

New Esteric Components in the Volatiles of Banana Fruit (*Musa sapientum* L.)

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The volatiles of the Delicious banana were analyzed by gas chromatography and a combined gas chromatograph-mass spectrometer. Eighty-six components were identified for the first time in banana among a total of 152 components. The acetate, butanoate, 3-methylbutanoate, and pentanoate esters of hex-4(*Z*)-en-1-ol, hept-4(*Z*)-en-2-ol, oct-4(*Z*)-en-1-ol, oct-5(*Z*)-en-1-ol, and dec-4(*Z*)-en-1-ol were newly found. Moreover, hexan-2-yl butanoate, 3-methylbutanoate, and pentanoate were also detected in company with pentan-2-yl esters. The odor and taste evaluation of these esters was carried out.

INTRODUCTION

One of the most common tropical fruits in Japan is the banana. The import volume of this fruit to Japan from the Philippines (78%), Ecuador (17%), and Taiwan (4%) is around 0.76 million tons for the past 3 years (Nichi-En-Ren, 1992). Bananas are cultivated in Okinawa, Japan, but the production is small (about 600 tons). Bananas are almost entirely consumed as a table fruit. Their import volume is ranked third in Japan below the local production of citrus (1.65 million tons) and apples (1.05 million tons), which are mostly sold at domestic markets.

Bananas are nutritious with a pleasant flavor and are widely consumed throughout the world. Analytical research on the aroma compounds of this fruit has been carried out for 30 years. The volatile aroma compounds have been identified by capillary column gas chromatography and combined gas chromatograph-mass spectrometry. The results of this previous research were summarized and published by TNO of the Netherlands (Maarse, 1989) and reviewed (Engel et al., 1991).

The aroma compounds of banana fruit were mainly 3-methylbutyl esters (60%), acetates (55%), and butanoates (20%) (Tressl et al., 1970). Furthermore, the fruity, estery odor is reported to be due to acetates and butanoates of butanol, 2-methylpropanol, and pentan-2-ol and hexyl acetate (Berger et al., 1991).

Unfortunately, further analytical research on banana fruit aroma has not occurred in the past decade. A more detailed approach is needed for understanding the natural banana flavor. It is important to identify the trace compounds contributing significantly to banana aroma.

MATERIALS AND METHODS

Fruit. Philippine bananas (Delmonte, Cavendish cultivar), Taiwanese bananas (Sen-nin cultivar), and Delicious bananas (hybrid between Philippine and Taiwanese) were purchased at a fruit shop in Japan. These three bananas are common in Japan.

Isolation of Volatiles. The edible part (200 g) of banana fruit was treated in a modified simultaneous distillation and extraction (SDE) apparatus (Flath and Forrey, 1977). The volatiles were extracted with a mixture of distilled pentane (14 g) and dichloromethane (6 g) for 15 min. The extract was concentrated at atmospheric pressure as described previously (Shiota et al., 1988).

Gas Chromatography (GC). A 30 m \times 0.25 mm i.d. DB-1 fused silica capillary column (0.25- μ m film thickness; J&W Scientific, Folsom, CA) was used for gas chromatography. The column was installed in a Hewlett-Packard 5890A GC equipped with injection splitter (split ratio 1:120) and flame ionization detector. The oven temperature was programmed from 50 °C

(5-min isothermal) to 240 °C at 3 °C/min. The temperatures of the injector and detector were 250 and 280 °C, respectively. Helium (1 mL/min) was used as carrier gas. Injection volume was 0.5 μ L. Peak area percentages were calculated automatically with a Hewlett-Packard 3392A integrator.

Gas Chromatography-Mass Spectrometry (GC-MS). A Hitachi 663 GC was combined with a Hitachi M-80A mass spectrometer (electron impact mode) and a Hitachi M-0101 data processor. A 60 m \times 0.25 mm i.d. DB-1 column (0.25- μ m film thickness; J&W) was used. The temperature program was isothermal for 5 min at 75 °C and was then raised to 240 °C at 3 °C/min. The temperature of the injector was 250 °C. The column outlet was inserted directly into the ion source block. The mass spectra were recorded at an ionization voltage of 20 eV at an ion source temperature of 180 °C and with a speed of 0.445 scan/s over a mass range m/z 35-350. Retention indices of compounds were calculated with the Hitachi M-0101 data processor on the basis of retention times of normal alkanes (C₆-C₂₅).

Identification of Components. Identification of components was confirmed by comparison of the experimental retention index and mass spectrum with that of an authentic reference standard. In the absence of matched reference spectra, samples of proposed compounds were obtained from reagent houses or synthesized if they were not available.

Column Chromatography. The volatile oil obtained by steam distillation of banana fruit was fractionated on a column packed with silica gel 60 (Merck, 0.063-0.200 mm) with diethyl ether solution (2-20%) in pentane into eight groups. The compounds synthesized were purified for odor and taste evaluation by using silica gel 60 (Nacalai tesque, Japan) with 4% diethyl ether in pentane.

Acetate, Butanoate, 3-Methylbutanoate, and Pentanoate of Pentan-2-ol. The mixture of pentan-2-ol (Tokyo Kasei, Japan), pyridine, and pentane was cooled to 5-10 °C, and acid chloride in pentane solution was added dropwise to it, as the temperature was maintained. The mixture was allowed to stand overnight and then treated in the usual manner (Shiota, 1991). Pentan-2-yl acetate: bp 133-135 °C; MS m/z (%) 43 (100), 87 (19), 70 (12), 55 (9), 42 (5), 45 (5), 61 (2). Pentan-2-yl butanoate: bp 88-90 °C/45 mmHg; MS m/z (%) 71 (100), 43 (67), 70 (23), 55 (12), 89 (12), 41 (7), 42 (7), 88 (7), 115 (7), 60 (5), 45 (2), 72 (2), 87 (2). Pentan-2-yl 3-methylbutanoate: bp 62-63 °C/7 mmHg; MS m/z (%) 85 (100), 70 (79), 57 (65), 43 (54), 103 (45), 71 (40), 60 (38), 55 (26), 102 (24), 129 (17), 41 (13), 86 (13). Pentan-2-yl pentanoate: bp 66 °C/7 mmHg; MS m/z (%) 85 (100), 70 (79), 57 (65), 43 (54), 103 (45), 71 (40), 60 (38), 55 (26), 102 (24), 129 (17), 41 (13), 86 (13).

