Volatile Constituents of Apricot (Prunus armeniaca)

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Volatile Constituents of Apricot (*Prunus armeniaca*)

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Volatile constituents of fresh apricots (*Prunus armeniaca*) of the Blenheim variety were analyzed by capillary gas chromatography and gas chromatography–mass spectrometry. The fruit was sampled by simultaneous vacuum steam distillation–extraction. A total of 49 components were identified in the extract, including 25 constituents reported for the first time in apricot. Linalool, lactones, and C_6 lipid peroxidation products were the major constituents in the extract. Odor unit values, calculated from concentration and odor threshold data, indicate that the following compounds are major contributors to blended apricot aroma: β-ionone, linalool, γ-decalactone, hexanal, (*E*)-2-hexenal, (*E*,*E*)-2,4-decadienal, (*E*)-2-nonenal, and γ-dodecalactone. Headspace analyses of the intact fruit led to the identification of 83 components, 60 of which had not been previously reported in apricot. Esters were the dominant constituents in the headspace samples.

The first significant studies on apricot flavor were performed by Tang and Jennings (1967, 1968) who utilized direct extraction, vacuum steam distillation, and charcoal adsorption to isolate the volatiles from the Blenheim variety. A number of terpene hydrocarbons, terpene alcohols, and lactones were identified by gas chromatographic retentions and infrared spectroscopy. Rodriguez et al. (1980) studied the variety Rouge du Roussillon and identified constituents such as camphene, γ-terpinene, hexanol, benzaldehyde, γ-butyrolactone, and nerol for the first time in apricot. Later studies on the same variety led to the identification of damascenone, β-ionone, dihydroactinidiolide, rose oxide, and nerol oxide (Chairote et al., 1981). These authors felt that the apricot aroma was dependent on several constituents such as lactones, terpene alcohols, and benzaldehyde. Guichard and Souty (1988) compared the relative concentrations of various volatiles present in six different apricot varieties (Precoce de Tyrinthe, Palsteyn, Moniqui, Rouge du Roussillon, Polonais, Bergeron) grown in the south of France. A total of 82 compounds were identified, 58 of which had not been previously reported in apricot. The most abundant constituents were C_6 lipid degradation products, lactones, terpene alcohols, and ketones. Sharaf et al. (1989) identified 31 components

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Table I. Headspace Constituents of Fresh Apricots (Ether Elution)

