Quantitative Determination of 46 Volatile Constituents in Fresh, Unpasteurized Orange Juices Using Dynamic Headspace Gas Chromatography

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A dynamic headspace gas chromatographic technique was used to quantify 46 volatile components of both hand extracted and mechanically extracted fresh orange juice samples from five different cultivars, Valencia, Pineapple, Hamlin, Pera, and navel, as well as one hybrid, Ambersweet. The effect of higher peel oil levels in the mechanically extracted juices on amounts of specific components was demonstrated. Comparison of reported aroma threshold values for 34 of the components with amounts present in juice showed certain components to be most likely to contribute to fresh orange flavor. This study provides a more extensive database for volatile components in fresh orange juice than previously available.

INTRODUCTION

It has long been believed that the delicate and desirable flavor of fresh-squeezed orange juice is due to a complex mixture of many volatile constituents blended in the proper proportions (Shaw, 1991). To better understand the complex mixture required for fresh orange flavor, there is a need for accurate quantitative information on as many volatile juice constituents as possible. It also would be useful to have these data for juices from a variety of orange cultivars and for juices produced by both hand extraction and mechanical extraction. Such data would help processors and flavorists better understand the quantitative relationships and the range of values for the volatile constituents present in various fresh orange juice samples.

In previously reported studies, from both our laboratory and others, either a relatively small number of constituents was quantified or, when a larger number of constituents was quantified, only one or two juice samples were studied. In an extensive study of a single fresh orange juice sample, Schreier et al. (1977) quantified 39 volatile constituents. Since preliminary solvent extraction and liquid column chromatographic separation were necessary prior to gas chromatographic (GC) analysis, this technique is not easily adaptable to analysis of a larger number of juice samples. In other limited studies using GC analysis, Pino (1982) quantified seven volatile constituents and Rodriguez and Culbertson (1983) quantified eight volatile constituents, each involving a single sample of fresh orange juice.

Recent studies at our laboratory have involved quantitation of up to 24 volatile constituents in several fresh orange juice samples. Moshonas and Shaw (1987) quantified 24 volatile constituents of one sample each of fresh Valencia and Temple orange juices. Nisperos-Carriedo and Shaw (1990) quantified 20 volatile constituents in 15 fresh orange juice samples using static headspace GC to establish a mean and range for each constituent for comparison with similar data from processed juices. With that same technique, Lizotte and Shaw (1992) quantified 22 volatile compounds in fresh Valencia orange juice and tabulated means and ranges for all compounds quantified in several juice samples. Moshonas and Shaw (1992) compared static and dynamic headspace GC for quantification of 16 volatile compounds in four fresh orange juice samples, while Shaw et al. (1993) used static headspace GC to quantify 19 volatile compounds in a different set

of four fresh orange juice samples. The relatively low sensitivity of the static headspace GC method used in these studies limited the number of constituents that could be quantified.

In the current study, dynamic headspace GC was used to quantify 46 volatile juice constituents in 13 fresh orange juice samples. Both hand expressed and mechanically expressed juices were included, and the juices were from six different cultivars, including the Ambersweet hybrid (Moshonas et al., 1991). Sixteen minor components have not yet been identified. This study provides the most extensive database yet determined for volatile compounds present in fresh orange juice.

