

components of the alloy steel plate, and, apparently, the juice dissolves them in a very uniform manner.

Limited information exists on the Al contents of citrus juices. McHard et al. (1980) reported a range of 0.041–0.155 mg Al/kg juice for noncanned single-strength orange juice, whereas Nikdel and Barros (1984) reported a range of 0.054–0.415 mg of Al/kg of juice for single-strength orange juice. No information is available on the Al content of noncanned SSGJ, but Nikdel (1985) determined a range of 0.079–0.094 mg of Al/kg of juice for freshly prepared single-strength grapefruit juice.

Table I reports the Al contents of freshly canned SSGJ, and Table II reports the Al contents of juices subject to a 12-week storage at varying temperatures. After 12 weeks, the average weekly rates of Al uptake (mg/kg of juice per wk) were 0.003 (10 °C), 0.005 (20 °C), 0.013 (30 °C), 0.027 (40 °C), and 0.073 (50 °C). A 2-fold uptake of Al occurred for every 10 °C rise between 10 and 40 °C, whereas from 40 to 50 °C uptake increased ca. 2.7-fold.

CONCLUSION

Internal corrosion of canned SSGJ is characterized by dissolution of the container metal into the juice. The uptake of tin, iron, and aluminum by canned SSGJ was related to storage time and temperature. The rates of uptake for the metals were Sn > Fe > Al. Storage of juices at temperatures below 40 °C showed a relatively uniform Q_{10} of ca. 2, whereas storage above 40 °C showed Q_{10} values of 2.6 (Sn), 3 (Fe), and 2.7 (Al). Iron and aluminum are found in canned SSGJ at relatively minor levels.

Examination of the Sn, Fe, and Al contents of 30 individual cans by linear regression analysis revealed definitive relationships. The correlation coefficients for Sn vs. Fe (0.809) and Sn vs. Al (0.791) were statistically significant, whereas the Fe vs. Al (0.995) value was highly significant. The data indicate that the tin coating material of the base plate dissolves inconsistently (considerable intercan tin variability, even with cans stored at the same temperature), whereas when the steel base corrodes, the Fe and Al of the alloy dissolve into the juice in a more uniform manner.

Registry No. Al, 7429-90-5; Fe, 7439-89-6; Sn, 7440-31-5.

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Received for review October 21, 1985. Revised manuscript received April 4, 1986. Accepted May 8, 1986. Florida Agricultural Experiment Stations Journal Series No. 6852.

Volatile Constituents of Peel of Quince Fruit, *Cydonia oblonga* Miller

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Oils obtained from the peel of quince fruit, *Cydonia oblonga*, by headspace sampling and simultaneous distillation and extraction (SDE) were analyzed by gas chromatography/mass spectrometry using fused silica capillary columns. Thirty-four volatile compounds were identified in a headspace sample. Over 77% of the total GC peak area consisted of esters. Sixty-three compounds were identified in an extract obtained by SDE. Major components were ethyl esters, which contribute the powerful fruity and floral odors to this fruit.

Japanese quince is the fruit of a deciduous tree, *Cydonia oblonga* Miller, native to China and widely planted as an ornamental in the occident. The trees bear oval, yellow

fruit about 10 cm long. Ripe quince fruit are not edible raw because of their hardness and bitterness, but they nevertheless emit a floral odor so strong that they are sometimes used as household room fresheners. This powerful fragrance itself suggests the presence of many low molecular weight esters in quince fruit.

The essential oil of quince (*Cydonia* species) has not been studied thoroughly. Schreyen et al. (1979) reported 79 components in the essential oil obtained from the whole