peak no.[a]	constituent	I^{DB-1} exptl	I^{DB-1} ref	% area[b]	T,[c] ppb	peak no.[a]	constituent	I^{DB-1} exptl	I^{DB-1} ref	% area[b]	T,[c] ppb
	(ethanol)[d-f]		440			54	linalool[f]	1088	1085	0.106	6[j]
	2,3-butanedione[e,f]	560	558			56	hexyl propanoate[e,f]	1091	1088	0.431	8
	(trichloromethane)[d-f]		601			57	2-methylbutyl 2-methylbutanoate[e]	1091	1090	ns	
	ethyl acetate[f]	600	600			58	methyl octanoate[f]	1108	1107	0.010	
	2-methylpropanol[e,f]	607	608			60	pentyl 2-methylbutanoate[e,f]	1126	1123	0.161	
1	butanol[f]	654	654	0.147	500[h]	61	hexyl 2-methylpropanoate[e,f]	1138	1137	0.766	13
3	ethyl propanoate[f]	700	699	0.009		62	2-methylpropyl hexanoate[e,f]	1139	1138	0.413	
4	methyl butanoate[f]	708	705	0.009	76	63	2-methylbutyl pentanoate[e]	1142	1142	0.014	
5	2-methylbutanol[e,f]	728	729	0.062	300	64	(*E*,*Z*)-1,3,5-undecatriene[e]	1167	1165	0.029	
6	ethyl 2-methylpropanoate[e,f]	751	751	0.018	0.1[g]		ethyl (*E*)-4-octenoate[e,f]	1169	1169		
9	methyl 2-methylbutanoate[e]	767	768	0.005		65	(*Z*)-3-hexenyl butanoate[e,f]	1173	1170	0.053	
10	hexanal	778	778	0.010	5[h]	66	butyl hexanoate[e,f]	1187	1176	24.544	700
11	ethyl butanoate[f]	789	789	2.000	1[i]	67	hexyl butanoate[e,f]	1188	1178	11.784	250
12	propyl propanoate[e]	796	796	0.006	57[i]	68	ethyl octanoate[e,f]	1189	1182	0.014	
13	butyl acetate[f]	799	796	0.417	66[i]	69	β-cyclocitral[e]	1196	1194	0.014	5
17	2-propyl butanoate[e]	834	834	0.006			dodecane[e,f]	1200	1200		
18	ethyl 2-methylbutanoate[e,f]	841	842	0.228	0.3	71	γ-octalactone	1208	1210	0.061	7[k]
22	hexanol[f]	860	860	0.307	2500	72	(*Z*)-3-hexenyl 2-methylbutanoate[e,f]	1218	1215	0.033	
23	propyl butanoate[e,f]	885	885	0.186	124	74	hexyl 2-methylbutanoate[e,f]	1228	1222	3.503	22
24	ethyl pentanoate[e,f]	887	888	0.030	5[i]	76	3-methylbutyl hexanoate[e,f]	1236	1233	0.024	
25	butyl propanoate[e,f]	895	894	1.301	200	77	2-methylbutyl hexanoate[e,f]	1240	1236	0.351	32
26	pentyl acetate	898	895	0.015		78	pentyl hexanoate[e,f]	1274	1270	0.696	
27	2-methylpropyl 2-methylpropanoate[e,f]	902	901	0.020	30	80	propyl octanoate[e,f]	1277	1277	0.039	
28	methyl hexanoate[f]	909	910	0.071	84		tridecane[e,f]	1300	1300		
29	propyl 2-methylbutanoate[e,f]	936	936	0.023		83	2-methylpropyl octanoate[e,f]	1334	1334	0.027	
30	butyl 2-methylpropanoate[e,f]	944	943	0.670	80	84	ethyl (*Z*)-4-decenoate[e,f] + ?	1363	1361	0.237	
31	2-methylpropyl butanoate[e,f]	946	945	0.572		85	hexyl hexanoate[e,f]	1374	1369	4.115	
32	2-methylbutyl propanoate[e,f]	961	961	0.020		86	butyl octanoate[e,f]	1374	1373	0.438	
33	butyl butanoate[e,f]	990	982	29.657	100	87	ethyl decanoate[e,f]	1381	1379	0.074	
34	ethyl hexanoate[f]	992	986	4.700	1	90	dihydro-β-ionone[e,f]	1414	1414	0.110	
36	2-methylpropyl 2-methylbutanoate[e,f]	995	991	0.141		91	γ-decalactone	1424	1422	1.825	11[k]
37	hexyl acetate[f]	999	995	0.146	2[g]	95	δ-decalactone	1444	1447	0.071	100[k]
38	2-methylbutyl 2-methylpropanoate[e,f]	1001	1002	0.041		97	dihydroactinidiolide	1473	1475	0.046	
40	limonene[f]	1020	1020	0.033	10	98	pentadecane[e,f]	1501	1500	0.440	
42	butyl 2-methylbutanoate[e,f]	1033	1030	2.759	17	100	hexyl benzoate[e]	1551	1551	0.020	
43	butyl 3-methylbutanoate[e]	1035	1035	0.016		101	hexyl octanoate[e]	1566	1565	0.024	
44	pentyl 2-methylpropanoate[e,f]	1040	1039	0.040		102	butyl decanoate[e]	1570	1571	0.016	
45	(*E*)-β-ocimene[e]	1042	1037	0.058		103	ethyl dodecanoate	1579	1578	0.033	
46	3-methylbutyl butanoate[e]	1044	1041	0.042		104	tetradecanal	1591	1592	0.015	
47	2-methylbutyl butanoate[e]	1047	1047	0.363		106	γ-dodecalactone	1631	1635	0.096	7[k]
51	pentyl butanoate[e,f] + ?	1081	1080	1.452	210	110	hexadecanal[e]	1795	1796	0.117	
52	propyl hexanoate[e,f]	1081	1081	0.199		111	(phthalate)[d]	1900		0.023	
53	ethyl heptanoate[e,f]	1084	1080	0.111	2.2	112	ethyl hexadecanoate	1978	1978	0.045	

[a] The peak numbers correspond to the numbers in Figure 1. Mass spectra were consistent with those of authentic reference standards. [b] Peak area percentage of total FID area excluding the solvent peaks (assuming all response factors of 1). ns = not separated from preceding compound. [c] Odor threshold in water. [d] Tentative or partial identifications enclosed in parentheses. [e] Identified for the first time in apricot. [f] Detected and identified with headspace method employing thermal desorption. [g] Buttery et al., 1982. [h] Buttery et al., 1971. [i] Flath et al., 1967. [j] Buttery et al., 1969. [k] Engel et al., 1988.

in overripe apricots of the Zibda variety. This study reports on the volatile constituents present in fresh apricots of the cv. Blenheim, an important California variety.

EXPERIMENTAL SECTION

Materials. Fresh, tree-ripened apricots (*Prunus armeniaca*) of the Blenheim variety were obtained from an orchard in Hollister, CA (June 1988). Ethyl antioxidant 330 (1,3,5-trimethyl-2,4,6-tris(3,5-di-*tert*-butyl-4-hydroxybenzyl)benzene) was received from Ethyl Corp. (Baton Rouge, LA).

Sample Preparation. *1. Dynamic Headspace Sampling. A. Solvent Elution.* Intact fruit (total weight 3.38 kg) were placed in a 9-L Pyrex glass container. A Pyrex head to allow the passage of air into and out was fitted into a standard ground glass joint in the upper part of the container. Purified air (passed through activated carbon filters) entered the bottom of the chamber via a Teflon tube and exited out the top through a Tenax trap. The traps consisted of glass tubes packed with 10 g of Tenax (Alltech Associates, Deerfield, IL) and terminated in standard ball and socket joints. The air stream was sampled at room temperature (ca. 24 °C) for 22 h at 3 L/min. The collected volatiles were eluted from the Tenax trap with 100 mL of freshly distilled diethyl ether containing ca. 0.001% Ethyl antioxidant 330. The ether extract was then concentrated with a Vigreux column (16 cm) to a final volume of ca. 100 μL.

