Chemical Composition of Volatile Oil from the Roots of *Periploca sepium*

Mitsuo MIYAZAWA^{1*}, Tomoko FUJITA², Chikako YAMAFUJI¹, Masahiro MATSUI³,
Nobuo KASAHARA², Yutaka TAKAGI² and Yukio ISHIKAWA⁴

¹ Department of Applied Chemistry, Faculty of Science and Engineering, Kinki University
(Kowakae, Higashiosaka-shi, Osaka 577-8502, JAPAN)

² Yutaka Pharmacy Co., Ltd.
(Hayashimati, Ogaki, Gifu 503-0015, JAPAN)

³ World One Co., Ltd.
(Hayashimati, Ogaki, Gifu 503-0015, JAPAN)

⁴ Laboratory of Applied Entomology, Graduate School of Agriculture and Life Sciences, University of Tokyo
(Bunkyo-ku, Tokyo, 113-8657, JAPAN)

Edited by J. Sekiya, Kyoto Univ., and accepted May 16, 2004 (received for review April 23, 2004)

Abstract: *Periploca sepium* is used in Chinese crude drugs and widely employed as a tonic. The volatiles obtained by steam distillation (yield, 0.10%) included 4-methoxysalicylaldehyde (87.8%), furfural (3.13%), and lavender lactone (0.77%) as the major components.

Key words: *Periploca sepium*, crude drug, volatile oil, 4-methoxysalicylaldehyde, folk medicine

1 Introduction

Periploca sepium is listed in the Japanese and Chinese Pharmacopoeia and has been widely used as a tonic. As the original plants of this drug, more than seventeen plants are recorded, most of which belong to Araliaceae, but only one, *Periploca sepium* BUNGE belongs to Asclepiadaceae (1). A few Asclepiadaceae plants of Indian origin having therapeutic value were reported (2). The volatile components of several Chinese crude drugs have been investigated in our research on flavour compounds or flavour ingredients (3-9). In earlier papers, various glycosides and 4-methoxysalicylaldehyde have been reported from *P. sepium* (10-13), but so far there is no attempt has been made to study the volatile oil from this plant, has not yet been reported. In this report, the chemical composition of volatile oil from the *P. sepium* was investigated.

2 Experimental

2.1 Material

Commercially available air-dried root bark of *P. sepium* was obtained from matsura kanpo Co., Ltd, (Aichi japan).

2.2 Isolation of Volatile Oil

The root bark of *P. sepium* (200g) was cut into small pieces, hydrodistilled in a Likens-Nickerson type apparatus, with diethyl ether to yield 199 mg, 0.10% of essential oil, which was dried over anhydrous sodium sulphate.

2.3 Gas Chromatography

GC was carried out using Hewlett-packard 5890 equipped with a flame ionization detector (FID) on a capillary column (DB-5, 30 m × 0.25 mm i.d.); the

*Correspondence to: Mitsuo MIYAZAWA, Department of Applied Chemistry, Faculty of Science and Engineering, Kinki University, Kowakae, Higashiosaka-shi, Osaka 577-8502, JAPAN
E-mail: miyazawa@apch.kindai.ac.jp

column temperature was programmed from 60°C to 240°C at a rate of 2°C/min and held at 240°C. The injector and detector temperatures were 270°C and 280°C. The flow rate of the carrier gas (He) was 30 cm/sec.

2·4 Gas-Liquid Chromatography-Mass Spectrometry (GC-MS)

GC-MS was carried out with a Hewlett-Packard 5972 instrument. GC conditions were the same as previously described. The detector interface temperature in the MS source reaching approximately 180°C and the ionization voltage 70 eV. Capillary GC was operated in the computer library and verified by comparison of mass spectra and literature data, and confirmed by GC analyses of authentic samples from previous work (3-9).

3 Results and Discussion

Hydrodistillation gave 0.10% (w/w) essential oil. A gas chromatogram of the volatile oil of *P. sepium* is shown in Fig. 1, from which 57 components were separated. As shown in Table 1, 57 components of oils were identified by direct comparison with authentic samples on the basis of retention time and GC-MS and confirmed by analyses of authentic samples from our previous work (3-9). Around 90% of the volatile oil was

accounted for by a single aromatic principle, viz. 4-methoxysalicylaldehyde, which readily crystallized out as fine needles. Thus identified compounds relative peak area percentages are given in Table 1.

The major constituents were aromatic, which were 4-methoxysalicylaldehyde (87.99%), *p*-anisaldehyde (0.17%), cinnamaldehyde (0.32%), eugenol (0.39%), 2-hydroxy-4-methoxy-acetophenone (0.25%), and anethole (0.22%). The total content of aromatic compounds in the volatile oil was 89.34%.

References

1. J. SHOJI, S. KAWANISHI, S. SAKUMA and S. SHIBATA, Studies on the Chemical Constituents of Chinese Drug "Wuji-api", *Chem. Pharm. Bull.*, Vol. **15**, 720-723 (1967).
2. S. NAGARJAN, L. JAGANA, MOHAN RAO and K.N. GURUDUTT, Chemical Composition of the Volatiles of *Decalepis hamiltonii* (Wight & Arn), *Flavour Fragr. J.*, Vol. **16**, 27-29 (2001).
3. M. MIYAZAWA, Y. MINAMINO and H. KAMEOKA, Volatile Flavor Components of Crude Drugs. Part IX, Volatile Components of the Rhizomes of *Pheum palmatum* L., *J. Essent. Oil Res.*, Vol. **11**, 57-60 (1996).
4. M. MIYAZAWA, Y. MINAMINO and H. KAMEOKA, Volatile Components of *Ephedra sinica* Stapf., *Flavour Fragr. J.*, Vol. **12**, 15-17 (1997).
5. M. MIYAZAWA, Y. MINAMINO and H. KAMEOKA, The Essential Oil of *Erigeron canadensis* L., *Flavour Fragr. J.*, Vol. **4**, 227-230 (1997).

