

Figure 2. SDS-PAGE of myrosinase purified in a single step by Con A-Sepharose affinity chromatography. Samples were run in 7% gel and stained with Coomassie Brilliant Blue R-250. Key: molecular weight standards (lane 1); myrosinase eluted with glucose (lane 2), mannose (lane 3), methyl  $\alpha$ -D-glucoside (lane 4), methyl  $\alpha$ -D-mannoside (lane 5).

culated molecular weight was ca. 140000.

The myrosinase retained by the Con A-Sepharose gel remained highly active toward its substrates. This observation can be usefully exploited for routine analyses of total glucosinolate content in crude extracts of cruciferous materials by determining the glucose concentration before and after the myrosinase-catalyzed hydrolysis of glucosinolates at room temperature and in a few minutes.

In conclusion, the procedure described here allows the preparation of hundreds of milligrams of pure myrosinase from aqueous crude extract of white mustard seeds in a few days. In view of the simple isolation procedure and the high yields, myrosinase isolated by this method can be used without further purification for glucosinolate analyses, particularly by the above-described polarographic technique (Iori et al., 1983). In addition, such high yields should allow a detailed structural characterization of the protein, improving our knowledge of chemical and biological properties of myrosinase.

# ACKNOWLEDGMENT

We thank Verena Ricci for skillful technical assistance.

**Registry No.** Glucose, 50-99-7; mannose, 3458-28-4; methyl  $\alpha$ -D-glucoside, 97-30-3; methyl  $\alpha$ -D-mannoside, 617-04-9; myrosinase, 9025-38-1.

#### LITERATURE CITED

- Björkman, R.; Janson, J. C. Biochim. Biophys. Acta 1972, 276, 508-518.
- Bio-Rad Laboratories, Bio-Rad Protein Assay 1979, Bulletin 1069 E.G.
- Clandinin, D. R.; Robblee, A. R. Proc. 5th Int. Rapeseed Conf., Malmö 1978, 2, 204-219.
- Croft, A. G. J. Sci. Food Agric. 1979, 30, 417-423.
- Heaney, R. K.; Fenwick, G. R. Z. Pflanzenzüchtg. 1981, 87, 89–95. Iori, R.; Leoni, O.; Palmieri, S. Anal. Biochem. 1983, 134, 195–198. Laemmli, U. K. Nature 1970, 227, 680–685.
- Ohtsuru, M.; Hata, T. Biochim. Biophys. Acta 1979, 567, 384-391.
- Olivieri, A. M.; Leoni, O; Ziliotto, U.; Palmieri, S. Riv. Agron. 1982, XVI, 43-46.
- Palmieri, S.; Leoni, O.; Iori, R. Anal. Biochem. 1982, 123, 320–324. Thies, W. L. Fette, Seifen, Anstrichm. 1976, 78, 231–234.
- Thomke, S. J. Am. Oil Chem. Soc. 1981, 58, 805-810.
- Underhill, E. W.; Kirkland, D. F. J. Chromatogr. 1971, 57, 47-54.
- Wetter, L. R.; Youngs, G. C. J. Am. Oil Chem. Soc. 1976, 53, 162-164.

Received for review April 22, 1985. Accepted September 25, 1985. This work was performed as a part of the project for yield and quality improvement of the oleaginous plants supported by the Italian Ministry of Agriculture.

# Volatile Components of Salted and Pickled Prunes (*Prunus mume* Sieb. et Zucc.)

Chu-Chin Chen,\* May-Chien Kuo, Su-Er Liu, and Chung-May Wu

Volatile components of salted and pickled prunes (*Prunus mume* Sieb. et Zucc.) were obtained by extraction and steam distillation of the prunes at pH 2.5 and 7.0. The volatile components were analyzed by capillary gas chromatography (GC) and identified by combined gas chromatography-mass spectrometry (GC-MS). There were 181 compounds detected by GC analyses, 92 compounds identified by GC-MS analyses, and some tentatively identified isomers of naphthalene and benzene derivatives. Aromatic compounds, monoterpenes, monoterpene alcohols, acids, and some aliphatic aldehydes and alcohols were the major components extracted at pH 2.5 of the salted and pickled prunes. The amount of most components extracted at pH 7.0 was less than those extracted at pH 2.5. Components that could be present in the salted and pickled prunes as glycosides were benzyl alcohol, 2-phenylethanol, monoterpene alcohols, linalool oxides and the tentatively identified isomers of (trimethylphenyl)but-3-en-2-one and a decahydronaphthol-like compound.