B. Thermal Desorption. The sampling chamber, activated charcoal, air purifier, sampling pump, and Tenax traps have been described previously (Flath and Ohinata, 1982; Takeoka et al., 1988). Intact fruit (669 g) were placed in the sampling chamber, and the system was purged overnight with a 25 mL/min flow of purified air. A Tenax trap was attached to the exit port of the sampling chamber, and the air stream was collected for 15 min at 50 mL/min. The trap was removed, and its contents were examined by capillary gas chromatography with flame ionization detection (GC/FID). A second trap was attached to the chamber and the headspace vapor sampled for 30 min at 50 mL/min. The trap was removed and used for capillary gas chromatography–mass spectrometry (GC–MS).

2. Vacuum Steam Distillation. Extraction. The fruit were cut in half, and the stones were removed and discarded. The skin and pulp (1.7–1.9 kg) were blended with 1 L of water for 15 s in a Waring blender. Two batches were prepared from a

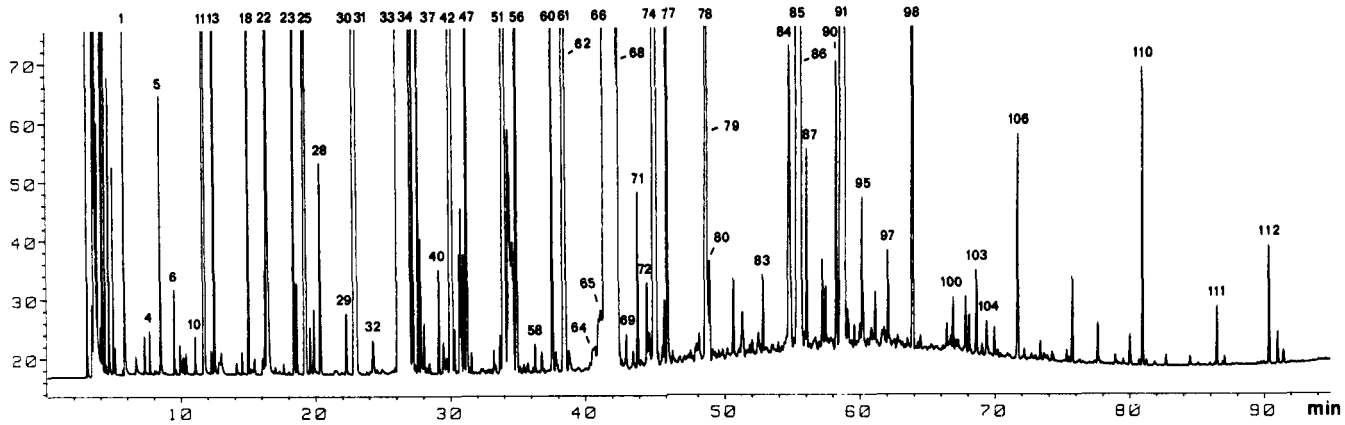

Figure 1. Capillary gas chromatogram of intact apricot headspace volatiles (ether elution). Temperature programmed from 30 °C (4 min isothermal) to 210 °C at 2 °C/min on a 60 m × 0.32 mm (i.d.) DB-1 column. The peak numbers correspond to the numbers in Table I.

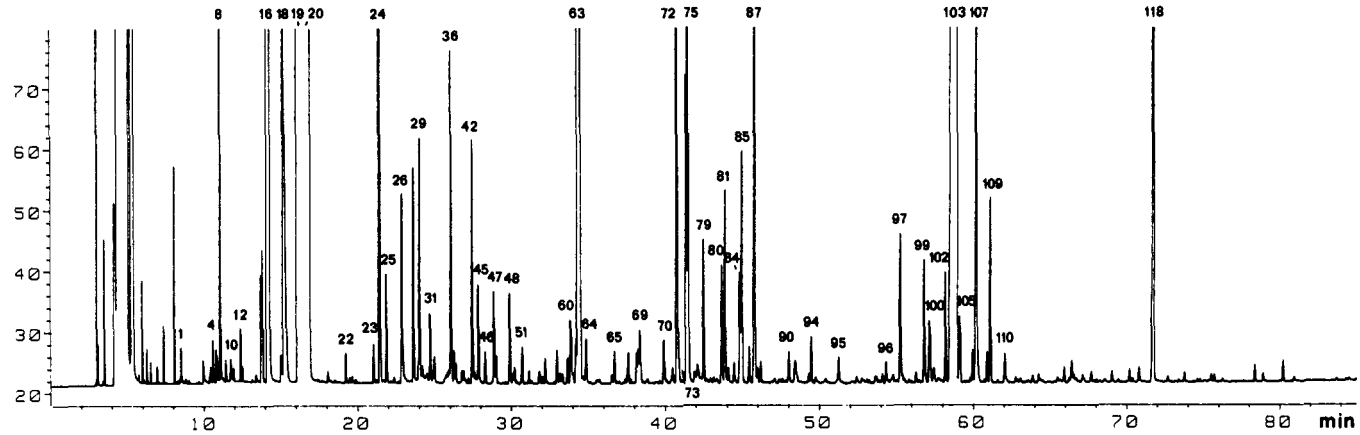

Figure 2. Capillary gas chromatogram of apricot volatiles obtained by vacuum steam distillation-extraction. Temperature programmed from 30 °C (4 min isothermal) to 210 °C at 2 °C/min on a 60 m × 0.32 mm (i.d.) DB-1 column. The peak numbers correspond to the numbers in Table II.