Fig. 1 Gas Chromatogram of the Essential Oil from the Root Bark of *P. sepium*
Column, DB-5 (30m × 0.25mm i.d.); flow rate of the carrier gas, 30 cm/sec; oven temperature: 60-240°C at 2 °C/min, Injector temperature: 270°C, Detector temperature: 280°C

Table 1 Chemical Composition of the Volatiles from the Root of *P. sepium*.

No.	RI ^a	compound ^b	peak area (%)	No.	RI ^a	compound ^b	peak area (%)
1	800	hexanal	tr*	30	1443	vanillin	0.03
2	830	furfural	3.13	31	1446	geranyl acetone	0.11
3	982	2-pentyl furan	0.01	32	1448	α -humulene	0.02
4	1031	limonene	0.17	33	1452	(Z)-methyl isoeugenol	0.02
5	1060	2-acetylpyrrole	0.09	34	1477	<i>ar</i> -curcumene	0.11
6	1071	lavender lactone	0.77	35	1481	β -selinene	0.06
7	1098	linalool	0.04	36	1488	α -selinene	0.02
8	1152	menthone	0.03	37	1492	α -muurolene	0.06
9	1158	nonanol	0.12	38	1503	β -bisabolene	0.06
10	1176	terpinen-4-ol	0.20	39	1512	δ -cadinene	0.05
11	1192	α -terpineol	0.03	40	1515	<i>trans</i> -calamenene	0.04
12	1205	(4Z)-decenal	tr*	41	1534	α -calacolene	0.01
13	1209	myrtenal	tr*	42	1542	elemol	0.03
14	1215	(2Z, 4Z)-nonadienal	tr*	43	1558	geranyl <i>n</i> -butyrate	0.01
15	1216	<i>trans</i> -carveol	tr*	44	1573	caryophyllene oxide	0.10
16	1234	pulegone	0.10	45	1600	hexadecane	0.05
17	1249	<i>p</i> -anis aldehyde	0.17	46	1631	hinesol	0.22
18	1268	(<i>E</i>)-cinnamaldehyde	0.32	47	1645	β -eudesmol	0.23
19	1283	(<i>E</i>)-anethole	0.22	48	1658	<i>ar</i> -turmerone	0.11
20	1292	2-undecanone	0.00	49	1700	heptadecane	0.08
21	1338	4-methoxy-salicyl aldehyde	87.99	50	1800	octadecane	0.12
22	1353	eugenol	0.39	51	1900	nonadecane	0.11
23	1360	γ -nonalactone	0.09	52	1924	methyl palmitate	0.03
24	1382	α -isocomene	0.03	53	2000	eicosane	0.06
25	1386	β -elemene	0.04	54	2088	methyl linoleate	0.02
26	1399	methyl eugenol	0.05	55	2100	heneicosane	0.04
27	1413	β -caryophyllene	0.07	56	2123	linoleic acid	0.07
28	1418	α -ionone	0.01	57	2200	docosane	0.01
29	1433	2-hydroxy-4-methoxy-acetophenone	0.01				

^a Retention index on TC-WAX.^b All components were identified by comparing retention times of GLC and MS with authentic samples.

* trace<0.01%

6. M. MIYAZAWA, Y. MINAMINO and H. KAMEOKA, Volatile Components of the Rhizomes of *Dioscorea japonica*, *Flavour Fragr. J.*, Vol. **9**, 245-248 (1997).
7. M. MIYAZAWA, S. OKAMURA, Y. OKUNO and S. MORII, Components of the Essential Oil of *Cryptotanenia japonica* Hassk "itomitsuba" for Japanese Food, *Flavour Fragr. J.*, Vol. **14**, 273-275 (1999).
8. M. MIYAZAWA, K. KUROSE, A. ITOH, N. HIROOKA and H. KAMEOKA, Components of the Essential Oil from *Glehnia littoralis*, *Flavour Fragr. J.*, Vol. **16**, 215-218 (2001).
9. M. MIYAZAWA, K. KUROSE, A. ITOH and N. HIRAOKA, Comparison of the Essential Oils of *Glehnia littoralis* from Northern and Southern Japan, *J. Agric. Food. Chem.*, Vol. **49**, 5433-5436 (2001).
10. LU GUIBAO and ZHANG MINHE, Determination of 4-methoxysalicylaldehyde by HPLC in Chinese silkvine (*Periploca sepium*), *Zhongcaoyao*, Vol. **19**, 16-18 (1988).
11. HUANG QING, ZHANG HONGYAN, LI YONGEN CHENG GUANGYU, ZHANG BEN and XI HONGSHAN, Determination of 4-methoxysalicylic Aldehyde in the *Periploca sepium* Bge. Bark or Root by Thin Layer Chromatogram Scanner, *Fenxi Huaxue*, Vol. **23**, 236 (1995).
12. Y. OSHIMA, T. HIROTA, H. HIKINO, Periplosides A, B and C, Steroidal Glycosides of *Periploca sepium* Root Barks, *Heterocycles*, Vol. **26**, 2093-2098 (1987).
13. H. ITOKAWA, J. XU and K. TAKEYA, Studies on Chemical Constituents of Antitumor Fraction from *Periploca sepium*. V. Structures of New Pregnane Glycosides, Periplocosides J, K, F and O, *Chem. Pharm. Bull.* Vol. **11**, 4441-4446 (1988).