Acetate, Butanoate, 3-Methylbutanoate, and Pentanoate of Hexan-2-ol. These esters were prepared from hexan-2-ol (Tokyo Kasei) and acid chloride in a similar manner as described above. Hexan-2-yl acetate: bp 154-155 °C; MS m/z (%) 43 (100), 87 (65), 84 (26), 55 (19), 56 (19), 58 (12), 69 (12), 61 (9), 103 (2), 129 (2). Hexan-2-yl butanoate: bp 66 °C/7 mmHg; MS m/z (%)

Table I. Volatile Components in Delicious Bananas Cultivated in the Philippines

compound	retention time, min	%	R.I. ^a		compound	retention time, min	%	R.I. ^a	
			exp	auth				exp	auth
ethyl acetate	1.50	0.39	580	590	oct-4(Z)-en-1-ol	13.48	0.35	1044	1041
2-methylpropanol	1.56	0.16	602	606	pentan-2-yl 3-methylbutanoate	14.42	0.13	1059	1060
3-methylbutanal	1.66	0.70	622	628	nonan-2-one	14.74	0.06	1070	1070
butan-1-ol	1.74	0.24	633	634	unidentified	15.06	0.12		
pentan-2-one	1.87	23.30	655	652	3-methylbutyl 2-methylbutanoate	15.43	0.32	1085	1086
pentan-2-ol	1.98	2.54	672	674	unidentified	15.58	0.16		
3-methylbutanol	2.31	4.23	710	720	3-methylbutyl 3-methylbutanoate	15.85	2.00	1090	1085
2-methylpropyl acetate	2.77	1.30	745	744	hexan-2-yl butanoate*	16.55	0.17	1101	1101
hexan-2-one	2.91	0.15	759	761	3-methylbutyl pentanoate	18.12	0.16	1134	1134
hexanal	3.06	0.26	769	775	unidentified	19.57	0.16		
unidentified	3.21	0.14			unidentified	19.81	0.31		
butyl acetate	3.45	1.49	789	794	hexyl butanoate	20.10	0.96	1173	1175
unidentified	3.48	0.58			oct-4(Z)-en-1-yl acetate	20.25	0.19	1177	1174
furfural*	4.09	0.60	800	805	unidentified	20.42	0.13		
pentan-2-yl acetate	4.41	6.61	827	829	hept-4(E)-en-2-yl butanoate*	20.99	0.09	1190	1188
unidentified	4.85	0.46			hept-4(Z)-en-2-yl butanoate*	21.28		1197	1197
hexanol	5.04	4.51	849	850	heptan-2-yl butanoate	21.28	2.38	1197	1194
3-methylbutyl acetate	5.22	5.71	853	856	hexyl 3-methylbutanoate	22.56	0.39	1224	1227
heptan-2-one	5.45	1.50	864	867	3-methylbutyl hexanoate	22.90	0.79	1232	1234
hept-4(Z)-en-2-ol	5.57	3.39	868	869	unidentified	23.13	0.23		
heptan-2-ol	6.09	0.43	880	880	hept-4(Z)-en-2-yl 3-methylbutanoate	23.66	0.12	1247	1248
hept-2(E)-4-one*	6.55	0.32	899	899	unidentified	24.03	0.22		
hept-3(E)-en-2-one*	7.04	0.57	909	909	eugenol	27.03	1.01	1333	1338
hexan-2-yl acetate	7.69	0.15	924	925	oct-4(Z)-en-1-yl butanoate*	27.17	0.31	1357	1358
5-methylfurfural*	8.01	0.13	929	927	unidentified	28.68	0.27		
2-methylpropyl butanoate	8.40	1.14	935	935	hexyl hexanoate	29.17	0.50	1367	1373
butyl butanoate	10.25	1.74	975	975	unidentified	30.09	0.11		
2-methylpropyl 3-methylbutanoate	10.84	0.07	989	991	oct-4(Z)-en-1-yl 3-methylbutanoate	30.85	0.17	1407	1404
hexyl acetate	11.08	0.64	992	995	oct-5(Z)-en-1-yl 3-methylbutanoate*	31.31	0.07	1416	1414
3-methylbutyl 2-methylpropanoate	11.17	0.11	995	998	elemicin	35.52	2.24	1519	1530
phenylacetaldehyde*	11.32	0.10	1012	1017	2,6-dimethoxy-4-(2-propenyl)phenol	37.19	1.43	1569	1579
pentan-2-yl butanoate	11.86	3.61	1008	1012	palmitoleic acid	50.46	0.39	**	**
hept-4(E)-en-2-yl acetate*	12.28	0.11	1014	1013	palmitic acid	51.37	1.25	**	**
hept-4(Z)-en-2-yl acetate	12.61		1023	1023	linolenic acid	56.52			
heptan-2-yl acetate	12.61	1.83	1023	1021	linoleic acid	56.52	1.06	**	**
butyl 3-methylbutanoate	12.69	0.59	1028	1029	unidentified	56.78	0.28		
unidentified	12.80	0.24							
3-methylbutyl butanoate	13.35	7.90	1039	1038	total			96.47	

^a R.I., retention index: exp, experimental value; auth, value of the authentic compound. * Newly identified in this study. ** Board peak in gas chromatogram of GC-MS.