total of 3.59 kg of fruit pulp. The following compounds were added to the blended slurry as internal standards: 4-methylpent-2-yl acetate, 4-nonanone, and eugenol (final concentrations 0.7 ppm each). The mixture was blended for another 15 s and then added to a 12-L round-bottomed flask. An additional 1000 mL of water was added to the flask. Fifty milliliters of antifoam solution was added. The antifoam solution was prepared by adding 12 mL of Hartwick antifoam 50 emulsion to 900 mL of water in a 1-L flask and boiling until the volume was reduced to ca. 600 mL to remove volatiles. A modified Likens-Nickerson distillation-extraction head was used (Schultz et al., 1977). The fruit slurry was subjected to simultaneous vacuum distillation-extraction for 3 h with 125 mL of hexane (60 mmHg). After freezing (-20 °C) to remove residual water, the hexane extract was concentrated with a Vigreux column under reduced pressure (60 mmHg) to a final volume of 0.6-0.8 mL.

Gas Chromatography. A Hewlett-Packard 5890 gas chromatograph (Hewlett-Packard, Avondale, PA) equipped with a flame ionization detector (FID) was used. Separations were performed on a 60 m × 0.32 mm (i.d.) DB-1 column (d_f = 0.25 μm; J&W Scientific, Folsom, CA). The oven temperature was programmed from 30 °C (4 min isothermal) to 210 °C at 2 °C/min. Helium carrier gas was used at a flow rate of 1.64 mL/min (30 °C; $\bar{u}$ = 34 cm/s). The injector temperature was maintained at 200 °C; the detector temperature was held at 230 °C. Split injections (1:30) were employed. Data processing was performed with an HP 5895 GC ChemStation.

The conditions and instrumentation used for the thermal desorption headspace analyses were described previously (Takeoka et al., 1988).

Gas Chromatography-Mass Spectrometry. A Finnigan MAT 4500 GC/MS/INCOS system (Finnigan MAT, San Jose, CA) equipped with the same type of column used in the GC analyses was used. For the ether extract (obtained by dynamic headspace sampling) the following conditions were employed: The column temperature was programmed from 50 to 250 °C at 4 °C/min; A split ratio of 1:25 was employed. For the hexane extract (obtained by simultaneous vacuum steam distillation-extraction) the following conditions were employed: The column temperature was programmed from 30 °C (4 min isothermal) to 210 °C at 2 °C/min. A split ratio of 1:23 was used. Helium carrier gas was used at a rate of 3.9 mL/min.

For thermal desorption headspace analyses, this unit was equipped with a headspace unit operationally identical with one described previously (Takeoka et al., 1988). The column temperature was programmed from 0 to 230 °C at 3 °C/min.

Reference Compounds. Esters were prepared by refluxing the corresponding alcohol (0.1 mol) and acid (0.05 mol) and *p*-toluenesulfonic acid (5-10 mg) in benzene overnight. Water was removed with a Dean-Stark trap. Propyl octanoate had the following mass spectrum: 186 (1), 157 (2), 146 (8), 145 (100), 128 (8), 127 (94), 115 (17), 102 (32), 101 (15), 87 (16), 83 (11), 73 (32), 61 (70), 60 (44), 57 (45), 55 (23), 43 (42). 2-Methylpropyl octanoate had the following mass spectrum: 200 (>1), 170 (1), 145 (49), 144 (10), 128 (9), 127 (100), 116 (6), 101 (10), 87 (7), 73 (13), 60 (16), 57 (74), 56 (71), 55 (14), 43 (16). Butyl octanoate had the following mass spectrum: 200 (>1), 157 (2), 146 (8), 145 (100), 144 (8), 128 (7), 127 (80), 116 (9), 101 (21), 89 (9), 87 (10), 83 (9), 73 (24), 60 (25), 57 (59), 56 (92), 55 (22), 43 (20). Butyl decanoate had the following mass spectrum: 228 (2), 185 (4), 174 (9), 173 (90), 172 (8), 155 (60), 129 (28), 116 (12), 101 (11), 85 (10), 73 (29), 71 (18), 60 (26), 57 (37), 56 (100), 43 (27). Hexyl octanoate had the following mass spectrum: 228 (>1), 157 (1), 145 (100), 127 (53), 115 (4), 101 (12), 85 (16), 84 (86), 73 (16), 69 (22), 61 (26), 57 (41), 56 (42), 55 (28), 43 (52).