MATERIALS AND METHODS

Juice Samples. Thirteen freshly squeezed (not pasteurized) orange juice samples from six cultivars were analyzed. Samples were extracted either by (1) standard commercial FMC in-line extraction (mechanically) of large (>20 box) samples of fruit or (2) halving and juicing a 20-fruit sample using a kitchen juicer (hand). In each case, the samples were analyzed immediately after extraction. During the time the first sample was being run, samples for repetitive runs were stored at -18 °C to maintain the fresh juice quality. The juice samples listed at the top of Table 1 were as follows: Valencia sample A from four boxes of fruit extracted mechanically on April 3; Valencia sample B from oranges delivered to a commercial plant and extracted mechanically on April 23; Valencia sample C obtained from a grove in Dundee, FL, and extracted by hand on May 30; Pineapple sample A from oranges delivered to a commercial plant and extracted mechanically on January 25; Pineapple sample B obtained from a local grove and extracted by hand on January 29; Pineapple sample C obtained from a local grove and extracted by hand on February 17; Hamlin sample A obtained from a local grove and extracted by hand on January 13; Hamlin sample B obtained from a local grove and extracted by hand on January 15; navel sample A (Florida) obtained from a local market and extracted by hand on November 30; navel sample B (California) obtained from a local market and extracted by hand on November 30; Pera sample obtained from the Citrus Arboretum, Florida Department of Agriculture and Consumer Services, Winter Haven, FL, and extracted by hand on February 17; Ambersweet sample A from 20 boxes of fruit extracted mechanically on December 2; Ambersweet sample B obtained from a local grove and extracted by hand on December 11.

Headspace GC Analysis of Juice. Juice samples were analyzed with a Hewlett-Packard Model 5890 gas chromatograph equipped with a purge and trap injector (Chrompack, Raritan,

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NJ). A 30 m × 0.53 mm i.d. HP-5 capillary column with 2.65-μm film thickness (Hewlett-Packard, Wilmington, DE) was employed with both the FID detector and injection port at 250 °C. Temperature programming was 40 °C for 6 min, increased at 6 °C/min to a final temperature of 200 °C. Column flow rate was 8 mL/min. Peak areas were used for integration of each component.

To purge the headspace above the juice sample and cryofocus components on the cold capillary trap, a 5-mL juice sample was placed in the sample flask and kept at 40 °C with a water bath as a helium flow purge of 18 mL/min swept the headspace over the sample for 5 min. The flow with the entrained juice volatiles passed a condenser cooled to 0 °C to remove some of the water and continued through a glass tube heated at 120 °C to prevent component condensation. The volatiles were then cryofocused on capillary tubing kept at -130 °C with liquid nitrogen. Once the sample was collected, the cold trap was flash heated to 250 °C to inject the sample onto the gas chromatographic column. These trap and purge sequences were fully automated.

Concentrations for each of the 46 compounds were calculated with regression equations, determined using standard solutions prepared by injecting four different concentrations of each compound added to a juice base to obtain a peak area calibration curve. The juice base was prepared by reconstitution to 11.8° Brix of concentrated orange juice (pumpout) from an evaporator that contained no added flavor fractions. Each standard solution was kept for 3 h at room temperature and then overnight at 5 °C to permit equilibration of the hydrocarbon standards between pulp and juice (Shaw et al., 1994).

Identification of Volatile Components. Volatile orange juice components were separated for identification by GC-MS. Fifty milliliters of aqueous distillate from freshly squeezed juice was extracted three times with 25-mL portions of methylene chloride (Burdick and Jackson, capillary GC-MS grade solvent), and the combined extracts were dried over sodium sulfate and concentrated to small volume (<0.5 mL) under reduced pressure on a rotary evaporator. Samples (2 μL) of the concentrated extract were used for GC-MS analyses. A Hewlett-Packard Model 5970B, MSD, GC-MS was used with a 0.32 mm × 50 m fused silica column of crossed-linked 5% phenylmethyl silicone. Column oven temperature programming was 55 °C for 9 min, raised at 7.5 °C/min to 220 °C and held there for 30 min. Injection port and ionizing source were kept at 275 °C, and the transfer line was kept at 280 °C. Mass spectral matches were made by comparison of mass spectra and retention times with those of authentic compounds. Retention times of components were also compared with those of standards prepared above and by enrichment of juice with authentic samples followed by analysis using the headspace GC system described above.

RESULTS AND DISCUSSION

Representative samples of 13 fresh-squeezed unpasteurized juices from five major orange cultivars and one orange hybrid processed in the United States and Brazil were used in this study. A dynamic headspace GC sampler was used to quantify 46 volatile constituents in each juice. The quantitative values are listed in Table 1 for each component in the 13 fresh orange juice samples. The samples include juices from six different cultivars as well as both hand expressed and mechanically expressed juices. A representative chromatogram for one of the juice samples is shown in Figure 1.