Table II. Odor Description of Fractionated Volatile Oil from Banana Fruit by Steam Distillation

fraction no.	volume ratio of Et ₂ O/pentane	odor description	concentrated oil weight, ^a g
Fr-1	0-2/98-100	faint, paper-like, vegetable-like, oily	0.02
Fr-2	4/96	strong, pineapple-like, green	0.30
Fr-3	4/96	baked sweet potato-like, sugary, burnt	0.06
Fr-4	6/94	earthy, esteric, powdery	0.06
Fr-5	6-8/94-92	potato-like, cinnamic, sweet	0.08
Fr-6	8/92	cooked, sweet potato-like, methional-like top note	0.06
Fr-7	10-20/90-80	sweet, burnt, metallic, fishy	0.07
Fr-8	*	banana-like	0.10

* Residue silica gel was extracted with 100 mL of CH₂Cl₂ after elution of 20% ether/pentane solution. ^a From 0.9 g of volatile oil.

71 (100), 43 (67), 84 (28), 115 (19), 55 (16), 89 (16), 85 (14), 56 (14), 88 (12), 69 (7), 60 (5), 101 (2). Hexan-2-yl 3-methylbutanoate: bp 73-74 °C/7 mmHg; MS *m/z* (%) 85 (100), 57 (44), 84 (35), 43 (33), 103 (21), 60 (19), 56 (16), 102 (16), 129 (12), 55 (9), 87 (9), 69 (7), 86 (7). Hexan-2-yl pentanoate: bp 80 °C/7 mmHg; MS *m/z* (%) 85 (100), 57 (33), 84 (33), 43 (30), 103 (19), 56 (14), 60 (14), 102 (14), 55 (9), 129 (9), 69 (7), 86 (7), 73 (5).

Acetate, Butanoate, 3-Methylbutanoate, and Pentanoate of Hept-4(Z)-en-2-ol. The diethyl acetal of hex-3(Z)-enal (Bedoukian Research, Danbury, CT) was treated with a small portion of oxalic acid and hydroquinone in aqueous acetone to free hex-3(Z)-enal (Winter, 1963). Hex-3(Z)-enal solution in diethyl ether was added to a methylmagnesium iodide solution in diethyl ether. Hept-4(Z)-en-2-ol was isolated in the usual manner (Buttery, 1979). Hept-4(Z)-en-2-yl esters were prepared by a similar manner to that described above and purified with silica gel chromatography. Hept-4(Z)-en-2-yl acetate: MS *m/z* (%) 43 (100), 96 (70), 81 (67), 87 (28), 55 (14), 41 (8), 54 (8), 97

(6), 67 (4), 68 (3), 69 (2), 112 (2). Hept-4(Z)-en-2-yl butanoate: MS *m/z* (%) 71 (100), 96 (53), 43 (44), 81 (33), 55 (12), 97 (7), 54 (5), 72 (5), 115 (2). Hept-4(Z)-en-2-yl 3-methylbutanoate: MS *m/z* (%) 85 (100), 96 (77), 57 (67), 81 (42), 55 (21), 97 (14), 41 (7), 86 (5), 43 (2), 54 (2), 129 (2). Hept-4(Z)-en-2-yl pentanoate: MS *m/z* (%) 85 (100), 57 (35), 81 (28), 55 (9), 96 (9), 97 (7), 41 (5), 86 (5), 54 (2), 67 (2), 129 (2).

Acetate, Butanoate, 3-Methylbutanoate, and Pentanoate of Oct-4(Z)-en-1-ol. These esters were prepared from oct-4(Z)-en-1-ol (Bedoukian) in a similar manner as described above. Oct-4(Z)-en-1-yl acetate: MS *m/z* (%) 81 (100), 68 (79), 67 (49), 43 (47), 54 (40), 110 (35), 82 (30), 55 (19), 95 (19), 41 (9), 69 (9). Oct-4(Z)-en-1-yl butanoate: MS *m/z* (%) 81 (100), 68 (93), 110 (44), 54 (37), 67 (37), 82 (35), 71 (30), 43 (28), 95 (21), 69 (19), 55 (14), 41 (12). Oct-4(Z)-en-1-yl 3-methylbutanoate: MS *m/z* (%) 81 (100), 68 (93), 110 (56), 54 (40), 82 (35), 67 (33), 57 (30), 69 (23), 55 (19), 85 (19), 95 (19), 41 (12), 43 (5). Oct-4(Z)-en-1-yl

Table III. Volatile Components in Delicious Bananas Identified by Treatment with Column Chromatography