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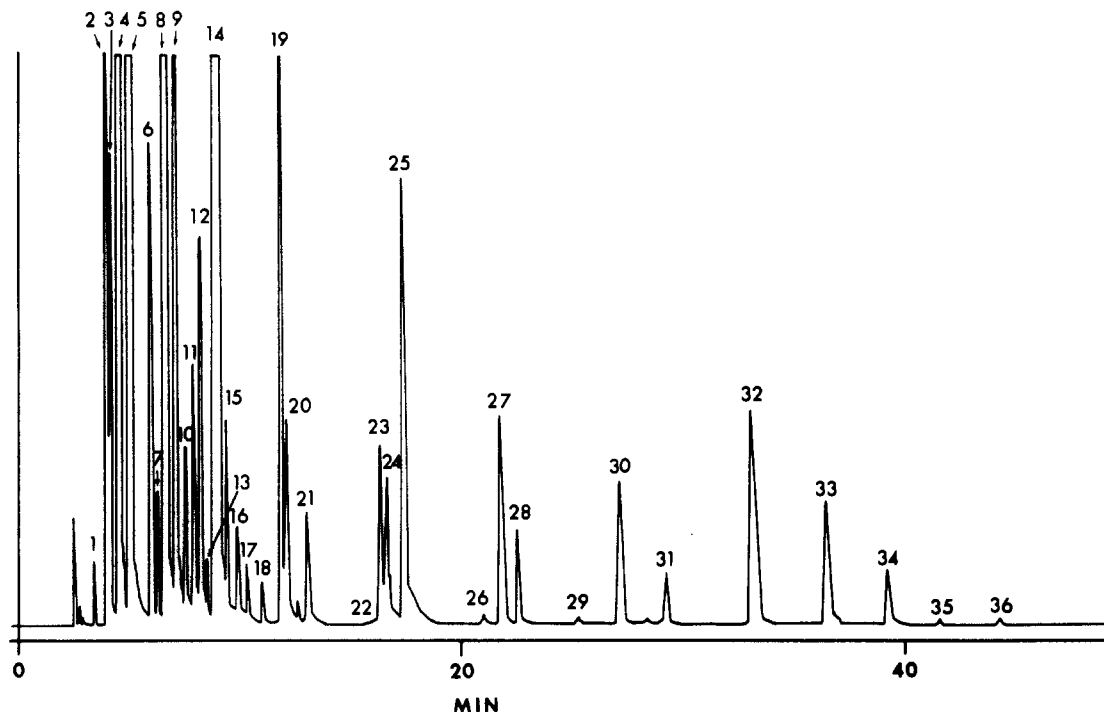


Figure 1. Gas chromatogram of the headspace sample from quince peel on DB-WAX bonded-phase fused silica capillary column. Column held at 60 °C for 10 min, programmed at 2 °C/min to 200 °C.

fruit (peel and pulp) of quince, *Cydonia vulgaris*. The oil of the whole fruit of Japanese quince, *C. oblonga*, has been analyzed by two groups of Japanese researchers. Shimizu and Yoshihara (1977) identified four ethyl esters of C₆, C₈, C₁₀, and C₁₂ saturated acids in the oil of the whole fruit of *C. oblonga*. Tsuneya et al. (1980) found two terpene-related lactones in the whole fruit of *C. oblonga* Mill., Marmelo. Most of the essential oil of quince fruit is obtained from the peel. In this study, therefore, volatile constituents of the peel of quince fruit were investigated.

EXPERIMENTAL SECTION

Sample Preparation. Fresh fruits of Japanese quince *C. oblonga* (Karin in Japanese) were harvested in November, 1984, in Nagano, Japan. The fruits were hand-peeled with a knife, and the peel was sliced into small pieces.

Headspace sampling was done by the method of Yabu-moto et al. (1977). Sliced peel weighing 237 g was placed in a 2-L round-bottom, two-neck flask. Purified nitrogen gas was introduced into the flask, and the volatile constituents of the quince peel were swept into a 30 cm × 8 mm i.d. glass column packed with 80/100 mesh Porapak Q (2.05 g; Waters Associates, Milford, MA) preconditioned at 200 °C for 4 h. Water vapor was removed with a trap packed with 2 g of molecular sieve 3A that was connected between the flask and the Porapak trap. The nitrogen flow was maintained at 14 mL/min for 12 h. The column was then disconnected and nitrogen (4 mL/min) passed through it in the reverse direction. The column temperature was raised to 100 °C for 2 h while the volatiles were trapped by condensation in a 30 cm × 1 mm i.d. glass capillary tube held at -78 °C. The volatiles were transferred from the glass capillary tube to a 10-μL microsyringe and then injected into the gas chromatography (GC) or the gas chromatography/mass spectrometry apparatus (GC/MS).