#### INTRODUCTION

The fruit of *Prunus mume* Sieb. et Zucc. is originally grown in the central and southern regions of China and

is harvested in the late spring. The average weight of a fruit is about 9–14 g. The fruit is used to make wines, beverages, and pickles, but the usual metod to preserve this fruit is to make salted and pickled prunes.

Salted and pickled prunes (*P. mume* Sieb. et Zucc.) is a traditional food and has been consumed in China and Japan over 1000 years. The processing of fresh fruit of *P. mume* Sieb. et Zucc. under high salinity (ca. 20%) has been the traditional practice in order to obtain good flavor and inhibit the microbial growth (Lee et al., 1984). The "sour"

Food Industry Research and Development Institute (FIRDI), Hsinchu, Taiwan, Republic of China (M.-C.K., S.-E.L., C.-M.W.), and Department of Food Science, Cook College, Rutgers, The State University of New Jersey, New Brunswick, New Jersey 08903 (C.-C.C.).

flavor and taste of this food makes it a palatable food or food ingredient in the daily diet of Chinese and Japanese.

Kameoka and Kitagawa (1976) investigated the volatiles from the fresh fruit of P. mume Sieb. et Zucc. and identified 30 volatile components; among these, benzaldehyde, benzoic acid, ethyl benzoate, benzyl alcohol, 5-methyl-2furfural, and 2,3-dimethylmaleic anhydride were the characteristic components.

The volatile components of salted and pickled prunes (umezuke) and the dried products of salted and pickled prunes (umeboshi) were analyzed by Kameoka et al. (1981). Of the 36 volatile components identified, the major components of salted and pickled prunes (umezuke) were furfural, 5-methyl-2-furfural, 2,3-dimethylmaleic anhydride, benzyl alcohol, 2-phenylethyl alcohol, and ethyl palmitate. The major components of dried products of salted and pickled prunes (umeboshi) were benzyl alcohol and methyl palmitate.

Recently, in a series reports, Williams et al. (1980, 1982) and Wilson et al. (1984) found that most of the monoterpene alcohols in the volatiles of grape may originate from the nonvolatile monoterpene glycosides by enzymic or acidic hydrolyses. Other volatile components of grape that could be originated from nonvolatile precursors include 2-phenylethanol, benzyl alcohol, damascenone, vitispirane, and 1,1,6-trimethyl-1,2-dihydronaphthalene (Williams et al., 1982). The glycosides of benzyl alcohol and 2-phenylethanol were further identified by Williams et al. (1983). Similar phenomena about the glycosides bound nature of volatile components were also observed in tea shoots (Takeo, 1981), passion fruit (Engel and Tressl, 1983), and papaya (Heidlas et al., 1984).

From the above mentioned reports, it is possible that, in fruit or in plant, a pool of nonvolatile glycosides exists that can be transformed into volatile compounds either by the action of enzymes or by acid hydrolyses at elevated temperature.

This study presents the investigation about the volatile components of salted and pickled prunes (P. mume Sieb. et Zucc.) at pH 2.5 and 7.0, respectively, and by comparing the differences between the two treatments, the glycosidically bound nature of some volatile components can be shown.

#### EXPERIMENTAL SECTION

Sample Preparation. Fresh fruits of P. mume Sieb. et Zucc. were purchased from Sui-Li, Taiwan. The fruits were washed and then placed in a ceramic vessel with one layer of refined salt (99.5%) over one layer of fruit. The salt concentration in the final product was about 20% (w/w). The pickling process was conducted under room temperature for 8 weeks (Lee et al., 1984). The prunes were deseeded manually, and 1 kg was blended with 2 kg of distilled water for 1 min in a Waring blender. The pH of this preparation was 2.5. The pH of another preparation was adjusted to 7.0 by adding 0.5 N NaOH solution after blending. The volatile components of each preparation were extracted for 2 h in a Likens-Nickerson apparatus (Römer and Renner, 1974). Glass-distilled pentane and ether (1:1) were used as extracting solvent. *n*-Nonanol (2.77 mg; Haarman and Reimer GmbH, West Germany) was added as an internal standard. The extracted volatiles were concentrated to minimal volume by distillation in a Vigreaux column (50 cm  $\times$  2 cm i.d.) before injection.

Gas Chromatography. Gas chromatography was carried out on a Gasukuro Model 370 gas chromatograph (Gasukuro Kogyo, Japan), equipped with a flame ionization detector and a 50 m  $\times$  0.32 mm (i.d.) fused silica capillary column (Chrompack, Middelburg, The Nether-

lands); the stationary phase was equivalent to bonded Carbowax-20M (CP WAX 57 CB). The oven temperature was programmed linearly from 50 to 200 °C at 2 °C/min and then held at 200 °C for 40 min. The injector and detector temperatures were 250 °C. The samples were injected in the split mode with a split ratio of 1:60. The carrier gas was hydrogen at a flow rate of 1.2 mL/min. Quantitative determinations (without consideration of FID response factors, i.e., calibration factors F = 1.00 for all components) were carried out by using a Hewlett-Packard 3390A laboratory integrator.