compound	R.I. exp (auth)	fraction no. (peak area, %)
Hydrocarbons		
toluene*	748 (752)	Fr-1 (3.6) ^a
ethylbenzene*	846 (850)	Fr-1
<i>p</i> -xylene*	854 (859)	Fr-1 (1.4)
styrene*	873 (880)	Fr-1
<i>o</i> -xylene*	879 (885)	Fr-1
1,2,4-trimethylbenzene*	956 (957)	Fr-1
2-pentylfuran*	976 (975)	Fr-1
δ -3-carene*	1006 (1006)	Fr-1 (1.2)
<i>p</i> -cymene*	1012 (1014)	Fr-1
limonene*	1022 (1024)	Fr-1 (5.0)
γ -terpinene*	1049 (1051)	Fr-1
β -caryophyllene*	1423 (1423)	Fr-1
δ -cadinene*	1516 (1519)	Fr-1
octane*	793 (800)	Fr-1 (1.7)
dec-1-ene*	985 (990)	Fr-1
undecane*	1097 (1100)	Fr-1
dodec-1-ene*	1186 (1193)	Fr-1 (4.1)
dodecane*	1197 (1200)	Fr-1
tridecane*	1296 (1300)	Fr-1
tetradec-1-ene*	1385 (1394)	Fr-1 (5.6)
tetradecane*	1396 (1400)	Fr-1 (3.5)
hexadec-1-ene*	1584 (1594)	Fr-1 (4.8)
hexadecane*	1595 (1600)	Fr-1 (6.2)
heptadecane*	1692 (1700)	Fr-1 (1.3)
octadec-1-ene*	1783 (1795)	Fr-1 (3.7)
octadecane*	1792 (1800)	Fr-1 (5.1)
nonadecane*	1890 (1900)	Fr-1
eicos-1-ene*	1982 (1993)	Fr-1 (1.4)
eicosane*	1991 (2000)	Fr-1 (2.1)
heneicosane*	2092 (2100)	Fr-1
docos-1-ene*	2183 (2193)	Fr-1
docosane*	2191 (2200)	Fr-1
tricosane*	2292 (2300)	Fr-1 (1.1)
tetracosane*	2391 (2400)	Fr-1
pentacosane*	2492 (2500)	Fr-1
Esters		
ethyl acetate	580 (590)	Fr-3, -4
propyl acetate	683 (689)	Fr-3, -4
2-methylpropyl acetate	743 (744)	Fr-2, -4, Fr-3 (4.2)
2-ketobutan-3-yl acetate* (acetoin acetate)	852 (856)	Fr-7
butyl acetate	786 (794)	Fr-2, -3
pentan-2-yl acetate	826 (829)	Fr-2 (7.0), Fr-3 (20.0)
3-methylbutyl acetate	850 (856)	Fr-2 (3.4), Fr-3 (30.8)
pentyl acetate	890 (894)	Fr-3
hexan-2-yl acetate	923 (925)	Fr-2, -3
hex-3(<i>Z</i>)-en-1-yl acetate*	979 (980)	Fr-3
hexyl acetate	991 (995)	Fr-2, Fr-3 (4.4)
hex-4(<i>Z</i>)-en-1-yl acetate	998 (1001)	Fr-3
hept-4(<i>E</i>)-en-2-yl acetate*	1014 (1013)	Fr-2, -3
heptan-2-yl acetate	1024 (1021)	Fr-2, -3
hept-4(<i>Z</i>)-en-2-yl acetate	1024 (1022)	Fr-2 (4.7), Fr-3 (12.2)
oct-4(<i>Z</i>)-en-1-yl acetate	1177 (1174)	Fr-2, Fr-3 (1.2)
oct-4(<i>E</i>)-en-1-yl acetate*	1180 (1178)	Fr-3
oct-5(<i>Z</i>)-en-1-yl acetate	1185 (1184)	Fr-3
octyl acetate	1190 (1197)	Fr-3
dec-4(<i>Z</i>)-en-1-yl acetate*	1367 (1370)	Fr-3
2-methylpropyl butanoate	935 (939)	Fr-2 (3.2), Fr-3
butyl butanoate	975 (975)	Fr-2 (5.2), Fr-3 (1.1)
2-ketobutan-3-yl butanoate (acetoin butanoate)	1036 (1038)	Fr-7
pentan-2-yl butanoate	1009 (1012)	Fr-2 (11.8), Fr-3 (1.1)
3-methylbutyl butanoate	1042 (1038)	Fr-2 (26.6), Fr-3 (3.4)
hexan-2-yl butanoate*	1101 (1101)	Fr-2
hex-3(<i>Z</i>)-enyl butanoate*	1163 (1162)	Fr-2
hexyl butanoate	1173 (1175)	Fr-2 (3.5), Fr-3
hex-4(<i>Z</i>)-en-1-yl butanoate*	1180 (1177)	Fr-2
hept-4(<i>E</i>)-en-2-yl butanoate*	1189 (1188)	Fr-2
heptan-2-yl butanoate	1198 (1194)	Fr-2
hept-4(<i>Z</i>)-en-2-yl butanoate*	1198 (1197)	Fr-2 (9.0), Fr-3
oct-4(<i>Z</i>)-en-1-yl butanoate*	1356 (1354)	Fr-2
oct-4(<i>E</i>)-en-1-yl butanoate*	1360 (1358)	Fr-2
dec-4(<i>Z</i>)-en-1-yl butanoate*	1544 (1550)	Fr-2
3-methylbutyl 2-methylpropanoate	994 (998)	Fr-2
3-methylbutyl 2-methylbutanoate	1084 (1086)	Fr-2

Table III (Continued)