Other reference standards were obtained commercially or

Table II. Volatile Constituents of Apricot: Vacuum Steam Distilled Blended Fruit

peak no.[a]	constituent	I^{DB-1} exptl	I^{DB-1} ref	approx concn,[b] μg/kg
1	2-methylbutanol[c]	730	729	1
4	2-hexanone	771	770	2
8	hexanal	779	772	220
9	3-hexanol[c]	784	784	1
10	2-hexanol	789	788	1
16	(*E*)-2-hexenal	831	827	730
18	(*Z*)-3-hexenol	844	843	56
19	(*E*)-2-hexenol	863	856	750
20	hexanol	867	860	740
22	butyl propanoate[c]	895	890	2
24	benzaldehyde	925	926	56
25	(*Z*)-2-heptenal[c]	931	927	6
26	butyl 2-methylpropanoate[c]	935	936	tr
31	6-methyl-5-hepten-2-one	967	966	4
36	butyl butanoate	982	982	23
38	ethyl hexanoate	986	986	tr
42	(*E*)-2-hexenyl acetate	997	994	16
45	phenylacetaldehyde	1002	1002	6
46	2,2,6-trimethylcyclohexanone[c]	1008	1008	2
48	butyl 2-methylbutanoate[c]	1030	1026	7
51	(*E*)-β-ocimene[c]	1041	1037	3
60	pentyl butanoate[c]	1079	1080	7
63	linalool	1086	1083	296
64	hexyl propanoate[c]	1086	1083	4
65	3-nonen-2-one	1114	1114	2
67	(*E*)-2-nonenal[c]	1134	1133	2
69	hexyl 2-methylpropanoate[c]	1137	1137	5
72	α-terpineol	1169	1170	49
73	(*Z*)-3-hexenyl butanoate[c]	1169	1170	tr
74	butyl hexanoate[c]	1176	1176	23
75	hexyl butanoate[c]	1179	1178	41
79	β-cyclocitral[c]	1190	1194	10
80	γ-octalactone	1205	1210	9
81	nerol	1208	1209	14
85	hexyl 2-methylbutanoate[c]	1224	1222	17
87	geraniol	1234	1234	40
88	2-methylbutyl hexanoate[c]	1236	1236	tr
91	pentyl hexanoate[c]	1272	1270	3
94	(*E*,*E*)-2,4-decadienal[c]	1286	1287	3
97	hexyl hexanoate[c]	1370	1369	11
99	3,4-didehydro-β-ionol[c]	1392	1397	10
102	dihydro-β-ionone[c]	1413	1414	8
103	γ-decalactone	1425	1422	492
105	geranylacetone[c]	1428	1428	6
107	δ-decalactone	1445	1447	40
108	epoxy-β-ionone[c]	1456	1456	2
109	β-ionone	1459	1462	14
110	dihydroactinidiolide	1473	1475	3
118	γ-dodecalactone	1632	1635	56

[a] The peak numbers correspond to the numbers in Figure 2. Mass spectra were consistent with those of authentic reference standards. [b] Only approximate concentrations since percent recoveries and FID response factors were not determined for each compound (assume all response factors of 1). tr represents concentration less than 1 ppb. [c] Identified for the first time in apricot.

received as gifts.

Odor Thresholds. Odor thresholds of GC purified standards were determined according to the procedure described by Guadagni and Buttery (1978) and Guadagni et al. (1973).

RESULTS AND DISCUSSION

The volatile constituents from fresh apricots were isolated by two headspace methods and by simultaneous vacuum steam distillation–extraction. The volatiles were analyzed by GC and GC–MS. Sample components were identified by comparison of the compound's Kovats index, *I* (Kovats, 1958), and mass spectrum with that of an authentic reference standard.

The intact apricots were sampled by two different headspace methods. The first method utilized small Tenax traps (0.7 g of Tenax), low sweep gas flow rates (50 mL/min), short sampling periods (15–30 min), and thermal desorption of trapped volatiles. This technique was useful for detecting low-boiling constituents that were obscured by solvent peaks. The second method involved large Tenax traps (10 g), fast sweep gas flow rates (3 L/min), long sampling times (22 h), and solvent elution (ether) of trapped constituents. This method was more suited to the analysis of higher boiling compounds and was less prone to artifact formation (all glass surfaces and minimal exposure to heat). The high air flow during trapping kept the sampling container well aerated; the fruit was maintained in good condition with no condensation observed in the interior of the vessel. The importance of high gas flows during fruit headspace sampling was discussed by Ismail et al. (1980) who felt these conditions were less conducive to microbial growth (which would contribute their own volatiles). Table I lists apricot headspace constituents isolated by ether elution. With the exception of early-eluting components, more constituents could be detected and identified by this procedure than by the thermal desorption method. A GC/FID chromatogram of apricot headspace volatiles (ether elution) is shown in Figure 1. Though many peaks overlapped or were incompletely separated at this concentration, dilution of the extract was effective in resolving most of the components. This procedure was used in determining the percent area values listed in Table I. These values should be considered as approximate since there were

Table III. Approximate Concentrations, Odor Thresholds in Water, and Odor Units of Some Apricot Constituents.

