Table I. Comparison of Residues of Carbaryl Found in Dry Lentil Straw as Determined by the Methanesulfonyl Chloride and the Trichloroacetyl Chloride Methods

			residues found, ppm	
sample	treatment rate, active ingredient per acre	plot no.	methanesulfonyl chloride method	trichloroacetyl chloride method
untreated control	0	6	ND[a]	ND
recovery[b] (1.0 ppm)	0	6	0.97	0.99
			1.05	1.08
recovery (0.1 ppm)	0	6	0.090	0.095
			0.118	0.102
treated samples	1.25	10	1.11	0.88
		4	0.79	0.81
		7	0.12	0.10
		11	11.00	12.90
		5	4.61	4.50
	2.50	8	1.07	1.07
		2	1.29	0.93
		14	0.81	0.75
		1	4.40	4.33
		10	0.07	0.08
		4	0.49	0.53

[a] ND (none detected) indicates that the residues in these samples were below the lower limit of detection for these samples which was 0.05 ppm. [b] Recoveries were prepared by fortifying control samples of straw with pure carbaryl prior to extraction.

its sulfoxide and sulfone metabolites by the mesylate procedure in crops of spinach, peas, celery, rhubarb, and strawberries has been completed and will be reported elsewhere.

Results in Table I show that the residues of carbaryl in dry lentil straw as determined by our methanesulfonyl chloride method and by the trichloroacetyl chloride method of Butler and McDonough (1968) are comparable. The chromatogram of the control sample of lentil straw, in Figure 4, is quite free of interfering peaks. This, generally, is not the case when residues of carbamate pesticide are determined with a method involving the use of the ECD. Also, high background peaks made it difficult to determine low carbaryl residues in lentil straw when the trichloroacetyl chloride method was used.

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Volatile Constituents of the Chestnut Flower

Kenji Yamaguchi and Takayuki Shibamoto*[1]

The volatile constituents of the chestnut flower (*Castanea creata* Sieb et Zucc), which have not been studied prior to this report, have been investigated by gas chromatography–mass spectrometry. Fifty-four compounds were positively identified in the oil which was obtained using a simultaneous distillation–extraction apparatus. The compounds identified include 20 aliphatic compounds, 10 monoterpenes, 1 sesquiterpene, 16 aromatic compounds, and 7 miscelleneous compounds. The main constituents of this oil were 1-phenylethyl alcohol and 2-phenylethyl alcohol, which comprise 44% of this oil.

Castanea grows wild over Europe, Asia, North America, and North Africa. The nuts are edible and very popular. *Castanea creata* Sieb et Zucc (Japanese chestnut) grows wild throughout Japan. The flower (catkin form) blooms in June and possesses a characteristic sweet odor. In this study, the volatile constituents of the chestnut flower were isolated and identified by means of gas chromatography–mass spectrometry.

EXPERIMENTAL SECTION

Taxonomic identification of this plant was made by Professor Masao Arai, Tokyo Agricultural University, Tokyo, Japan. Samples were collected near Konosu City on June 15, 1978, by K. Yamaguchi. Freshly collected flowers were carefully sorted on white paper to remove any kind of foreign materials (barks, twigs, soil, etc.). Five hundred grams of flowers were subjected to simultaneous distillation and extraction (SDE) (Likens and Nickerson, 1964). The extracting solvent was methylene chloride and steam distillation–extraction (water/methylene chloride = 1200 mL/200 mL) was continued for 16 h. The extract was dried over anhydrous sodium sulfate for 12 h and the

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Ogawa & Co., Ltd., 6-32-9 Akabanenishi, Kita-Ku, Tokyo, Japan.

[1] Present address: Department of Environmental Toxicology, University of California, Davis, CA 95616.