compound	R.I. exp (auth)	fraction no. (peak area, %)
Esters		
pentan-2-yl 3-methylbut-2(<i>E</i>)-enoate* (crotonate)	1049 (1049)	Fr-2
2-methylpropyl 3-methylbutanoate	988 (991)	Fr-2
butyl 3-methylbutanoate	1028 (1029)	Fr-2, -3
pentan-2-yl 3-methylbutanoate	1059 (1060)	Fr-2
3-methylbutyl 3-methylbutanoate	1090 (1085)	Fr-2 (6.9), Fr-3
hexan-2-yl 3-methylbutanoate*	1152 (1153)	Fr-2
hexyl 3-methylbutanoate	1224 (1227)	Fr-2 (1.5), Fr-3
hept-4(<i>E</i>)-en-2-yl 3-methylbutanoate*	1241 (1238)	Fr-2
hept-4(<i>Z</i>)-en-2-yl 3-methylbutanoate*	1246 (1248)	Fr-2
oct-4(<i>Z</i>)-en-1-yl 3-methylbutanoate*	1406 (1404)	Fr-2
oct-4(<i>E</i>)-en-1-yl 3-methylbutanoate*	1410 (1408)	Fr-2
oct-5(<i>Z</i>)-en-1-yl 3-methylbutanoate*	1415 (1414)	Fr-2
octyl 3-methylbutanoate*	1420 (1420)	Fr-2
dec-4(<i>Z</i>)-en-1-yl 3-methylbutanoate*	1594 (1599)	Fr-2
3-methylbutyl pentanoate	1133 (1134)	Fr-1, -2
hexyl pentanoate*	1268 (1272)	Fr-2
heptan-2-yl pentanoate*	1289 (1292)	Fr-2
hept-4(<i>Z</i>)-en-2-yl pentanoate*	1289 (1290)	Fr-2
hexyl hexanoate	1366 (1373)	Fr-2 (1.5)
3-methylbutyl hexanoate	1232 (1234)	Fr-2 (3.1), Fr-3
3-methylbutyl hex-2(<i>E</i>)-enoate*	1277 (1282)	Fr-2
2-phenylethyl butanoate*	1411 (1415)	Fr-3
3-phenylpropyl butanoate*	1529 (1536)	Fr-3
2-phenylethyl 3-methylbutanoate*	1465 (1468)	Fr-3
3-phenylpropyl 3-methylbutanoate*	1582 (1594)	Fr-3
2-phenylethyl hexanoate*	1613 (1619)	Fr-3
dibutyl phthalate ^b	1912 (1923)	Fr-1, -5, -6, -7
Carbonyls		
3-methylbutanal	619 (628)	Fr-2, -3
hexanal	768 (775)	Fr-2, -3
hex-2(<i>Z</i>)-enal*	817 (814)	Fr-4
hex-2(<i>E</i>)-enal	826 (825)	Fr-4 (4.5), Fr-5
hexa-2,4-dienal*	878 (883)	Fr-6
phenylacetaldehyde*	1012 (1017)	Fr-4, Fr-5
octa-2,4-dienal*	1082 (1084)	Fr-5
furfural*	801 (805)	Fr-6 (3.4), -7
5-methylfurfural*	929 (927)	Fr-7 (1.8)
pentan-2-one	658 (652)	Fr-4 (44.2), Fr-5 (12.9), Fr-6
penta-2,3-dione*	652 (651)	Fr-3
pent-3-en-2-one*	710 (707)	Fr-7 (6.8)
hexan-2-one	761 (761)	Fr-4 (1.2), Fr-5
heptan-2-one	864 (867)	Fr-3, Fr-4 (18.0)
5-methylhex-3-en-2-one**	875	Fr-4 (13.5)
hept-4(<i>Z</i>)-en-2-one*	868 (874)	Fr-5 (2.6)
hept-2(<i>Z</i>)-en-4-one*	902 (899)	Fr-4 (3.5), Fr-5
hept-3(<i>E</i>)-en-2-one*	911 (909)	Fr-5 (1.2), Fr-6 (27.6), Fr-7
oct-1-en-3-one*	952 (958)	Fr-3
nonan-2-one	1069 (1070)	Fr-3, Fr-4
undecan-2-one	1272 (1275)	Fr-3, Fr-4 (1.1)
tridecan-2-one*	1473 (1479)	Fr-3, Fr-4
pentadecan-2-one*	1675 (1682)	Fr-3, Fr-4
heptadecan-2-one*	1875 (1884)	Fr-3
α -ionone*	1411 (1414)	Fr-4
acetophenone*	1038 (1043)	Fr-4
Alcohols		
2-methylpropanol (isobutyl alcohol)	605 (606)	Fr-8
butan-1-ol	633 (634)	Fr-8
3-methylbutanol (isoamyl alcohol)	713 (720)	Fr-8 (12.2)
pentan-2-ol	674 (674)	Fr-7, Fr-8 (5.8)
hex-2(<i>E</i>)-en-1-ol	843 (844)	Fr-7
hexan-2-ol	777 (777)	Fr-8
hex-3(<i>E</i>)-en-1-ol	829 (826)	Fr-8
hex-3(<i>Z</i>)-en-1-ol	834 (836)	Fr-8
hexan-1-ol	851 (850)	Fr-8 (25.2)
hex-4(<i>Z</i>)-en-1-ol	856 (857)	Fr-8
heptan-2-ol	881 (880)	Fr-7, Fr-8
hept-4(<i>E</i>)-en-2-ol*	863 (863)	Fr-8
hept-4(<i>Z</i>)-en-2-ol	869 (869)	Fr-7 (5.2), Fr-8 (5.5)
oct-4(<i>Z</i>)-en-1-ol	1043 (1045)	Fr-8 (2.5)
oct-5(<i>Z</i>)-en-1-ol	1051 (1048)	Fr-8 (0.4)
dec-4(<i>Z</i>)-en-1-ol*	1240 (1242)	Fr-8 (1.9)

Table III (Continued)

compound	R.I. exp (auth)	fraction no. [peak area (%)]
Phenols and Phenol Methyl Ethers		
2-methoxy-4-methylphenol* (creosol)	1169 (1171)	Fr-5
2-methoxy-4-(2-ethenyl)phenol* (<i>p</i> -vinylguaiacol)	1289 (1286)	Fr-5 (1.0)
2-methoxy-4-(2-propenyl)phenol (eugenol)	1343 (1338)	Fr-5 (57.3), Fr-6 (14.4), Fr-7
1,2-dimethoxy-4-methylbenzene	1208 (1207)	Fr-5
1,2-dimethoxy-4-(2-propenyl)benzene (methyleugenol)	1373 (1373)	Fr-5
1,2,6-trimethoxy-4-(2-propenyl)benzene (elemicin)	1531 (1530)	Fr-7 (57.9)
2,6-dimethoxy-4-(2-propenyl)phenol	1575 (1579)	Fr-8 (18.9)
Acids		
palmitic acid	c	Fr-8 (1.9)
linoleic acid	c	Fr-8 (1.2)
linolenic acid	c	Fr-8
oleic acid	c	Fr-8

^a Peak area percentage in the fraction. ^b Contaminated in water. ^c Not described for a broad peak in the chromatogram. * Newly identified in this study. ** Tentative.

pentanoate: MS *m/z* (%) 68 (100), 81 (95), 110 (56), 82 (40), 54 (37), 67 (33), 57 (26), 95 (21), 69 (19), 85 (19), 55 (14), 41 (9).

Acetate and 3-Methylbutanoate of Oct-5(*Z*)-en-1-ol. These esters were prepared from oct-5(*Z*)-en-1-ol (Bedoukian) in a similar manner. Oct-5(*Z*)-en-1-yl acetate: MS *m/z* (%) 82 (100), 67 (79), 43 (60), 81 (47), 68 (44), 110 (28), 41 (21), 56 (19), 69 (14), 95 (14), 55 (12). Oct-5(*Z*)-en-1-yl 3-methylbutanoate: MS *m/z* (%) 82 (100), 67 (37), 85 (33), 57 (30), 81 (30), 68 (28), 110 (28), 69 (19), 41 (9), 65 (9), 95 (9), 103 (2).