The linear retention indices of the volatile components were calculated by using *n*-paraffin ( $C_8-C_{25}$ ; Alltech Associates) as references (Majlát et al., 1974).

Capillary Gas Chromatography-Mass Spectrometry. The capillary gas chromatography-mass spectrometry was carried out on a Hewlett-Packard 5985B system. The gas chromatograph (Hewlett-Packard 5840A) was installed with a Quadrex (Quadrex Co., New Haven, CT) fused silica capillary column (bonded Carbowax-20M; 50 m  $\times$  0.32 mm (i.d.)). The conditions were as follow: temperature program, 5 min isothermal at 50 °C, 50–200 °C, 2 °C/min, 40 min isothermal at 200 °C; carrier gas, helium; flow rate, 1.2 mL/min; ion source temperature, 200 °C; electron voltage, 70 eV; electron multiplier voltage, 2400 V.

#### **RESULTS AND DISCUSSION**

Figure 1 shows the gas chromatograms of volatile components of salted and pickled prunes obtained at pH 7.0 (A) and pH 2.5 (B). The fruit pulp materials after separation of the seeds were simultaneously steam distilled and solvent extracted (Römer and Renner, 1974); internal standard had been added before extraction. There are 181 compounds that can be detected in the gas chromatograms.

Table I lists 114 volatile components of salted and pickled prunes, among these, 92 components were identified by comparing the mass spectra of the compounds with the published mass spectral data (MSDC, 1974; Kameoka and Kitagawa, 1976; EPA/NIH, 1980; Jennings and Shibamoto, 1980; Kameoka et al., 1981; TNO, 1981); the other 22 components were tentatively identified according to the mass spectral fragmentations.

Of the volatile components identified in this study, aromatic compounds, monoterpenes, monoterpene alcohols, acids, and some aliphatic aldehydes (saturated and unsaturated) and alcohols are the major components of the pH 2.5 sample. The amounts of components listed in Table I are in most cases less than those of the pH 2.5 sample except peak 3 (ethyl alcohol), peak 71 (methyl benzoate), peak 141 (*p*-methoxyacetophenone), and peak 155 (bis(*p*-methylbenzyl) ether).

When the composition of volatile components identified in this study is compared with previous reports about the volatile components in fresh fruits (Kameoka and Kitagawa, 1976) and in salted and pickled prunes (Kameoka et al., 1981), it can be seen that, besides the characteristic components of salted and pickled prunes, the volatile components isolated in this study also have the characteristics of fresh fruit.

In Table I, aromatic compounds such as benzaldehyde (peak 58) and benzyl alcohol (peak 107) are the two most abundant components of both treatments, and this agrees well with the intensive "almondlike" aroma of both flavor isolates as perceived organoleptically during the preparations.

In this study, the acids (peaks 46, 69-1, 76, 91, 106, 115, 128, 145, 153, 158, 161, 173, and 180) isolated in the pH 2.5 sample are important contributors to the "sour" character of the prunes; however, all these acidic compo-