The volatile constituents present in fresh orange juice originate from three sources (Nagy and Shaw, 1990). The juice contained in the juice sacs which is liberated during extraction is the source for the volatile water-soluble compounds. Two types of oil, juice oil and peel oil, contribute the oil-soluble compounds to the flavor of fresh orange juice. Juice oil is present in globular bodies within the juice sacs (Davis, 1932), and it becomes dispersed in the juice during extraction. Rice et al. (1952) demonstrated the presence of about 0.005% juice oil in juice extracted from fruit that had been carefully hand-peeled before
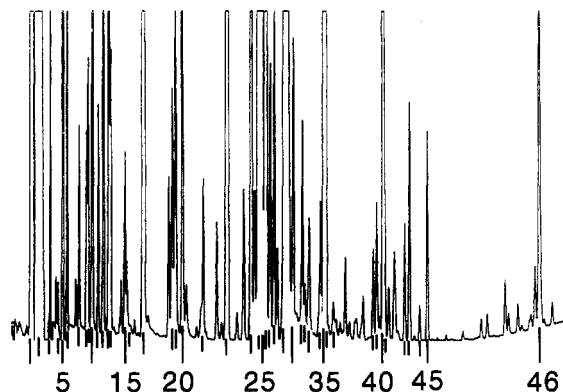
Figure 1. Chromatogram of Valencia orange juice volatiles. Vertical lines below the baseline mark each of the 46 compounds identified in Table 1.

extraction to exclude the presence of peel oil. Hand extraction of unpeeled oranges introduced peel oil into the juice, in addition to juice oil, and doubled the amount of total oil present. Since commercial orange juice typically contains about 0.015-0.025% total oil (Kimball, 1991), the juice oil contributes only one-third to one-fifth of the oil-soluble components, thus making peel oil the major source for most of these components in mechanically extracted juices.

A comparison of the hand expressed and mechanically expressed juices for Valencia and Pineapple cultivars and Ambersweet hybrid in Table 1 shows the contribution of peel oil to the levels of several juice constituents. The 17 oil-soluble constituents present at higher levels in the mechanically expressed juices included α-pinene, myrcene, limonene, octanal, nonanal, decanal, neral, geranial, and linalool. These nine constituents are generally considered to make important contributions to orange juice flavor (Shaw, 1991). Except for limonene, the levels in juice believed to be optimum for these compounds have not been established. Thus, higher levels of these substances in juice are not necessarily better than lower levels for good fresh orange flavor. For limonene, the optimum level in processed orange juice can be estimated from the optimum total oil level recommended, which is 150-220 ppm (Carter, 1990). Since peel oil contains more than 90% limonene, the optimum range for limonene in processed juice is about 135-180 ppm. In fresh unprocessed juice, the optimum level may be different, however.

The remaining eight constituents found at higher levels in mechanically expressed juices were the monoterpene hydrocarbons sabinene, α-phellandrene, δ-3-carene, β-ocimene, and γ-terpinene and the oxygenated compounds octanol, (E)-linalool oxide, and carvone. Although these trace constituents in orange juice have not been directly associated with orange juice flavor, some have been used in synthetic citrus flavors, including α-phellandrene, δ-3-carene, and β-ocimene (Arctander, 1969). γ-Terpinene in dilute solutions has a pleasant, citrus-like taste and is known to be important in mandarin oil aroma (Wilson and Shaw, 1981).

Most of the compounds not discussed above that were quantified in this study are water-soluble constituents present prior to juice extraction in the aqueous portion of the juice sacs. Comparison of quantities present in mechanically expressed vs hand expressed Valencia and Pineapple juices in Table 2 shows no appreciable difference between the amounts present.