Acetate, Butanoate, and 3-Methylbutanoate of Dec-4(*Z*)-en-1-ol. These esters were prepared from dec-4(*Z*)-en-1-ol (Bedoukian) in a similar manner. Dec-4(*Z*)-en-1-yl acetate: MS *m/z* (%) 68 (100), 81 (65), 67 (53), 54 (44), 43 (40), 82 (33), 55 (26), 95 (26), 96 (21), 138 (21), 138 (21), 69 (14), 110 (14), 109 (12). Dec-4(*Z*)-en-1-yl butanoate: MS *m/z* (%) 68 (100), 81 (53), 54 (35), 67 (35), 82 (33), 71 (26), 96 (26), 138 (26), 43 (21), 95 (21), 55 (19), 110 (14), 69 (12), 109 (9). Dec-4(*Z*)-en-1-yl 3-methylbutanoate: MS *m/z* (%) 68 (100), 81 (53), 82 (37), 67 (35), 54 (33), 96 (30), 138 (28), 57 (26), 95 (21), 55 (19), 110 (19), 69 (14), 85 (14), 109 (12).

RESULTS AND DISCUSSION

Analysis of Volatiles. Flavor evaluation of commercial banana fruit in Japan showed that Cavendish bananas cultivated in the Philippines had a beautiful appearance but slightly weak aroma. Taiwanese bananas had not so good an appearance but had a strong aroma and sweet taste. The Delicious banana (commercial brand), which is a hybrid between the Philippine and Taiwanese, is cultivated in the Philippines solely for export to Japan and handled with very careful attention to hold its position as an excellent fruit in the Japanese market. The Delicious banana had good aroma and taste and was therefore used for this study. The volatiles were separated with SDE as described under Materials and Methods and analyzed with GC and GC-MS. The results are summarized in Table I.

The major component in the volatiles was pentan-2-one (23.3%), with other carbonyl compounds present in small amounts. Apart from pentan-2-one, the other principal components were esters, which would contribute significantly to banana aroma. In addition to common esters such as 3-methylbutyl (isoamyl) and 2-methylpropyl (isobutyl), uncommon esters of pentan-2-ol, hexan-2-ol, hept-4(*Z*)-en-2-ol, oct-4(*Z*)-en-1-ol, and oct-5(*Z*)-en-1-ol were detected in the volatiles. They would probably be responsible for the fruity estery aroma of the Delicious banana. The other components were alcohols which constitute the alcohol moiety of such esters. These alcoholic compounds would contribute to the succulent character of the banana aroma.

For further investigation of volatile compounds in banana fruit, a scaled-up steam distillation of the Delicious banana (6.5 kg) was carried out. The distillate was

extracted with a solvent mixture of pentane and dichloromethane and concentrated. The oil obtained was fractionated by column chromatography on silica gel into 8 fractions (Fr-1–Fr-8). Organoleptic evaluation of each fraction by sniffing is described in Table II. Each fraction was analyzed by GC and GC-MS. The results are summarized in Table III.

Alkenyl Esters. Esters (acetate, butanoate, 3-methylbutanoate, and pentanoate) of hex-4(*Z*)-en-1-ol, hept-4(*Z*)-en-2-ol, oct-4(*Z*)-en-1-ol, oct-5(*Z*)-en-1-ol, and dec-4(*Z*)-en-1-ol were detected in banana volatiles. They are uncommon as components of fruit volatiles and have a double bond in the alcoholic moiety. In general, unsaturated esters in fruit volatiles have a double bond in the acidic moiety such as pear (Shiota, 1990) and pineapple (Umano et al., 1992). Among such esters, hex-3(*Z*)-enyl, hex-2(*E*)-enyl, and prenyl (3-methylbut-2-en-1-yl) esters are available and well-known in the flavor and fragrance industry as those with an unsaturated alcoholic moiety (Burdock, 1992).

These esters except hex-4(*Z*)-en-1-yl, hept-4(*Z*)-en-2-yl, oct-4(*Z*)-en-1-yl, and oct-5(*Z*)-en-1-yl acetates were detected for the first time in the volatiles of banana. Hex-4(*Z*)-en-1-ol and its acetate were reported in the volatiles of banana (Berger et al., 1986) and passionfruit (Murray et al., 1972). Hex-4(*Z*)-en-1-yl butanoate was reported only in passionfruit volatiles (Murray et al., 1972) but was detected also in banana in this study.

Hept-4(*Z*)-en-2-ol was found previously in banana (Tressl et al., 1970), in corn leaves (Buttery and Ling, 1984), in corn silk and tassel (Buttery et al., 1980), and in kernel and husk of corn (Buttery et al., 1978). The acetate was reported only in banana aroma (Tressl et al., 1970). The butanoate, 3-methylbutanoate, and pentanoate were detected in this study. Hept-4(*E*)-en-2-ol, which is a geometric isomer of hept-4(*Z*)-en-2-ol, was reported in pickled tea (Miang) in Thailand (Kawakami et al., 1986). The esters of hept-4(*E*)-en-2-ol have not been detected yet, but hept-4(*E*)-en-2-yl acetate, butanoate, 3-methylbutanoate, and pentanoate were detected in banana volatiles in this study in company with the 4(*Z*)-isomer. Mass spectra of these esters were very similar to those of the 4(*Z*)-isomer.

Oct-4(*Z*)-en-1-ol and its acetate were reported in banana (Berger et al., 1986), but the butanoate and 3-methylbutanoate were also detected in this study. The acetate, butanoate, 3-methylbutanoate, and pentanoate of the 4(*E*)-isomer were also detected as in the case of hept-4-en-2-ol.