Simultaneous distillation and extraction (SDE) was done by a modified method of Likens and Nickerson (1966). Sliced peel weighing 250 g was mixed with 1 L of deionized water and put in a 2-L round-bottom flask. The volatile constituents were isolated with an SDE apparatus (Schultz

Table I. Compounds Identified in the Headspace Sample from Quince Fruit Peel

peak no. (Figure 1)	compound	peak area %	I ^a
1	acetaldehyde	b	744
2	ethyl acetate	22.83	880
3	ethanol	12.02	925
4	ethyl propionate	29.23	966
5	ethyl isobutyrate	9.6	975
6	isobutyl acetate	0.95	1018
7	2-butanol	0.31	1037
8	ethyl butyrate	5.38	1044
9	ethyl 2-methylbutyrate	1.71	1064
10	butyl acetate	0.40	1078
11	isobutyl propionate	0.50	1085
12	isobutyl isobutyrate	0.76	1095
13	ethyl 3-butenolate	b	1105
14	2-methyl-1-propanol	8.43	1114
15	isoamyl acetate	0.34	1130
16	ethyl pentanoate	0.15	1142
17	butyl isobutyrate	0.06	1149
18	isobutyl butyrate	b	1160
19	ethyl 2-butenolate	1.11	1172
20	butanol	0.21	1175
21	limonene	0.23	1190
22	butyl butyrate	b	1221
23	(E)-2-hexenal	0.58	1228
24	unknown	0.29	1231
25	ethyl hexanoate	1.91	1241
26	amyl alcohol	b	1271
27	hexyl acetate	0.68	1278
28	unknown	0.19	1284
29	ethyl 3-hexenoate	b	1303
30	(Z)-3-hexenyl acetate	0.58	1315
31	(E)-2-hexenyl acetate	0.08	1322
32	hexanol	0.80	1357
33	(Z)-3-hexenol	0.47	1394
34	(E)-2-hexenol	0.19	1420
35	ethyl octanoate	b	1441
36	acetic acid	b	1541

^a Kovats index. ^b Peak area percent less than 0.01.

et al., 1977). The steam distillation-extraction (water/dichloromethane, 1 L/50 mL) was continued for 3 h and the extract dried over anhydrous sodium sulfate for 12 h.

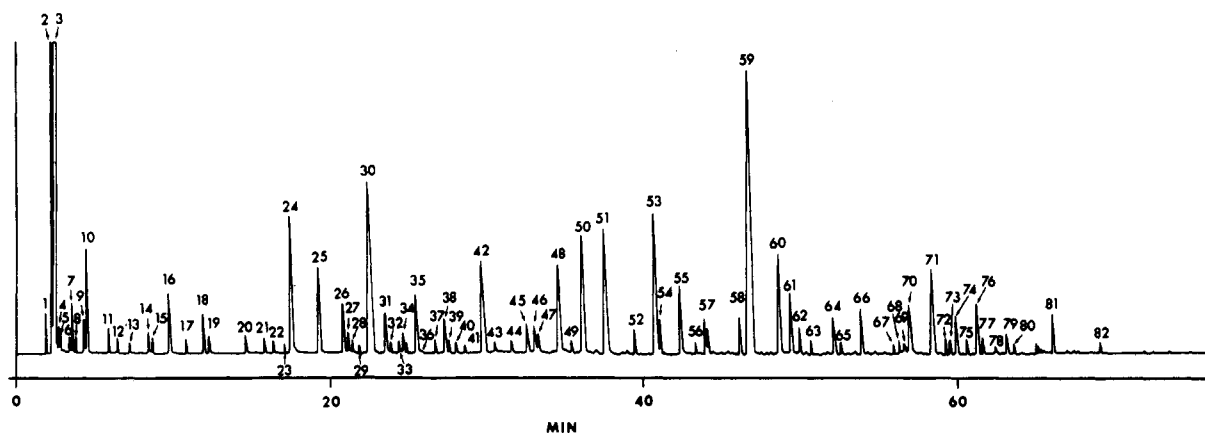


Figure 2. Gas chromatogram of the SDE sample from quince peel on DB-WAX bonded-phase fused silica capillary column. Column held at 60 °C for 10 min, programmed at 2 °C/min to 200 °C and held.