# Table I. Volatile Components of Salted and Pickled Prunes Identified at pH 7.0 and 2.5

		$L(\mathbf{CW})$	wt,° n	ng/kg	mass
neek no a	component	20M)b	<b>pH</b> 7.0	pH 2.5	spec ref <sup>d</sup>
		954	0.02	0.97	0.0-0
2	ethyl acetate	024	0.02	0.27	a, c=e
3	ethyl alcohol	929	0.20	1.05	а, с-е а
9	hexanal	1009	0.39	0.05	u, t-e
11	camphene	1193	0.11	0.20	u, c-e
13	1-butanol	1101	0.00	1 97	u, t e
14	β-pinene	1133	0.30	0.96	u, c, e
15	myrcene	1149	0.07	0.20	u, c, e
17	heptanal	11/1	0.17	0.33	a, c, e
17-1	limonene	1180		0.02	a, c=e
18	1,4-cineol	1100	0.00	0.11	<i>a</i> , <i>c</i> , <i>e</i>
19	isoamylalcohol	1196	0.02	0.09	a, c-e
21	2-pentylfuran	1217	0.14	0.35	a, c-e
22	1,8-cineol	1226	0.15	0.56	a, c, e
23	ocimene	1232	0.01	0.03	a, c, e
24	amyl alcohol	1236	0.01	0.40	a, c-e
25	hexyl formate	1256	0.09	0.20	a, c-e
27	$\gamma$ -terpinene	1266	0.06	0.06	a, c, e
28	octanal	1274	0.01	0.06	a, c, e
30	trans-2-heptanal	1287	0.01	0.11	c, e
32	1-hexen-3-ol	1304	0.01	0.01	a, c, e
36	isomer of tetrahydrotrimethylnaphthalene $[M^+/e \ 174, 159 \ (100), 174 \ (23), 144 \ (18), 129 \ (16), 131 \ (16), 91 \ (12), 39 \ (10)]^{i}$	1325	-	0.04	
37	1-hexanol	1343	0.04	0.37	a, c-e
39	nonanal	1378	0.12	1.14	a, c-e
40	isophorone	1386	0.01	0.08	с, е
42	butyl hexanoate	1395	0.02	0.15	a, c, e
43	hexyl butanoate	1398	0.02	0.08	a, c, e
43-1	isomer of tetrahydrotrimethylnaphthalene $(M^+/e \ 174)^{f}$	1405		0.08	
44	trans-2-octenal	1417	0.07	0.56	с, е
44-1	ethyl octanoate	1419	+8	0.05	a, c-e
44-2	4-propyl-1-methylbenzene	1422		0.03	с
45	$c_{is-linalool}$ oxide + isomer of tetrahydrotrimethylnaphthalene $(M^+/e \ 174)^f$	1428	0.11	0.39	c-e
45-1	l-octen-3-ol	1438	0.01	0.05	a, c-e
45-2	isomer of 24-heptadienal <sup>f</sup>	1451	-	+	
46	acatic acid	1455	-	0.85	a, c
40	trans-linalool oxide	1457	0.11	1.66	c-e
48		1460	0.09	2.58	a, c-e
59	trans-	1481	0.01	0.29	a, c, e
52	and a baranti butancata	1485	+	0.03	a. c. e
55		1495	0.06	0.49	a. c-e
50		1514	30.20	84.23	b. c-e
50		1519	0.02	0.34	с. е
60	$\frac{1}{2} \frac{1}{2} \frac{1}$	1534	_	+	-, -
64		1544	+	0.18	c-e
64		1563	+	0.06	b-d
00	5-methyl-2-turfulai	1569	+	0.75	c.d
67	methyl 2-furdate	1577	+	0.09	с, ц
68	4-nydroxy-3-methyl-2-(2-propenyl)-2-cyclopenten-1-one	1589	+	0.04	а <i>-</i> е
69	nexyl nexanoate	1592	-	0.02	ac
69-1	propionic acid	1597	_	0.01	u, t
69-2	(21), 41 (20), 55 (19), 119 (8), 136 (6), 152 (6)] <sup><math>f</math></sup>	1007		0.01	
70	phellandral	1603	0.02	0.08	e
71	methyl benzoate	1608	0.56	0.26	c−e
76	butanoic acid	1627	-	0.37	a, c
76-1	phenylacetaldehyde	1635	-	0.02	a, c, e
77	$\overline{eta}$ -terpineol	1640	0.03	0.04	a, c–e
78	1-nonanol	1645	2.77	2.77	а, с-е
79	ethyl benzoate	1651	0.10	0.16	c-e
81	methyl undecanoate + isomer of tetrahydrotrimethylnaphthalene $(M^+/e \ 174)^r$	1665	0.04	8.13	a, c
82	benzyl formate	1671	0.01	0.18	c-e
83	isomer of decahydronaphthol-like compd $(M^+/e \ 152?)^f$	1673	0.04	0.16	
84	a-terpineol	1680	0.04	0.31	а, с-е
85	isomer of $\alpha$ -terpineol <sup><math>\prime</math></sup>	1683	+	0.08	
88	benzyl acetate	1710	0.03	0.23	c-e
89	2,3-dimethylmaleic anhydride	1714	-	0.06	b, c
90	isomer of dihydrotrimethylnaphthalene [ <i>M</i> <sup>+</sup> / <i>e</i> 172, 157 (100), 142 (52), 170 (30), 141 (22), 115 (15), 128 (12), 91 (8)] <sup><i>t</i></sup>	1720	+	0.03	
91	valeric acid	1729	-	0.19	a, c
91-1	4-ethyl-2.6-dimethyl-4-heptanol	1733	+	0.06	с
Q1_9	trans cis-2 4-decadienal	1740	-	+	a, c
01-2 09	methyl salicylate	1753	0.07	5.25	с-е
02 02	isomer of decabydronaphtbaol-like compd $(M^+/e, 152?)^{t}$	1762	-	0.02	
95 05	nero]	1773	+	0.03	c-e
90 07	trans trans-2.4-decadienal	1780	0.02	0.13	a, c
91	benzyl propionate + isomer of decabydronaphthol-like compd $(M^+/e\ 152?)^f$	1783	0.04	0.06	c, e
50	sounds be obtained a record and an analysis and sound a first to the second state of t				