Oct-4(*Z*)-en-1-ol was reported in Kogyoku apple volatiles (Yajima et al., 1984), but the esters have not been found

Table IV. Odor and Taste Evaluation of Acetate, Butanoate, 3-Methylbutanoate, and Pentanoate of Alk-4(Z)-enol and Alkan-2-ol^a

compound	taste in water	odor	purity ^b
oct-4(Z)-en-1-yl acetate	green, coarse, ripened banana-like, overripened pear-like, woody, fishy (1.3 ppm) ^c	fruity, estery, pear-like, coarse, diffusible, fishy	95.67
oct-4(Z)-en-1-yl butanoate	banana flesh-like, fruity, sweet, pineapple-like, banana tissue-like, fermented, wine-like (5 ppm) ^c	banana-like, ripe, fruity, smooth, sweet, heavy, whiskey-like, bright	94.34
oct-4(Z)-en-1-yl 3-methylbutanoate	fruity, juicy, succulent, fish oil-like, overripened pear-like, heavy (5 ppm) ^c	fruity, melon-like, banana undertone, green, cool, sweet, bright	94.86
oct-5(Z)-en-1-yl acetate	banana-like, melon-like, estery, juicy, pineapple-like, green, fruit flesh-like (2.5 ppm) ^c	banana tissue-like, melon-like, green, coarse, fruit flesh-like, fermented	98.85
oct-5(Z)-en-1-yl 3-methylbutanoate	melon-like, woody, dry fruit-like, pineapple-like, ripe, fruity, smooth (5 ppm) ^c	ripened banana-like, melon-like, woody, fruit flesh-like, smooth, sour	98.90
dec-4(Z)-en-1-yl acetate	herbaceous, citrus-like, bitter, fatty, pungent (3 ppm) ^c	fruity, juicy, orange-like, marmelo-like, heavy, estery, sweet	89.49
dec-4(Z)-en-1-yl butanoate	citrus-like, estery, fruit flesh-like, smooth, diffusible (5 ppm) ^c	banana-like, apple-like, blood orange-like, vegetable-like, fatty	89.71
dec-4(Z)-en-1-yl 3-methylbutanoate	persimmon-like, pear flesh-like, apple-like, sweet, juicy, smooth (2.5 ppm) ^c	mango-like, apple-like, sweet, fatty, paper-like, burnt, smooth	89.60
pentan-2-yl acetate	sweet, soft, fruity, Japanese pear-like, dry (5 ppm) ^c	banana-like, pear-like, sweet, bright, succulent, fruity, solvent-like	99.52
pentan-2-yl butanoate	banana-like, fruit flesh-like, coarse (5 ppm) ^c	banana-like, pale, sweet, slimy, fruit flesh-like	99.51
pentan-2-yl 3-methylbutanoate	ripe, round, brandy-like, bitter, full-bodied (10 ppm) ^c	fruity, firm, metal-like, cool, brandy-like, apple-like	99.31
pentan-2-yl pentanoate	smooth, warm, pear-like, good taste (10 ppm) ^c	banana-like, cool, sweet, heavy, wine-like, fatty	99.50
hexan-2-yl acetate	sweet-sour, fruity, diffusive, soft, fruit skin-like, apple-like (5 ppm) ^c	sweet-sour, fruity, soft, pear-like, apple-like, pale	99.85
hexan-2-yl butanoate	green, firm, fruit flesh-like, astringent, berry-like (5 ppm) ^c	banana-like, sweet, ripe, pale, fruity, bright, fruit flesh-like	99.16
hexan-2-yl 3-methylbutanoate	soft, mild, smooth, green, brandy-like, berry-like (10 ppm) ^c	brandy-like, sweet, soft, fruity, dry	99.30
hexan-2-yl pentanoate	soft, fruity, pale, astringent, fruit flesh-like, good taste (10 ppm) ^c	green, astringent, pear-like, apple-like, fatty	99.40

^a Evaluated by seven expert panelists. ^b Peak area percentage using GC in the same conditions as the volatile analysis. ^c Suitable concentration for taste evaluation.

in nature. Oct-5(Z)-en-1-yl acetate and 3-methylbutanoate were detected in this study.

Dec-4(Z)-en-1-ol is known as a component in commercial buchu leaf oil (Kaiser et al., 1975), but the esters have not been found. The acetate, butanoate, and 3-methylbutanoate were detected in this study.

Odor and taste evaluation of these new esters was carried out at a suitable concentration in water, except hept-4-en-2-yl esters, which were not so pure. The results are shown in Table IV.

Alkan-2-yl Esters. The esters of pentan-2-ol, hexan-2-ol, and heptan-2-ol also would be important components for banana aroma. They have been found almost only in banana volatiles, although the acetate, butanoate, and hexanoate of heptan-2-ol are known as components in passion fruit volatiles (Murry et al., 1972). Therefore, they might be characteristic components for banana aroma.

Pentan-2-ol was reported in banana (Tressl et al., 1969, 1970), passionfruit (Murry et al., 1972), papaya (Flath et al., 1977), cherimoya (Idstein et al., 1984), starfruit (Frohlich et al., 1989), and other fruit. The acetate, butanoate, 3-methylbutanoate, and pentanoate were detected in banana volatiles (Tressl et al., 1969, 1970). Hexan-2-ol was reported in banana (Tressl et al., 1969, 1970), starfruit (Frohlich et al., 1989), and papaya (Flath et al., 1972). The acetate was found in banana (Tressl et al., 1969, 1970), and the butanoate was only in black tea (Mick and Schreier, 1984). The pentanoate and 3-methylbutanoate have not been found yet in nature. Their odor and taste were also evaluated by expert panelists. The results are summarized also in Table IV. As these esters included an optically active carbon, research on difference of odor and taste between (+)- and (-)-isomers will be necessary in the future. In this study racemates were evaluated. Organoleptic evaluation of these alkenyl and alkan-2-yl esters would be suggestive that they would be important for banana aroma.