The seven water-soluble constituents quantified that are considered important to orange juice flavor include ethyl acetate, ethyl propionate, methyl butanoate, ethyl

Volatile Constituents in Orange Juice

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Table 1. Quantities (Parts per Million) of Volatile Components in Fresh-Squeezed Unpasteurized Orange Juice

component[a]	Valencia mech[b] A	B	hand[c] C	Pineapple mech A	hand B	C	Hamlin, hand A	B	navel, hand[d] A	B	Pera, hand	Ambersweet mech A	hand B
methanol[e]	60	126	37	56	85	51	36	78	3	31	7	7	76
ethanol	660	1230	1150	460	730	580	240	330	22	890	760	22	415
1-propanol	0.21	0.44	0.31	0.20	0.38	0.25	0.16	0.19	0.050	1.14	0.31	0.077	0.16
ethyl acetate	0.15	0.25	0.28	0.15	0.23	0.17	0.12	0.13	0.077	0.17	0.28	0.082	0.12
2-methyl-3-buten-2-ol	0.18	0.34	0.40	0.18	0.32	0.22	0.11	0.13	0.047	0.21	0.41	0.057	0.11
2-methylpropanol	0.010	0.087	0.048	0.026	0.11	0.024	0.015	0.069	tr[f]	0.10	0.008	0.0010	0.046
butanol	0.0064	0.069	0.019	0.026	0.027	0.026	0.027	0.024	0.006	0.22	0.015	0.012	0.031
1-penten-3-ol	0.12	0.037	0.037	0.034	0.15	0.12	0.064	0.066	0.021	0.004	0.11	0.064	0.081
1-penten-3-one	0.054	0.024	0.013	0.029	0.047	0.018	0.015	0.023	tr	0.008	0.016	0.11	0.092
2-pentanol	0.46	0.30	0.037	0.11	0.13	0.11	0.14	0.14	0.02	0.02	0.066	0.21	0.24
ethyl propionate	0.0038	0.012	0.008	0.028	0.016	0.010	0.003	0.0036	0.023	0.028	0.017	0.008	0.021
methyl butanoate	0.006	0.033	0.016	0.019	0.027	0.011	0.018	0.033	0.0001	0.008	0.003	0.006	0.028
3-methylbutanol	0.014	0.39	0.17	0.12	0.39	0.10	0.074	0.22	0.021	0.15	0.0004	0.007	0.33
2-methylbutanol	0.001	0.053	0.025	0.018	0.067	0.012	0.009	0.029	tr	0.025	0.0027	0.002	0.037
1-pentanol	0.093	0.055	0.013	0.019	0.031	0.026	0.021	0.021	tr	tr	0.023	0.031	0.045
3-methyl-2-buten-1-ol	tr	0.033	0.0074	0.034	0.052	0.018	0.071	0.048	0.020	tr	0.11	tr	0.096
ethyl butanoate[g]	0.83	1.53	0.84	0.70	0.89	0.82	0.70	0.95	tr	0.43	1.03	0.85	0.81
(E)-2-hexenal	0.010	0.037	0.041	0.037	0.058	0.032	0.011	0.018	0.015	0.010	0.018	0.005	0.012
(Z)-3-hexen-1-ol[h]	0.31	0.27	0.17	0.15	0.66	0.48	0.26	0.30	0.16	0.54	0.47	0.84	0.34