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Table I. Compounds Identified in Chestnut Flower

peak no.	compound	peak area %	I_u	I_k
1	octane	0.01	800	800
2	ethyl acetate	0.41	857	858
3	diacetyl	[a]	938	935
4	pentanal	[a]	954	951
5	hexanal	[a]	1054	1053
6	2-methyl-2-vinyl-5-isopropyltetrahydrofuran (tentative)	[a]	1074	
7	2,2,6-trimethyl-6-vinyltetrahydropyran (tentative)	0.16	1095	
8	myrcene	[a]	1137	1137
9	heptanal	0.29	1150	1151
10	α-terpinene	[a]	1157	1157
11	limonene	0.18	1179	1181
12	amyl alcohol	[a]	1191	1192
13	*cis*-ocimene (tentative)	[a]	1200	
14	styrene	[a]	1217	1216
15	*trans*-ocimene (tentative)	0.15	1219	
16	acetoin	[a]	1230	1231
17	*p*-cymene	[a]	1239	1241
18	octanal	0.10	1248	1248
19	terpinolene	0.30	1257	1257
20	hexanol	[a]	1286	1290
21	*cis*-3-hexen-1-ol	0.61	1322	1322
22	*trans*-2-hexen-1-ol	[a]	1343	1340
23	nonanal	2.80	1349	1348
24	*trans*-linalool oxide	0.19	1384	1383
25	furfural	0.91	1393	1394
26	*cis*-linalool oxide	0.25	1409	1410
27	2-acetylfuran	[a]	1435	1434
28	decanal	[a]	1447	1448
29	benzaldehyde	0.11	1456	1456
30	linalool	8.50	1478	1479
31	octanol	[a]	1488	1490
32	5-methylfurfural	[a]	1491	1495
33	unknown $M^+ = 154$ (monoterpene alcohol)	0.51	1534	
34	1-*p*-menthen-9-al (tentative)	0.47	1552	
35	unknown	0.46	1555	
36	phenylacetaldehyde	[a]	1563	1563
37	furfuryl alcohol	[a]	1572	1572
38	acetophenone	3.80	1574	1575
39	nonanol	0.86	1585	1586
40	unknown $M^+ = 154$ (monoterpene alcohol)	0.10	1594	
41	α-terpineol	3.00	1619	1621
42	unknown	0.16	1674	
43	methyl salicylate	[a]	1691	1694
44	α-farnesene (tentative)	0.15	1696	
45	*o*-hydroxyacetophenone	[a]	1709	1714
46	1-phenylethyl alcohol	31.80	1713	1716
47	ethyl salicylate	[a]	1728	1730
48	unknown	[a]	1743	
49	hexanoic acid	[a]	1748	1746
50	geraniol	6.50	1759	1763
51	benzyl alcohol	7.90	1772	1774
52	2-phenylethyl alcohol	12.20	1808	1807
53	unknown	0.10	1821	
54	heptanoic acid	[a]	1846	1850
55	vinylphenylcarbinol (tentative)	0.31	1863	
56	phenol	[a]	1886	1886
57	unknown	[a]	1903	
58	1,8-menthadien-9-ol (tentative)	0.25	1928	
59	3-phenylpropyl alcohol	0.85	1935	1936
60	nerolidol	0.11	1951	1954
61	eugenol	0.28	2045	2040
62	nonanoic acid	0.51	2050	2047
63	2-methoxy-4-vinylphenol (tentative)	0.41	2067	
64	*o*-aminoacetophenone	0.20	2087	2091
65	heneicosane	1.70	2100	2100
66	cinnamyl isobutyrate	0.15	2107	2108
67	β-eudesmol (tentative)	0.76	2113	
68	unknown	0.67	2121	
69	cinnamyl alcohol	3.90	2149	2153
70	docosane	0.11	2200	2200
71	cinnamyl isovalerate	[a]	2210	2210
72	tricosane	1.00	2300	2300

[a] Peak area percent less than 0.01%.