constituent[a]	approx concn,[b] μg/kg	odor threshold,[c] ppb	odor units (U_0)[d]
β-ionone	14	0.007[e]	2000
linalool	296	6[f]	49
γ-decalactone	492	11[g]	45
hexanal	220	5[e]	44
(*E*)-2-hexenal	730	17[e]	43
(*E*,*E*)-2,4-decadienal	3	0.07[e]	43
(*E*)-2-nonenal	2	0.08[e]	25
γ-dodecalactone	56	7[g]	8
β-cyclocitral	10	5	2
phenylacetaldehyde	6	4[e]	1.5
γ-octalactone	9	7[e]	1.3
geraniol	40	40	1.0
hexyl 2-methylbutanoate	17	22	0.8
(*Z*)-3-hexenol	56	70[e]	0.8
hexyl propanoate	4	8	0.5
hexyl 2-methylpropanoate	5	13	0.4
butyl 2-methylbutanoate	7	17	0.4
δ-decalactone	40	100[g]	0.4
hexanol	740	2500	0.3
butyl butanoate	23	100	0.23
benzaldehyde	56	350[e]	0.16
hexyl butanoate	41	250	0.16
α-terpineol	49	330	0.15
geranylacetone	6	60[e]	0.1
6-methyl-5-hepten-2-one	4	50[e]	0.08
pentyl butanoate	7	210	0.03
butyl hexanoate	23	700	0.03
2,2,6-trimethylcyclohexanone	2	100	0.02
epoxy-β-ionone	2	100	0.02
butyl propanoate	2	200	0.01
3-nonen-2-one	2	800	0.003
2-methylbutanol	1	300	0.003

[a] The constituents were isolated by vacuum steam distillation–extraction and are listed in descending order of their odor units. [b] Only approximate concentrations since percent recoveries and FID response factors were not determined for each compound (assume all response factors of 1). [c] Odor threshold in water. [d] U_0 = compound concentration divided by its odor threshold. [e] Buttery et al., 1971. [f] Buttery et al., 1969. [g] Engel et al., 1988.

constituents coeluting with solvent peaks and also sample breakthrough during trapping was not determined.

Many of the esters are reported for the first time as apricot constituents. Previous studies have generally involved blending of the fruit prior to sampling. This disruption of the fruit tissues may have caused changes in ester concentrations due to enzymic activity (Schreier et al., 1985). Guichard and Souty (1988) sampled apricot volatiles under enzymic inhibition (ammonium sulfate) and identified 19 esters, 18 of which had not been previously reported in apricot. It was hoped that headspace sampling of the intact fruit would help to identify compounds responsible for the pleasant fruity apricot aroma, i.e., esters. The variety of esters was notably absent in the vacuum steam distilled samples from blended apricots.

The esters were clearly the dominant constituents in the headspace sample. The major esters identified were butyl butanoate (29.66%), butyl hexanoate (24.54%), hexyl butanoate (11.78%), ethyl hexanoate (4.70%), hexyl hexanoate (4.12%), hexyl 2-methylbutanoate (3.50%), butyl 2-methylbutanoate (2.76%), ethyl butanoate (2.00%), pentyl butanoate (1.45%), and butyl propanoate (1.30%).

The hydrocarbon (*E,Z*)-1,3,5-undecatriene has been identified as an impact compound of Galbanum essential oil (Chretien-Bessiere et al., 1967; Naves, 1967) and pineapple (Berger et al., 1985a). Though the threshold of this hydrocarbon in water has not been reported, sniffing of the gas chromatographic effluent permitted detection in the picogram (1–2) range (Berger et al., 1985a). This potent odorant has been detected in various fruits and vegetables (Berger et al., (1985b).

The odor thresholds of some of the headspace constituents are also listed in Table I. Based on their odor threshold and their amount present in the headspace, the following compounds probably contribute to the intact apricot odor: ethyl butanoate, ethyl 2-methylbutanoate, butyl butanoate, ethyl hexanoate, butyl 2-methylbutanoate, hexyl 2-methylbutanoate, and γ-decalactone.

It appears that longer trapping times are necessary to detect the higher boiling compounds; no compounds eluting beyond C_{15} were detected with the heat desorption procedure though many of the compounds listed in Table I were additionally confirmed by this procedure.

Table II lists the apricot compounds identified in samples prepared by vacuum steam distillation–extraction. The concentrations listed in the table should be considered as only approximate values since the percent recoveries and FID response factors were not determined for the individual compounds. Figure 2 shows a GC/FID chromatogram of apricot volatiles obtained by vacuum steam distillation–extraction.

There was no attempt to inhibit the enzyme systems of the fruit. The enzymic formation of secondary volatiles caused by disruption of the fruit tissues (blending of the fruit) was reflected in the high level (>50% of the total volatiles) of C_6 lipid peroxidation products.

Benzaldehyde probably arises from the cyanogenic glycoside, amygdalin, a typical constituent of many *Prunus* species such as apricot.

Dihydroactinidiolide, β-cyclocitral, and dihydro-β-ionone can be regarded as carotenoid metabolism products (Ohloff, 1978). Dihydro-β-ionone has been found as a major constituent (15.7%) in the essential oil of the blossoms of *Osmanthus fragrans* Lour. (Sisido et al., 1967). It has also been reported in cassie (Demole et al., 1969), raspberry (Winter and Enggist, 1971), passion fruit (Winter and Kloti, 1972), tea (Yamanishi et al., 1973), arctic bramble (Kallio, 1976), and black chokeberry (Hirvi and Honkanen, 1985).

3,4-Didehydro-β-ionol has been found in quince fruit essential oil prepared by steam distillation (Ishihara et al., 1986). This labile compound appears to be the precursor of the bicyclic hydrocarbon 2,2,6,7-tetramethylbicyclo[4.3.0]nona-4,7,9(1)-triene as refluxing the ionol in acidic solution produced 80% conversion (Ishihara et al., 1986). The precursor of 3,4-didehydro-β-ionol has not yet been identified though Winterhalter and Schreier (1988) have discussed possible formation pathways. They have identified glycosidically bound 3-hydroxy-β-ionol as a likely precursor.

