ref. *Infraspecific Chemical Taxa of Medicinal Plants* (edited by P. TETENYI), p. 112, Akademiai Kiado, Budapest (1970).
[11] S. KOHLMÜNZER, *Dissertationes Pharm.* **11**, 257 (1959); *Chem. Abstr.* **54**, 16750 (1960).
[12] M. M. CHOPRA, V. N. VASHIST and K. L. HANDA, *Ind. Soap. J.* **30**, 41 (1964); *Chem. Abstr.* **62**, 8928 (1965).
[13] K. L. HANDA, D. M. SMITH, J. C. NIGAM and L. LEVI, *J. Pharm. Sci.* **53**, 1407 (1964).
[14] F. J. SCHNELLE and H. HORSTER, *Planta Med.* **16**, 48 (1968).
[15] O. STICKER and H. FLÜCK, *Pharm. Acta Helv.* **43**, 411 (1968).

RESULTS AND DISCUSSION

The plants were collected in full bloom so they could be properly identified. After air drying the plants were steam distilled giving 0·95% essential oil on a dry weight basis, and the oil was investigated by GLC (Table 1).

TABLE 1. COMPOSITION OF THE ESSENTIAL OIL OF *Mentha pulegium* L.

Peak No.	Substance	%
1–6	Uncharacterized terpenes	1·5
7	Limonene	11·0
8	Unknown	0·2
9	Oct-3-yl-acetate	0·8
10	Octan-3-ol	1·0
11	Menthone	8·0
12	Isomenthone	7·0
13–14	Unknown	0·3
15	Piperitone	70·0
16–17	Unknown	0·2

A number of components (1–6, 8, 13, 14–16 and 17) are present in quantities too small to be properly identified. The other peaks were identified by co-chromatography of known reference substances on two columns and by modified Kovat's indices.[16] Piperitone was trapped from the outlet of the gas chromatograph and identified by its IR spectrum. The most striking result of this investigation is the complete absence of pulegone, which has hitherto been found in the oil from all collections of *M. pulegium*. Naves[6] investigated a number of different collections and found pulegone contents varying from 50–90%. According to him, ecological factors were responsible for the variation in the composition of the essential oil. Hefendehl,[5] on the contrary, found no influence of ecological factors (light and humidity).

The plants used in our investigation were collected in a marshy area, with a special salt vegetation caused by the accumulation of sodium- and magnesium salts in the soil. These ecological factors may have led to the development of a special type of *M. pulegium*. There is one report[17] which describes an oil containing only traces of pulegone originating from *M. pulegium* var. *hirsuta*. This plant clearly differs from the plants investigated by us, by having white hairs on leaf and stalk. The oil of this variety, like our own, contained a large amount of piperitone. Probably these plants accumulate piperitone instead of pulegone. For the sake of completeness the work of Hrimlian,[18] cited by Tetenyi, may be mentioned. He distinguished seven chemical races of *M. pulegium*, but further information about this investigation is lacking.

[16] H. V.D. DOOL and P. D. KRATZ, *J. Chromatog.* **11**, 463 (1963).
[17] V. MORANI, *Ann. Chim. Appl.* **14**, 293 (1924); E. GILDEMEISTER and FR. HOFFMANN, *Die Ätherische Ole*, Bd. VII, p. 255, Akademie Verlag, Berlin (1960).
[18] A. J. HRIMLIAN, Ref. cited in *Infraspecific Chemical Taxa of Medicinal Plants* (edited by P. TETENYI), p. 112, Akademiai Kiado, Budapest (1970).

EXPERIMENTAL

After air drying, the plants were steam-distilled.[19] The oil was trapped in xylene, separated, dried (Na_2SO_4) and injected in the gas chromatograph. The analysis was made using two GLC apparatuses, Hewlett-Packard 700 and Hewlett-Packard 5750.

The columns (2 m × 6 mm) contained Carbowax 20 M and SE 30 respectively. They were programmed from 60–220° with He, or 60–280° with N_2, respectively.

The amount of compounds present in the oil was calculated from the peak areas.

Acknowledgement—The authors wish to thank Prof. F. H. L. van Os for his stimulating interest and valuable discussion.

[19] F. H. L. VAN OS, *Pharm. Weekbl.* **100**, 377 (1965).

Phytochemistry, 1971, Vol. 10, pp. 1953 to 1954. Pergamon Press. Printed in England.

LAURACEAE

PALMITONE AND PHYTOSTEROLS FROM *NEOLITSEA SERICEA*

H. KOMAE and N. HAYASHI

Department of Chemistry, Faculty of General Education, Hiroshima University, Hiroshima, Japan

(*Received* 10 *December* 1970)

Abstract—Palmitone was isolated from the leaves, and phytosterols (β-sitosterol, stigmasterol, campesterol) were detected by gas chromatography from the wood.

Plant. Neolitsea sericea Koidz.

Occurrence. Hiroshima prefecture, Japan.

Previous work. Terpenic constituents of the leaves.[1–4]

Leaves and wood. Crushed to pieces with a chip machine.

Palmitone. Pieces of wood (4·0 kg) were extracted with Et_2O (16 l.) at room temp. for 48 hr. The solvent was concentrated into 100 ml to give white crystalline substance (2·2 g, 0·05% yield). Recrystallization from warm EtOH. m.p. 82–83°. GLC, t_R 14·2 (SE-30 5% on Celite 545 at 260°), only one peak. Mass spectrum (M^+ 450, direct inlet). $C_{31}H_{62}O$. (Found: C, 83·05; H, 13·3, Calc. C, 82·60; H, 13·78%.) Identified by IR, NMR and mass spectra (parent ion 450, major peak 239 ($CH_3(CH_2)_{14}CO^+$), other peaks at 255, 194, 267, 281 and below 100).

[1] S. HAYASHI, N. HAYASHI, T. MATSUURA, *Tetrahedron Letters* 1999, 2647, 4957 (1968).
[2] N. HAYASHI, *J. Sci. Hiroshima Univ.* **33**, 107 (1969).
[3] S. HAYASHI, N. HAYASHI, N. NISHIO, A. MASUDA and T. MATSUURA, *J. Sci. Hiroshima Univ.* **33**, 135 (1969).
[4] K. TAKEDA, *J. Chem. Soc.* (C) 985, 1547 (1970).