Table II. Compounds Identified in the SDE Sample from Quince Fruit Peel

peak no. (Figure 2)	compounds	peak area %	<i>I</i> ^a	peak no. (Figure 2)	compounds	peak area %	<i>I</i> ^a
1	acetone	b	818	43	isobutyric acid	b	1574
2	ethyl acetate	4.7	880	44	ethyl (<i>E</i>)-6-nonenolate	b	1592
3	dichloromethane (solvent)	c	905	45	ethyl (<i>Z</i>)-6-nonenolate	0.48	1609
4	ethyl propionate	0.18	966	46	unknown	1.64	1616
5	diacetyl	b	980	47	butyl octanoate	b	1619
6	impurity from solvent	c	1029	48	ethyl decanoate	3.08	1643
7	ethyl butyrate	0.22	1044	49	unknown	b	1659
8	ethyl 2-methylbutyrate	b	1062	50	ethyl (<i>E</i>)-4-decenoate	5.62	1672
9	hexanal	0.44	1094	51	ethyl (<i>Z</i>)-4-decenoate	4.96	1699
10	2-methyl-1-propanol	1.73	1101	52	unknown	0.84	1729
11	butanol	0.39	1160	53	<i>trans</i> - α -farnesene	5.92	1753
12	ethyl 2-methyl-2-propenoate	0.33	1175	54	unknown	b	1760
13	heptanal	b	1194	55	unknown	1.29	1778
14	isoamyl alcohol	0.78	1218	56	unknown	1.10	1795
15	(<i>E</i>)-2-hexenal	0.20	1222	57	unknown	b	1803
16	ethyl hexanoate	1.43	1241	58	unknown	1.49	1835
17	amyl alcohol	b	1261	59	ethyl dodecanoate + unknown	13.65	1847
18	hexyl acetate	1.01	1280	60	ethyl (<i>E</i>)-7-dodecenoate	3.26	1888
19	acetoin	0.84	1285	61	unknown	2.61	1905
20	(<i>Z</i>)-3-hexenyl acetate	0.28	1317	62	β -ionone	0.73	1917
21	(<i>E</i>)-2-hexenyl acetate + ethyl heptanoate	0.81	1332	63	unknown	b	1934
22	hexyl isobutyrate	0.79	1339	64	unknown	0.90	1966
23	isobutyl hexanoate	b	1351	65	unknown	b	1976
24	hexanol	5.58	1359	66	eugenyl methyl ether	1.65	2003
25	(<i>Z</i>)-3-hexenol	2.95	1390	67	ethyl tetradecanoate	b	2043
26	(<i>E</i>)-2-hexenol	1.93	1411	68	ethyl (<i>E</i>)-9-tetradecenoate	b	2052
27	hexyl butyrate	0.69	1419	69	capric acid	b	2056
28	(<i>E</i>)-2-octenal	b	1424	70	ethyl (<i>Z</i>)-9-tetradecenoate	1.79	2065
29	hexyl 2-methylbutyrate	b	1431	71	unknown	2.55	2094
30	ethyl octanoate	6.37	1441	72	γ -decalactone	b	2113
31	furfural	1.22	1459	73	unknown	b	2118
32	(<i>Z</i>)-3-hexenyl butyrate	b	1464	74	2,7-dimethyl-4-hydroxy-5(<i>E</i>),7- octadienoic acid lactone (stereoisomers)	1.82	2127
33	unknown	b	1474	75	eugenol	0.79	2141
34	ethyl (<i>E</i>)-6-octenoate	0.65	1478	76	2,7-dimethyl-4-hydroxy-5(<i>E</i>),7- octadienoic acid lactone (stereoisomers)	1.95	2152
35	ethyl (<i>Z</i>)-6-octenoate	1.45	1493	77	unknown	0.72	2159
36	(<i>E</i>)- or (<i>Z</i>)-theaspirane	b	1496	78	anisyl propionate	0.28	2175
37	benzaldehyde	0.65	1512	79	unknown	0.67	2189
38	ethyl 3-hydroxybutyrate	1.19	1522	80	1,2,3-trimethyl-5-(2-propenyl)benzene	b	2198
39	propyl octanoate	b	1526	81	ethyl hexadecanoate	b	2243
40	(<i>E</i>)- or (<i>Z</i>)-theaspirane	0.40	1533	82	γ -dodecalactone	b	2315
41	ethyl nonanoate	b	1541				
42	isobutyl octanoate	3.02	1561				

^a Kovats index. ^b Peak area percent less than 0.01. ^c Peak areas of solvent are excluded.

The solvent was removed on a rotary flash evaporator, and the concentrated sample (1 mL) was injected into the GC or the GC/MS.

Identification of the Volatile Constituents. Samples of volatiles were analyzed by both GC and GC/MS. Identification of the GC peaks of the samples was made by comparison of their GC retention indices and their mass spectra to those of authentic compounds.