The monoterpene alcohols linalool, α-terpineol, nerol, citronellol, and geraniol have been shown to exist in glycosidically bound forms in apricot (Salles et al., 1988). With the exception of citronellol the previously mentioned monoterpene alcohols were identified in this study. Geraniol and nerol must exist predominantly in their bound forms as Guichard and Souty (1988) did not detect these compounds in apricot samples prepared under enzymic inhibition. Salles et al. (1988) have also found the glycosidically bound forms of the linalool oxides, benzyl alcohol and 2-phenylethanol, in apricot. These compounds have been previously reported in apricot (Tang and Jennings 1967, 1968; Rodriguez et al., 1980; Chairote et al., 1981; Guichard and Souty, 1988; Sharaf et al., 1989) though they were not identified in this study.

The relative contribution of various constituents to the blended apricot aroma was determined by calculating the number of odor units (U_0). The odor unit was defined by Guadagni et al. (1966) as the concentration of the compound divided by its odor threshold. This value gives some idea of the significance of the volatiles to the apricot aroma. Table III lists the odor units of some apricot constituents calculated from their concentrations and odor thresholds. Compounds are listed in descending order of their odor unit values. Despite its low concentration, β-ionone (14 ppb) appears to be an important apricot constituent due to its rather low odor threshold (0.007 ppb). Other important contributors include linalool, γ-decalactone, hexanal, (*E*)-2-hexenal, (*E,E*)-2,4-decadienal, (*E*)-2-nonenal, γ-decalactone, β-cyclocitral, phenylacetaldehyde, and γ-octalactone. δ-Octalactone was previously identified in the Blenheim variety (Tang and Jennings, 1968). It was not found in this study though it would have coeluted with other constituents on the nonpolar DB-1 column employed. With its relatively high odor threshold of 400 ppb, it probably does not contribute to the apricot aroma. β-Ionone and linalool may be responsible for the floral character of apricots while the lactones provide the fruity, peach, and coconut-like background odor (Spencer et al., 1978). The esters may also play a role in the fruity odor. Though their concentrations were below their odor thresholds, these values may not reflect their actual levels in the intact fruit. Schreier et al. (1985) have shown a decrease in the concentration of certain esters in papaya as the result of enzymic activity. The esters were the dominant constituents in the headspace sample yet were relatively minor components in samples prepared by vacuum steam distillation–extraction. An additional experiment to check the effect of enzymic inhibition on the concentration of apricot volatiles would help clarify the role of the esters to the total apricot odor.

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Registry No. Ethanol, 64-17-5; 2,3-butanedione, 431-03-8; trichloromethane, 67-66-3; ethyl acetate, 141-78-6; 2-methylpropanol, 78-83-1; butanol, 35296-72-1; ethyl propanoate, 105-37-3; methyl butanoate, 623-42-7; 2-methylbutanol, 137-32-6; ethyl 2-methylpropanoate, 97-62-1; methyl 2-methylbutanoate, 868-57-5; hexanal, 66-25-1; ethyl butanoate, 105-54-4; propyl propanoate, 106-36-5; butyl acetate, 123-86-4; 2-propyl butanoate, 638-11-9; ethyl 2-methylbutanoate, 7452-79-1; hexanol, 111-27-3; propyl butanoate, 105-66-8; ethyl pentanoate, 539-82-2; butyl propanoate, 590-01-2; pentyl acetate, 628-63-7; 2-methylpropyl 2-methylpropanoate, 97-85-8; methyl hexanoate, 106-70-7; propyl 2-methylbutanoate, 37064-20-3; butyl 2-methylpropanoate, 97-87-0; 2-methylpropyl butanoate, 539-90-2; 2-methylbutyl propanoate, 2438-20-2; butyl butanoate, 109-21-7; ethyl hexanoate, 123-66-0; 2-methylpropyl 2-methylbutanoate, 2445-67-2; hexyl acetate, 142-92-7; 2-methylbutyl 2-methylpropanoate, 2445-69-4; limonene, 138-86-3; butyl 2-methylbutanoate, 15706-73-7; butyl 3-methylbutanoate, 109-19-3; pentyl 2-methylpropanoate, 2445-72-9; (*E*)-β-ocimene, 3779-61-1; 3-methylbutyl butanoate, 106-27-4; 2-methylbutyl butanoate, 51115-64-1; pentyl butanoate, 540-18-1; propyl hexanoate, 626-77-7; ethyl heptanoate, 106-30-9; linalool, 78-70-6; hexyl propanoate, 2445-76-3; 2-methylbutyl 2-methylbutanoate, 2445-78-5; methyl octanoate, 111-11-5; pentyl 2-methylbutanoate, 68039-