#### Table I (Continued)

		$I_{\rm F}(\rm CW)$	wt,  n	ng/kg	mass
peak no.ª	component	20 <b>M</b> ) <sup>b</sup>	pH 7.0	pH 2.5	spec ref <sup>d</sup>
99	1-phenylethanol	1791	+	0.13	a, c, e
100	isomer of dihydrotrimethylnaphthalene $(M^+/e \ 172)^f$	1793	-	0.03	
101	isomer of tetrahydrotrimethylnaphthalene $(M^+/e \ 174)^f$	1797	-	0.02	
105	geraniol	1823	0.04	0.22	c-e
106	hexanoic acid	1831	-	0.90	<b>a</b> , c
107	benzyl alcohol	1862	1.84	11.79	c-e
110	isomer of dihydrotrimethylnaphthalene $(M^+/e \ 172)^{t}$	1880	-	0.06	
111	γ-octalactone	1883	+	0.07	c−e
112	2-phenylethanol	1896	+	0.06	a, c, e
113	<i>β</i> -ionone	1911	0.12	0.21	c-e
115	heptanoic acid	1939	-	0.06	a, c
121	diethylene glycol	1968	0.01	0.58	a, c
125	isomer of dihydrotrimethylnaphthalene $(M^+/e \ 172)^{t}$	2009	+	+	
126	cinnamic aldehyde	2017	0.02	0.16	с-е
127	2-methyl-5-phenyl-2-hexenal	2029	0.03	0.04	a
128	octanoic acid	2035	-	0.09	a, c
128-1	ethylphenol	2054	0.01	0.03	с
129	4- $(2',3',6'$ -trimethylphenyl)but-3-en-2-one $(M^+/e \ 188)^f$	2078	+	0.21	
134	$\gamma$ -decalactone	2110	0.07	0.09	с, е
135	conifervl alcohol	2114	0.02	0.09	c
136	eugenol	2140	0.02	1.81	с-е
137	1.2-dicyclohexen-1-yl-1,2-ethanediol	2147	+	0.03	с
140	methyl hexadecanoate	2171	+	0.34	a, c, e
141	<i>p</i> -methoxyacetophenone	2173	0.77	+	с
142	ethyl hexadecanoate + isomer of dihydrotrimethylnaphthalene $(M^+/e \ 172)^{\prime}$	2205	0.05	0.26	a, c
145	decanoic acid	2244	-	0.51	a, c
146	isomer of 4-(trimethylphenyl)but-3-en-2-one $(M^+/e \ 188)^f$	2264	+	0.01	
147	2.6.6-trimethyl-3-cyclohexylidene lactone	2323	0.01	0.08	а
148	isoeugenol	2332	0.01	0.34	a, c, e
150	terephthaldehyde	2347	0.03	0.11	a, c
153	undecanoic acid	2379	-	0.18	a, c
155	bis(methylbenzyl) ethyl [ $M^+/e$ 226?, 120 (100), 91 (34), 119 (24), 121 (10), 65 (8), 51 (7), 39 (7)] <sup>f</sup>	2416	1.20	0.34	
156	methyl octadecanoate	2424	+	0.18	a, c
158	benzoic acid	2486	-	5.80	b, c
160	an alkenal compd <sup>f</sup>	2554	0.06	0.29	
161	lauric acid	2575	-	2.38	a, c
162	an alkenal compd <sup>f</sup>	_h	0.11	0.45	,
173	myristic acid	h	-	2.18	a, c, e
180	palmitic acid	_h	3.37	23.38	a, c
	•				

<sup>a</sup> Number refers to Figure 1. <sup>b</sup> Calculated Kovats retention indices. <sup>c</sup> Average of two experiments. <sup>d</sup> Mass spectral data from the following sources: (a) MSDC, 1974; (b) Kameoka and Kitagawa, 1976; (c) EPA/NIH, 1980; (d) Jennings and Shibamoto, 1980; (e) TNO, 1981. <sup>e</sup> Does not exist. <sup>f</sup> Tentatively identified. <sup>g</sup> Amount less than 0.01 mg/kg. <sup>h</sup> Index greater than 2600.

nents were neutralized by the alkali solution in the pH 7.0 sample except some minor amount of palmitic acid (peak 180).