hexanol	0.20	0.16	0.12	0.048	0.29	0.17	0.067	0.096	0.005	tr	0.26	0.039	0.066
heptanal	0.0038	0.0029	tr	0.0006	tr	tr	tr	tr	tr	tr	tr	0.002	tr
α-pinene	0.57	0.94	0.10	1.09	0.16	0.13	0.13	0.13	0.25	0.43	0.29	1.03	0.39
sabinene	0.15	0.039	0.023	0.051	0.017	0.018	0.015	0.015	0.16	0.26	0.079	0.19	0.023
myrcene	0.76	3.3	0.34	4.1	0.58	0.44	0.48	0.49	1.06	1.90	1.09	4.0	1.5
ethyl hexanoate	0.047	0.13	0.16	0.076	0.076	0.070	0.086	0.13	0.0087	0.24	0.14	0.040	0.049
octanal	0.18	0.59	0.004	0.65	0.005	0.006	0.008	0.008	0.056	0.086	0.004	0.041	0.012
α-phellandrene	0.021	0.024	0.009	0.029	0.009	0.008	0.008	0.008	0.011	0.018	0.014	0.022	0.012
δ-3-carene	0.016	0.043	0.009	0.097	0.009	0.009	ND[i]	ND	0.025	0.064	0.014	0.009	ND
limonene	76	134	18	191	29	24	24	24	43	62	53	167	65
β-ocimene	0.025	0.072	0.018	0.28	0.023	0.027	0.026	0.030	0.020	0.040	0.017	0.058	0.028
γ-terpinene	0.010	0.010	0.002	0.013	0.003	0.003	0.004	0.030	0.003	0.002	0.004	0.004	0.004
octanol	0.13	0.37	0.089	0.46	0.080	0.086	0.078	0.073	0.166	0.23	0.080	0.083	0.075
(Z)-linalool oxide	0.048	0.11	ND	0.14	ND	ND	ND	ND	0.012	0.014	0.0078	0.011	0.017
(E)-linalool oxide	0.081	0.14	0.022	0.30	0.038	0.033	0.031	0.036	0.054	0.15	0.047	0.078	0.033
linalool	0.75	0.92	0.13	3.7	0.033	0.053	0.016	0.013	0.17	0.47	0.21	1.02	0.39
nonanal	0.022	0.082	0.001	0.087	0.003	0.003	0.004	0.003	0.007	0.025	tr	0.025	0.007
ethyl 3-hydroxyhexanoate	0.27	0.49	0.28	0.39	0.35	0.32	0.32	0.34	tr	tr	0.33	0.34	0.38
ethyl octanoate	0.035	0.023	0.031	0.20	0.009	0.008	0.010	0.008	0.006	0.010	0.063	0.010	0.015
terpinen-4-ol	0.11	0.20	ND	ND	0.18	0.17	0.17	0.15	0.071	0.14	ND	0.10	0.085
decanal	0.16	0.45	0.016	0.50	0.022	0.022	0.021	0.019	0.057	0.29	0.057	0.18	0.043
α-terpineol	0.19	0.91	tr	tr	tr	tr	tr	tr	tr	0.18	tr	3.7	0.13
neral	0.0005	0.028	tr	0.0014	0.0004	0.001	tr	0.0004	0.023	0.023	0.007	0.020	tr
carvone	0.058	0.11	0.004	0.067	0.012	0.016	0.018	0.020	tr	tr	0.013	0.10	0.10
geranial	0.0005	0.035	tr	0.0019	0.0003	0.0013	0.012	0.0004	0.033	0.032	0.004	0.022	tr
perillaldehyde	0.034	0.097	0.012	0.060	0.003	0.004	0.004	0.003	0.034	0.016	0.069	0.010	0.022
valencene	2.1	4.4	3.30	5.1	3.6	2.4	5.1	1.6	1.2	3.7	12.1	6.5	0.83