26-9; hexyl 2-methylpropanoate, 2349-07-7; 2-methylpropyl hexanoate, 105-79-3; 2-methylbutyl pentanoate, 55590-83-5; 1(*E*,*Z*),3,5-undecatriene, 19883-27-3; ethyl (*E*)-4-octenoate, 78989-37-4; (*Z*)-3-hexenyl butanoate, 16491-36-4; butyl hexanoate, 626-82-4; hexyl butanoate, 2639-63-6; ethyl octanoate, 106-32-1; β-cyclocitral, 432-25-7; dodecane, 112-40-3; γ-octalactone, 104-50-7; (*Z*)-3-hexenyl 2-methylbutanoate, 53398-85-9; hexyl 2-methylbutanoate, 10032-15-2; 3-methylbutyl hexanoate, 2198-61-0; 2-methylbutyl hexanoate, 24551-95-9; pentyl hexanoate, 540-07-8; propyl octanoate, 624-13-5; tridecane, 629-50-5; 2-methylpropyl octanoate, 5461-06-3; ethyl (*Z*)-4-decenoate, 7367-84-2; hexyl hexanoate, 6378-65-0; butyl octanoate, 589-75-3; ethyl decanoate, 110-38-3; dihydro-β-ionone, 17283-81-7; γ-decalactone, 706-14-9; δ-decalactone, 705-86-2; dihydroactinidiolide, 17092-92-1; pentadecane, 629-62-9; hexyl benzoate, 6789-88-4; hexyl octanoate, 1117-55-1; butyl decanoate, 30673-36-0; ethyl dodecanoate, 106-33-2; tetradecanal, 124-25-4; γ-dodecalactone, 2305-05-7; hexadecanal, 629-80-1; ethyl hexadecanoate, 628-97-7; 2-hexanone, 591-78-6; 3-hexanol, 623-37-0; 2-hexanol, 626-93-7; (*E*)-2-hexenal, 6728-26-3; (*Z*)-3-hexenol, 928-96-1; (*E*)-2-hexenol, 928-95-0; benzaldehyde, 100-52-7; (*Z*)-2-heptanal, 57266-86-1; 6-methyl-5-hepten-2-one, 110-93-0; (*E*)-2-hexenyl acetate, 2497-18-9; phenylacetaldehyde, 122-78-1; 2,2,6-trimethylcyclohexanone, 2408-37-9; 3-nonen-2-one, 14309-57-0; (*E*)-2-nonenal, 18829-56-6; α-terpineol, 10482-56-1; nerol, 106-25-2; geraniol, 106-24-1; (*E*,*E*)-2,4-decadienal, 25152-84-5; 3,4-didehydro-β-ionol, 3293-47-8; geranylacetone, 3796-70-1; epoxy-β-ionone, 36340-49-5; β-ionone, 79-77-6.

Determination of the Protein Activity of Corn Zeins in Alkaline Solutions from ^{1}H Nuclear Spin Relaxation Data as a Function of Concentration and Heat Treatments

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A marked nonlinear concentration dependence was observed for 10-MHz ^{1}H NMR transverse relaxation rates of corn zeins in solution at pH 11.5 for both unheated and heat-treated solutions that were cooled to room temperature. Simplex and Gauss–Newton nonlinear regression analyses of the NMR transverse relaxation data were employed to calculate the average virial expansion coefficients of protein activity. Two different virial expansions were found to fit the entire concentration range of the NMR data. The first model (A) contains three virial coefficients, B_0, $B_{3/2}$, and B_2, for the c_p, $c_p^{3/2}$, and c_p^2 terms, respectively, where c_p is corn protein concentration up to 80% (w/w). The second model (B) included four virial coefficients, B_0, B_2, B_3, and B_4, for the c_p, c_p^2, c_p^3, and c_p^4 terms, respectively, with slightly improved standard deviations over the first model. In the lower concentration range, up to 30%, an improved fit was obtained with only one virial coefficient, $B_0 = 2.46 \pm 0.18$ mL/g, which is significantly lower than the value of 6.9 mL/g obtained from the best fit over the entire concentration range with model B. All three models yielded a decrease in the *average* protein activities and the average transverse relaxation rate ($1/T_2$) of bound water after heat treatments. Since the heated samples were measured at room temperature after cooking, the changes in the average virial coefficients reflect *irreversible* protein conformational changes induced by heating. The alternating signs of the virial coefficients in both models indicate the presence of both repulsive and attractive interactions among corn zeins. A plot of the ratio T_2/T_1 as a function of corn protein concentration indicates that cross-relaxation is much less significant than chemical exchange for these samples. The decreasing T_2/T_1 ratio with increasing protein concentration suggests the presence of certain, relatively slow, water motions that are detected by the T_2 relaxation measurements and not by T_1. For this reason, the T_2 relaxation dependence on concentration is much steeper than the T_1 relaxation dependence.

1. INTRODUCTION

With the current emphasis of corn wet-milling focused on the production of high-fructose corn syrups and corn starch, there is an increasing need to find uses for the corn gluten byproducts, particularly zein. This prolamine protein fraction of corn is currently used as a coating agent in pharmaceuticals and as animal feed due to the low "quality" of the product (Shroder and Heiman, 1970). In order to exploit this byproduct for its possible use in human foods, zein's physicochemical properties must be characterized so that technologists may predict and control its behavior during food processing. Nondestructive, useful techniques for conformation, composition, and hydration analyses of proteins are provided by nuclear magnetic resonance (NMR) relaxation and spectroscopy. High-resolution NMR and relaxation techniques have already been used to study other food systems such as wheat (Baianu et al., 1982), corn (Augustine and Baianu, 1987), and soy proteins (Kakalis and Baianu, 1989).

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