It is interesting to note that there are four groups of tentatively identified compounds, although their concentrations are low, that repeatedly appear in the gas chromatograms of pH 2.5 sample: the isomers of tetrahydrotrimethylnaphthalene,  $M^+/e$  174 (peaks 36, 43-1, 45, 62, 81, and 101); the isomer of dihydrotrimethylnaphthalene,  $M^+/e$  172 (peaks 90, 100, 110, 125, and 142); the isomer of a decahydronaphthol-like compound, possible  $M^+/e$  152 (peaks 69-2, 83, 93, and 98); the isomers of 4-(trimethylphenyl)but-3-en-2-one,  $M^+/e$  188 (peaks 129 and 146). Because of the low concentrations of these compounds, some of these compounds were confirmed by the single-ion monitor (SIM) method of the GC-MS system used in this study. The SIM method is especially useful for the detection of minor component with characteristic mass spectra, even if it is hidden beneath a larger component of the gas chromatogram (Chien, 1984).

To isolate the nonvolatile glycosides or "flavor precursors" in grape and passion fruit, a reversed-phase adsorbent was used to absorb these glycosides directly from the juice (Williams et al., 1982; Engel and Tressl, 1983). Following the acidic hydrolyses (at pH 3.0 or pH 1.0) of the glycosidic fraction under elevated temperature, the corresponding volatile components can be liberated. In this study, the reverse adsorbent was not adopted to isolate the nonvolatile glycosides; instead, steam distillation at different pH was adopted. According to the results of Engel and Tressl (1983), steam distillation at acidic pH (in the case of passion fruit, pH 3.0) could enhance the amount of monoterpene alcohols from 30-fold up to 50-fold as compared with steam distillation at pH 7.0.

Since the nonvolatile glycosides are susceptible to the acidic hydrolyses under elevated temperature, in this study, steam distillation at pH 2.5 did form many categories of compounds that were only minor components or did not exist in the pH 7.0 sample. In Table I, compounds that have been shown previously to be glycosidically bound (Williams et al., 1980, 1981, 1982, 1983; Engel and Tressl, 1983) and are possibly glycosidically bound in this study are monoterpene alcohols (peaks 84, 95 and 105), linalool oxides (peaks 45 and 47), benzyl alcohol (peak 107), and 2-phenylethanol (peak 112). Other compounds that may be glycosidically bound and are tentatively identified in this study include isomers of a decahydronaphthol-like compound (peaks 69-2, 83, 93, and 98) and the isomers of 4-(trimethylphenyl)but-3-en-2-one (peaks 129 and 146).

Besides the above mentioned compounds, there are two groups of naphthalene derivatives that are repeatedly appearing in the pH 2.5 sample, that is the isomers of dihydrotrimethylnaphthalene and the isomers of tetrahydrotrimethylnaphthalene. It is possible that these two groups of compounds are also originating from some nonvolatile precursors by the action of thermal cleavage under acidic pH. It is worthwhile to note 1,2-dihydro-1,1,6-trimethylnaphthalene, an isomer of dihydrotri-



Figure 1. Fused silica capillary gas chromatographic separations of volatiles obtained from salted and pickled prunes by distillation-extraction at pH 7.0 (A) and pH 2.5 (B). Further conditions are given under the Experimental Section. Peak numbers correspond to those in Table I.

methylnaphthalene, which was first identified in strawberry by Stoltz et al. (1970) and then claimed to be originated from nonvolatile precursor in grape by Williams et al. (1982). However, the exact relationship between the precursor and this compound is not clear yet.

## ACKNOWLEDGMENT

We thank Fwu-Ling Lee of FIRDI for the supply of salted and pickled prunes. This research was supported in part by the Council for Agricultural Planning and Development (Grant CAPD-73A-4.1-B-178(4)) of the Republic of China.