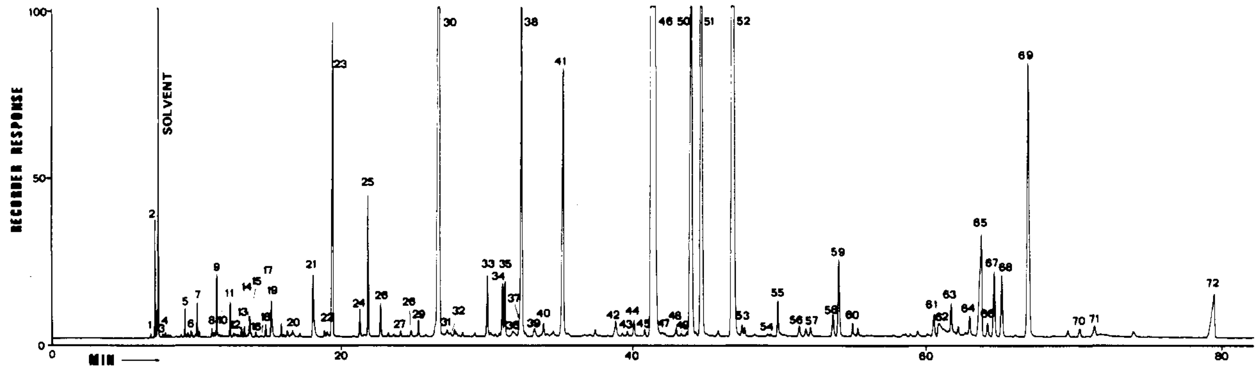

Figure 1. A typical gas chromatogram of the oil of chestnut flower. A Hewlett-Packard Model 5710A gas chromatograph equipped with a flame ionization detector and 50 m × 0.28 mm i.d. glass capillary column coated with PEG Carbowax 20M was used. The column temperature was programmed from 80 to 170 °C at 1 °C/min. Nitrogen carrier gas flow was 0.6 mL/min. The injector and detector temperature were 250 °C. See Table I for peak identification.

solvent was removed using a rotary flash evaporator. Approximately 0.29 g (0.057% relative to the quantity of flowers used) of volatile oil was obtained. This figure includes the residue of the solvent; gas chromatographic peak area percent of solvent is 4.20%. The identification of volatiles was conducted following the GC/MS technique described in Yamaguchi and Shibamoto (1979).

RESULTS AND DISCUSSION

Table I shows the compounds identified in the oil of the chestnut (*Castanea creata* Sieb et Zucc) flower. Peak numbers on the left side show the elution order on the Carbowax 20M column (Figure 1). Those peak areas which had values of less than 0.01% are not listed. I_u designates retention indices of unknowns. I_k represents the retention indices of authentic samples.

Many aromatic alcohols were identified in this oil (total peak area percent is 57.80). A main constituent was 1-phenylethyl alcohol (peak no., 46; peak area percent, 31.80), which has also been found in the fruits of apple (Drawert et al., 1968), cloudberry (Honkanen and Pyysalo, 1976), and grape (Schreier et al., 1976), but never before in ordinary essential oils. 2-Methyoxy-4-vinylphenol (peak no., 63) was also found in cloudberry (Honkanen and Pyysalo, 1976). These aromatic alcohols give a mild floral and balsamic note to this oil. Acetophenone and its derivatives (*o*-hydroxyacetophenone, *o*-aminoacetophenone) were identified as aromatic compounds in addition to the aromatic alcohols. *o*-Aminoacetophenone has been identified in concentrated milk (Arnold and Lindsay, 1969) and beer (Palamand and Grigsby, 1974). The above three acetophenones were also found in the oil of *Castanopsis* flower (Yamaguchi and Shibamoto, 1979). The sweet floral note of this flower is due to these acetophenone derivatives.

Monoterpene alcohols were quantitatively the second largest constituents of the chestnut flower (total peak area percent is 19.20). Linalool and other terpene alcohols give the oil its fresh floral note.

Many aliphatic alcohols, aldehydes, and acids were identified. Nonanal possesses a strong animal note which is a characteristic odor of this flower. Straight chain hydrocarbones (heneicosane, docosane, tricosane) may derive from the wax component of the flower. 2,2,6-Trimethyl-6-vinyltetrahydropyran (peak no. 7) may form from geraniol during steam distillation (Stevens et al., 1972). Some constituents (diacetyl, acetoin, furfural, 5-methylfurfural, acetylfuran, and furfuryl alcohol), which gave a burnt odor, may be artifacts produced by heating during distillation. This oil possesses a strong characteristic animal note and recently has received much attention as it is used to give an animal note to fragrances.

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