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- Registry Numbers Supplied by Author:** Toluene, 108-88-3; ethyl benzene, 100-41-4; *p*-xylene, 106-42-3; styrene, 100-42-5; *o*-xylene, 95-47-6; 1,2,4-trimethylbenzene, 95-63-6; 2-pentylfuran, 3777-69-3; δ -3-carene, 13466-78-9; *p*-cymene, 99-87-6; limonene, 138-86-3; γ -terpinene, 99-85-4; β -caryophyllene, 87-44-5; γ -cadinene, 483-76-1; octane, 111-65-9; dec-1-ene, 872-05-9; undecane, 1120-21-4; dodec-1-ene, 112-41-4; dodecane, 112-40-3; tridecane, 629-50-5; tetradec-1-ene, 1120-36-1; tetradecane, 629-59-4; hexadec-1-ene, 629-73-2; hexadecane, 544-76-3; heptadecane, 629-78-7; octadec-1-ene, 112-88-9; octadecane, 593-45-3; nonadecane, 629-92-5; eicos-1-ene, 3452-07-1; eicosane, 112-95-8; heneicosane, 629-94-7; docos-1-ene, 1599-67-3; docosane, 629-97-0; tricosane, 638-67-5; tetracosane, 646-31-1; pentacosane, 629-99-2; ethyl acetate, 141-78-6; propyl acetate, 109-60-4; 2-methylpropyl acetate, 110-19-0; 3-ace-
toxybutan-2-one, 4906-24-5; butyl acetate, 123-86-4; pentan-2-yl acetate, 626-38-0; 3-methylbutyl acetate, 123-92-2; pentyl acetate, 628-63-7; hexan-2-yl acetate, 5953-49-1; hex-3(*Z*)-en-1-yl acetate, 3681-71-8; hexyl acetate, 142-92-7; hex-4(*Z*)-en-1-yl acetate, 42125-17-7; heptan-2-yl acetate, 5921-82-4; hept-4(*Z*)-en-2-yl acetate, 94088-33-2; oct-4(*Z*)-en-1-yl acetate, 105450-79-1; oct-5(*Z*)-en-1-yl acetate, 71978-00-2; octyl acetate, 112-14-1; 2-methylpropyl butanoate, 539-90-2; butyl butanoate, 109-21-7; 3-[(propylcarbonyl)oxy]butan-2-one, 84642-61-5; pentan-2-yl butanoate, 60415-61-4, 3-methylbutyl butanoate, 106-27-4; hexan-2-yl butanoate, 6963-52-6; hex-3(*Z*)-en-1-yl butanoate, 16491-36-4; hexyl butanoate, 2639-63-6; heptan-2-yl butanoate, 39026-94-3; 3-methylbutyl 2-methylpropanoate, 2050-01-3; 3-methylbutyl 2-methylbutanoate, 27625-35-0; 2-methylpropyl 3-methylbutanoate, 589-59-3; butyl 3-methylbutanoate, 109-19-3; 3-methylbutyl 3-methylbutanoate, 659-70-1; hexyl 3-methylbutanoate, 10032-13-0; octyl 3-methylbutanoate, 7786-58-5; 3-methylbutyl pentanoate, 2050-09-1; hexyl pentanoate, 1117-59-5; hexyl hexanoate, 6378-65-0; 3-methylbutyl hexanoate, 2198-61-0; 2-phenylethyl butanoate, 103-52-6; 3-phenylpropyl butanoate, 7402-29-1; 2-phenylethyl 3-methylbutanoate, 140-26-1; 2-phenylethyl hexanoate, 6290-37-5; dibutyl phthalate, 84-74-2; 3-methylbutanal, 590-86-3; hexanal, 66-25-1; hex-2(*Z*)-enal, 16635-54-4; hex-2(*E*)-enal, 6728-26-3; hexa-2,4-dienal, 142-83-6; phenylacetaldehyde, 122-78-1; octa-2,4-dienal, 5577-44-6; furfural, 98-01-1; 5-methylfurfural, 620-02-0; pentan-2-one, 107-87-9; penta-2,3-dione, 600-14-6; pent-3-en-2-one, 625-33-2; hexan-2-one, 591-78-6; heptan-2-one, 110-43-0; hept-4(*Z*)-en-2-one, 38397-37-4; hept-2(*Z*)-en-4-one, 4643-25-8; hept-3(*E*)-en-2-one, 5609-09-6; oct-1-en-3-one, 4312-99-6; nonan-2-one, 821-55-6; undecan-2-one, 112-12-9; tridecan-2-one, 593-08-8; pentadecan-2-one, 2345-28-0; heptadecan-2-one, 2922-51-2; α -ionone, 127-41-3; acetophenone, 98-86-2; 2-methylpropanol, 78-83-1; butan-1-ol, 71-36-3; 3-methylbutanol, 123-51-3; pentan-2-ol, 6032-29-7; hex-2(*E*)-en-1-ol, 928-95-0; hexan-2-ol, 626-93-7; hex-3(*E*)-en-1-ol, 928-97-2; hex-3(*Z*)-en-1-ol, 928-96-1; hexan-1-ol, 111-27-3; hex-4(*Z*)-en-1-ol, 928-91-6; heptan-2-ol, 543-49-7; hept-4(*E*)-en-2-ol, 58927-81-4; hept-4(*Z*)-en-2-ol, 34146-55-9; oct-4(*Z*)-en-1-ol, 54393-36-1; oct-5(*Z*)-en-1-ol, 64275-73-6; dec-4(*Z*)-en-1-ol, 57074-37-0; 2-methoxy-4-methylphenol, 93-51-6; 2-methoxy-4-(2-ethenyl)phenol, 7786-61-0; 2-methoxy-4-(2-propenyl)phenol, 97-53-0; 1,2-dimethoxy-4-methylbenzene, 494-99-5; 1,2-dimethoxy-4-(2-propenyl)benzene, 93-15-2; 1,2,6-trimethoxy-4-(2-propenyl)benzene, 487-11-6; 2,6-dimethoxy-4-(2-propenyl)phenol, 6627-88-9; palmitic acid, 57-10-3; linoleic acid, 60-33-3; linolenic acid, 463-40-1; oleic acid, 112-80-1.

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