Registry No. EtOAc, 141-78-6; EtOH, 64-17-5; Me(CH<sub>2</sub>)<sub>4</sub>CHO, 66-25-1; Me(CH<sub>2</sub>)<sub>3</sub>OH, 71-36-3; Me(CH<sub>2</sub>)<sub>5</sub>CHO, 111-71-7; Me<sub>2</sub>CH(CH<sub>2</sub>)<sub>2</sub>OH, 123-51-3; Me(CH<sub>2</sub>)<sub>4</sub>OH, 71-41-0; HCO<sub>2</sub>-(CH<sub>2</sub>)<sub>5</sub>Me, 629-33-4; Me(CH<sub>2</sub>)<sub>6</sub>CHO, 124-13-0; (E)-Me-(CH<sub>2</sub>)<sub>3</sub>CH=CHCHO, 18829-55-5; CH<sub>2</sub>=CHCH(OH)(CH<sub>2</sub>)<sub>2</sub>Me, 4798-44-1; Me(CH<sub>2</sub>)<sub>5</sub>OH, 111-27-3; Me(CH<sub>2</sub>)<sub>7</sub>CHO, 124-19-6; Me(CH<sub>2</sub>)<sub>4</sub>CO<sub>2</sub>Bu, 626-82-4; Me(CH<sub>2</sub>)<sub>2</sub>CO<sub>2</sub>(CH<sub>2</sub>)<sub>5</sub>Me, 2639-63-6; (E)-CHOCH=CH(CH<sub>2</sub>)<sub>4</sub>Me, 2548-87-0; Me(CH<sub>2</sub>)<sub>6</sub>CO<sub>2</sub>Et, 106-32-1; p-MeC<sub>6</sub>H<sub>4</sub>Pr, 1074-55-1; CH<sub>2</sub>=CHCH(ÕH)(ČH<sub>2</sub>)<sub>4</sub>Me, 3391-86-4; HOAc, 64-19-7; (E,E)-CHO(CH=CH)<sub>2</sub>Et, 4313-03-5; (Z)-Me(CH<sub>2</sub>)<sub>2</sub>CO<sub>2</sub>(CH<sub>2</sub>)<sub>2</sub>CH=CHEt, 16491-36-4; PhCHO, 100-52-7; (E)- $CHOCH = CH(CH_2)_6Me$ , 3913-81-3;  $HO(CH_2)_7Me$ , 111-87-5;  $Me(CH_2)_4CO_2(CH_2)_5Me$ , 6378-65-0;  $MeCH_2CO_2H$ , 79-09-4;  $PhCO_2Me$ , 93-58-3;  $PrCO_2H$ , 107-92-6;  $PhCH_2CHO$ , 122-78-1; HO(CH<sub>2</sub>)<sub>8</sub>Me, 143-08-8; PhCO<sub>2</sub>Et, 93-89-0; Me(CH<sub>2</sub>)<sub>9</sub>CO<sub>2</sub>Me, 1731-86-8; HCO<sub>2</sub>CH<sub>2</sub>Ph, 104-57-4; AcOCH<sub>2</sub>Ph, 140-11-4; BuCO<sub>2</sub>H, 109-52-4; (Me<sub>2</sub>CHCH<sub>2</sub>)<sub>2</sub>C(OH)Et, 54460-99-0; (E,Z)-CHO(CH= CH)<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>Me, 25152-83-4; (E,E)-CHO(CH=CH)<sub>2</sub>(CH<sub>2</sub>)<sub>4</sub>Me, 25152-84-5; MeCH<sub>2</sub>CO<sub>2</sub>CH<sub>2</sub>Ph, 122-63-4; MeCH(OH)(Ph), 98-85-1; Me(CH<sub>2</sub>)<sub>4</sub>CO<sub>2</sub>H, 142-62-1; PhCH<sub>2</sub>OH, 100-51-6; Ph(CH<sub>2</sub>)<sub>2</sub>OH, 60-12-8; Me(CH<sub>2</sub>)<sub>5</sub>CO<sub>2</sub>H, 111-14-8; PhCH=CHCHO, 104-55-2; CHOC(Me)=CHCH2CH(Ph)Me, 99532-37-3; Me(CH2),CO2H, 124-07-2; EtC<sub>6</sub>H<sub>4</sub>OH, 25429-37-2; Me(CH<sub>2</sub>)<sub>14</sub>CO<sub>2</sub>Me, 112-39-0; p-MeOC<sub>6</sub>H<sub>4</sub>Ac, 100-06-1; Me(CH<sub>2</sub>)<sub>14</sub>CO<sub>2</sub>Et, 628-97-7; Me-334-48-5;  $Me(CH_2)_9CO_2H$ , 112-37-8;  $(CH_2)_8CO_2H$ ,  $(MeC_6H_4CH_2)_2O$ , 38460-98-9;  $Me(CH_2)_{16}CO_2Me$ , 112-61-8; PhCO<sub>2</sub>H, 65-85-0; Me(CH<sub>2</sub>)<sub>10</sub>CO<sub>2</sub>H, 143-07-7; Me(CH<sub>2</sub>)<sub>12</sub>CO<sub>2</sub>H, 544-63-8; Me(CH<sub>2</sub>)<sub>14</sub>CO<sub>2</sub>H, 57-10-3; camphene, 79-92-5; β-pinene, 127-91-3; myrcene, 123-35-3; limonene, 138-86-3; 1,4-cineol, 470-67-7; 2-pentylfuran, 3777-69-3; 1,8-cineol, 470-82-6; ocimene, 13877-91-3;  $\gamma$ -terpinene, 99-85-4; tetrahydrotrimethylnaphthalene, 99532-35-1; isophorone, 78-59-1; cis-linalool oxide, 11063-77-7; trans-linalool oxide, 11063-78-8; furfural, 98-01-1; 2-acetylfuran, 1192-62-7; 5-methyl-2-furfural, 620-02-0; methyl 2-furoate, 611-13-2; 4-hydroxy-3-methyl-2-(2-propenyl)-2-cyclopenten-1-one, 551-45-1; decahydronaphthol, 30756-60-6; phellandral, 21391-98-0;  $\beta$ -terpineol, 138-87-4;  $\alpha$ -terpineol, 98-55-5; 2,3-dimethylmaleic anhydride, 766-39-2; dihydrotrimethylnaphthalene, 80793-13-1;