A Shimadzu 7A gas chromatograph equipped with a flame ionization detector (FID), and a 30 m \times 0.25 mm i.d. fused silica bonded phase DB-WAX capillary column (J & W Scientific, Rancho Cordova, CA) was used for the GC analysis. The nitrogen carrier gas flow was 0.6 mL/min. An injector split ratio was 30:1. The injector and detector temperatures were 250 °C. GC column and oven conditions were as described in Figures 1 and 2. GC peak areas

were integrated by a Shimadzu Chromatopac C-R3A combined with the Shimadzu 7A gas chromatograph.

A Shimadzu QP 1000 gas chromatograph/mass spectrometer was used for GC/MS analysis. Operating conditions were as follows: ion source temperature, 250 °C; filament voltage, 70 eV; interface temperature, 200 °C.

RESULTS AND DISCUSSION

The results of the GC and GC/MS analysis of the volatile constituents of quince fruit peel are shown in the chromatograms of Figure 1 (headspace sample) and 2 (SDE sample). Table I lists the compounds identified in the headspace sample and Table II those identified in the SDE sample. These results show that the chemical constituents of the peel oil of the quince fruit of *C. oblonga* differ substantially from those of the oil of the whole quince fruit of *C. vulgaris* (Schreyer et al., 1979).

The headspace sample contained a greater proportion of the low boiling point esters that give quince its powerful floral odor (Arctander, 1969) than did the SDE sample. Esters composed over 77% of the total GC peak area of the headspace sample, and by themselves ethyl acetate and ethyl propionate composed over 50%. Schreyer et al. (1979) found may alkane and alkene alcohols to be major components in headspace sample in the whole fruit of *C. vulgaris*. The major alcohols found in headspace samples of the peel of *C. oblonga* in the present study were ethanol (peak area percent 12.02) and 2-methylpropanol (peak area percent 8.43). And compared to the oil of whole fruit of *C. vulgaris*, the oil of the peel of *C. oblonga* had a much lower proportion of total alcohols to total esters. A trace amount of acetic acid was identified in the headspace sample of the present study. Acetic acid gives a slightly pungent odor to the peel oil.

Many less volatile compounds were identified in the SDE sample. Instead of a simple floral odor, this sample possessed a rather sweet floral odor, which may be due to the low ester content (total area percent 56.24) compared to that of the headspace sample (77.3). Schreyer et al. (1979) recovered large amounts of furfural and 5-methylfurfural from the steam distillate of whole fruit. These furfurals may form from the effects of the heat treatment applied during distillation on a sugar in the fruit. Much less furfural was found in the peel oil than in the whole fruit oil, indicating a lower sugar content in the peel. The major characteristic of this peel oil that differentiates it from other essential oils, however, is its low content of terpenes. Thus, *trans*- α -farnesene and β -ionone were the only terpenes found in the present study. The terpene-related compound theaspirane (peak 36 in Figure 2) was not reported in Schreyer et al. (1979), but it has been found in other plant products such as raspberry, yellow passion fruit, and tea (Ohloff, 1978). Two isomeric terpene-related lactones (peaks 74 and 76 in Figure 2) were identified as major constituents in whole quince fruit oil (Tsuneya et al., 1980). The *cis* form of 3-hexenol predominates, and the *trans* form predominates in 2-hexenol. The same trend was observed in the case of hexenyl ace-

tate. These phenomena were also reported in a tea aroma (Kobayashi et al., 1985). The high content of volatile esters of quince fruit peel suggests a new source of essential oils.