methyl salicylate, 119-36-8; nerol, 106-25-2; geraniol, 106-24-1; γ-octalactone, 104-50-7; β-ionone, 79-77-6; diethylene glycol, 111-46-6; 4-(2',3',6'-trimethylphenyl)but-3-en-2-one, 56681-06-2; γ-decalactone, 706-14-9; coniferyl alcohol, 458-35-5; eugenol, 97-53-0; 1,2-dicyclohexen-1-yl-1,2-ethandiol, 35811-99-5; 4-(trimethylphenyl)but-3-en-2-one, 99532-36-2; isoeugenol, 97-54-1; terephthaldehyde, 623-27-8.

### LITERATURE CITED

Chien, M. Perfum. Flavor. 1984, 9 (2), 167.

- Engel, K.-H.; Tressl, R. J. Agric. Food Chem. 1983, 31, 998.EPA/NIH "EPA/NIH Mass Spectral Data Base"; U.S. Department of Commerce: Washington, DC, 1980.
- Heidlas, J.; Lehr, M.; Idstein, H.; Schreier, P. J. Agric. Food Chem. 1984, 32, 1020.
- Jennings, W.; Shibamoto, T. "Qualitative Analysis of Flavor and Fragrance Volatiles by Glass Capillary Gas Chromatography"; Academic Press: New York, 1980.
- Kameoka, H.; Kitagawa, C. Nippon Nogeikagaku Kaishi 1976, 50, 389.
- Kameoka, H.; Tsujino, H.; Yabuno, K.; Inoue, H. Nippon Nogeikagaku Kaishi 1981, 55, 1233.
- Lee, F.-L.; Lii, J.-D.; Hwang, M.-J.; Hwa, J. J. "Development of Pickled and Seasoned Ginger and Prune"; Food Industry Research and Development Institute: Hsinchu, Taiwan, Republic of China, 1984; Report No. 322.
- Majlát, P.; Erdös, Z.; Takács, J. J. Chromatogr. 1974, 91, 89.
- MSDC "Eight Peak Index of Mass Spectra", 2nd ed.; MSDC, AWRZ, Aldermaston: Reading RG7 4PR, U.K., 1974.
- Römer, G.; Renner, E. Z. Lebensm.-Unters.-Forsch. 1974, 156, 329.
- Stoltz, L. P.; Kemp, T. R.; Smith, W. O., Jr.; Smith, W. T. Jr.; Chaplin, C. E. Phytochemistry 1970, 9, 1157.
- Takeo, T. Phytochemistry 1981, 20, 2145.
- TNO "Compilation of Mass Spectra of Volatile Compound in Food"; Central Institute or Nutrition and Food Research, TNO: The Netherlands, 1981.
- Williams, P. J.; Strauss, C. R.; Wilson, B. J. Agric. Food Chem. 1980, 28, 766.
- Williams, P. J.; Strauss, C. R.; Wilson, B. Am. J. Enol. Vitic. 1981, 32, 230.
- Williams, P. J.; Strauss, C. R.; Wilson, B.; Massy-Westropp, R. A. J. Chromatogr. 1982, 235, 471.
- Williams, P. J.; Strauss, C. R.; Wilson, B.; Massy-Westropp, R. A. Phytochemistry 1983, 22, 2039.
- Wilson, B.; Strauss, C. R.; Williams, P. J. J. Agric. Food Chem. 1984, 32, 919.

Received for review September 26, 1984. Revised manuscript received September 26, 1985. Accepted October 30, 1985.