Registry No. AcH, 75-07-0; AcOEt, 141-78-6; EtOH, 64-17-5; EtCO₂Et, 105-37-3; *i*-PrCO₂Et, 97-62-1; AcOBu-*i*, 110-19-0; MeCH₂CH(OH)Me, 78-92-2; PrCO₂Et, 105-54-4; EtCH(Me)CO₂Et, 7452-79-1; AcOBu, 123-86-4; EtCO₂Bu-*i*, 540-42-1; *i*-PrCO₂Bu-*i*, 97-85-8; CH₂=CHCH₂CO₂Et, 1617-18-1; *i*-PrCH₂OH, 78-83-1; AcO(CH₂)₂Pr-*i*, 123-92-7; Me(CH₂)₃CO₂Et, 539-82-2; *i*-PrCO₂Bu, 97-87-0; PrCO₂Bu-*i*, 539-90-2; MeCH=CHCO₂Et, 10544-63-5; BuOH, 71-36-3; PrCO₂Bu, 109-21-7; (*E*)-CHOCH=CH(CH₂)₂Me, 6728-26-3; Me(CH₂)₄CO₂Et, 123-66-0; Me(CH₂)₄OH, 71-41-0; AcO(CH₂)₅Me, 142-92-7; MeCH₂CH=CHCH₂CO₂Et, 2396-83-0; (*Z*)-AcO(CH₂)₂CH=CH₂, 3681-71-8; (*E*)-AcOCH₂CH=CHPr, 2497-18-9; Me(CH₂)₅OH, 111-27-3; (*Z*)-MeCH₂CH=CH(CH₂)₂OH, 928-96-1; (*E*)-Me(CH₂)₂CH=CHCH₂OH, 928-95-0; Me(CH₂)₂CO₂Et, 106-32-1; AcOH, 64-19-7; AcMe, 67-64-1; Me(CO)₂Me, 431-03-8; Me(CH₂)₄CHO, 66-25-1; CH₂=C(Me)CO₂Et, 97-63-2; Me(CH₂)₅CHO, 111-71-7; *i*-Pr(CH₂)₂OH, 123-51-3; Me(CH₂)₅CO₂Et, 106-30-9; *i*-PrCO₂(CH₂)₅Me, 2349-07-7; Me(CH₂)₄CO₂Bu-*i*, 105-79-3; PrCO₂(CH₂)₅Me, 2639-63-6; (*E*)-CHOCH=CH(CH₂)₄Me, 2548-87-0; EtCH(Me)CO₂(CH₂)₂Me, 10032-15-2; (*Z*)-PrCO₂(CH₂)₂CH=CH₂, 16491-36-4; (*E*)-MeCH=CH(CH₂)₄CO₂Et, 25143-92-4; (*Z*)-MeCH=CH(CH₂)₄CO₂Et, 101773-18-6; PhCHO, 100-52-7; MeCH(OH)CH₂CO₂Et, 5405-41-4; Me(CH₂)₆CO₂Pr, 624-13-5; Me(CH₂)₇CO₂Et, 123-29-5; Me(CH₂)₆CO₂Bu-*i*, 5461-06-3; *i*-PrCO₂H, 79-31-2; (*E*)-EtCH=CH(CH₂)₄CO₂Et, 75567-02-1; (*Z*)-EtCH=CH(CH₂)₄CO₂Et, 101773-19-7; Me(CH₂)₆CO₂Bu, 589-75-3; Me(CH₂)₈CO₂Et, 110-38-3; (*E*)-Me(CH₂)₄CH=CH(CH₂)₂CO₂Et, 76649-16-6; (*Z*)-Me(CH₂)₄CH=CH(CH₂)₂CO₂Et, 7367-84-2; Me(CH₂)₁₀CO₂Et, 106-33-2; (*E*)-Me(CH₂)₃CH=CH(CH₂)₅CO₂Et, 101773-20-0; Me(CH₂)₁₂CO₂Et, 124-06-1; (*E*)-Me(CH₂)₃CH=CH(CH₂)₇CO₂Et, 68862-25-9; Me(CH₂)₈CO₂H, 334-48-5; (*Z*)-Me(CH₂)₃CH=CH(CH₂)₇CO₂Et, 24880-50-0; (*E*)-CH₂=C(Me)CH=CHCH(OH)CH₂CH(Me)CO₂H (isomer 1), 101773-21-1; Me(CH₂)₁₄CO₂Et, 628-97-7; (*E*)-CH₂=C(Me)CH=CHCH(OH)CH₂CH(Me)CO₂H (isomer 2), 101773-22-2; limonene, 138-86-3; acetoin, 513-86-0; furfural, 98-01-1; (*E*)-theaspirane, 66537-39-1; (*Z*)-theaspirane, 66537-40-4; *trans*- α -farnesene, 502-61-4; β -ionone, 79-77-6; eugenyl methyl ether, 93-15-2; γ -decalactone, 706-14-9; eugenol, 97-53-0; γ -dodecalactone, 2305-05-7; 1,2,3-trimethyl-5-(2-propenyl)benzene, 101773-24-4; anisyl propionate, 101773-23-3.

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Received for review November 8, 1985. Accepted March 4, 1986.