[a] Listed in increasing retention order on a nonpolar capillary GC column. [b] Mech, mechanically expressed with an FMC in-line extractor. [c] Hand, hand expressed with an electric kitchen juicer. [d] Sample A, Floridal navel; sample B, California navel. [e] A minor amount of acetaldehyde coeluted with this component. [f] tr, trace, detected but too small to quantify. [g] A minor amount of hexanal coeluted with this component. [h] A minor amount of (E)-2-hexenol coeluted with this component. [i] ND, not detected.

butanoate, ethyl 3-hydroxyhexanoate, ethanol, and (Z)-3-hexen-1-ol (Shaw, 1991). Four of these, ethyl propionate, ethyl butanoate, ethanol, and (Z)-3-hexen-1-ol, are present in most juices in amounts above their previously determined flavor threshold values in water (Shaw, 1991). A corresponding value for ethyl 3-hydroxyhexanoate has not been reported. Two additional components considered important in orange flavor that were identified, but not quantified, in this study were acetaldehyde and (E)-2-hexenol. Each of these constituents eluted from the GC column with a more prevalent component (see footnotes to Table 1).

All compounds identified in this study had been identified earlier as constituents of orange juice, peel oil, or other natural orange flavor fractions (Maarse and Visscher, 1989). For many of these compounds, the contribution to orange juice flavor has not been determined. Some indication of the importance of individual components to flavor can be seen by comparing aroma threshold in air or water to amount present in orange juice.

Table 2 lists reported aroma threshold values for 34 of the 46 quantified components. For some compounds in Table 2, additional threshold values have been reported (Fazzalari, 1978), but the values listed are representative aroma threshold values reported in water. In 24 of the 34 components, the amount present was above the threshold level in some or all of the juice samples. The components generally present at highest relative levels (10-fold or higher above their aroma thresholds) were among those most often shown to be important in orange flavor. They were limonene, myrcene, α-pinene, decanal, octanal, ethyl butanoate, and linalool (Shaw, 1991; Ahmed et al., 1978).

The results reported in this study provide the most complete database yet determined for quantities of volatile flavor compounds present in fresh orange juice. The wide range of values present in fresh orange juices for most components, and the relative contribution of components from peel oil, which is introduced during juice extraction, were demonstrated also.

Table 2. Comparison of Aroma Thresholds in Water with Amounts of Volatile Components Present in Orange Juice

component	aroma threshold[a] (ppb)	range of values found[b] (ppb)
hydrocarbons		
δ-3-carene		<2–97
limonene	60[c]; 229[d]	24 000–191 000
myrcene	36[d]; 46[d]	440–4100
β-ocimene		17–280
α-phellandrene		8–29
α-pinene	9.5[c]; 62[d]	100–1090
sabinene	37[d]	15–260
γ-terpinene		2–30
valencene		830–12 100
aldehydes		
citral	27.6[d]; 85[c]	<1–63
decanal	2[c]; 4.9[d]	19–500
heptanal	2	<0.6–3.8
(E)-2-hexenal	17; 24[c]	5–58
nonanal	2.5[c]; 4.4[d]	<1–87
octanal	1.4[c]; 6.4[d]	4–890
perillaldehyde	30[c]	3–97
esters		
ethyl acetate	6; 8.5[d]; 686	77–280
ethyl butanoate	0.13[c]; 1.1[d]	<430–1530
ethyl hexanoate	76[e]	8.7–240
ethyl 3-hydroxyhexanoate		<270–490
ethyl octanoate	240[e]	6–63
ethyl propionate	9.9[c]	3–28
methyl butanoate	15[d]; 43[c]	0.1–33
alcohols		
1-butanol	300	6–69
ethanol	100 000; 1 150 000[d]	22 000–1 230 000
hexanol	10	<5–290
(Z)-3-hexen-1-ol	70	150–840
linalool	4.7[d]; 5.3[c]	13–3700
methanol	53 000; 200 000[d]	3000–126 000
2-methylbutanol	1005[d]	<1–67
3-methylbutanol	170	0.4–390
2-methyl-3-buten-2-ol		47–400
3-methyl-2-buten-1-ol		<7.4–110
2-methylpropanol	1800	<1–110
octanol	190	73–460
1-pentanol	120; 225	<13–93
2-pentanol		20–460
1-penten-3-ol	400	4–150
1-propanol	9000; 81 000[d]	50–1140
terpinen-4-ol	340	<71–200
α-terpineol	46[d]; 280[c]	<130–3700
others		
carvone	2.7[c]; 6.7[d]	<4–110
(Z)-linalool oxide		<11–140
(E)-linalool oxide		22–300
1-penten-3-one	0.9[c]; 1.3	<8–110

[a] Value tabulated by Fazzalari (1978) in water unless noted otherwise. [b] Data from Table 1. When "<" precedes lowest reported value, it indicates at least one sample contains a value too small to quantify. [c] Ahmed et al. (1978). [d] Pino et al. (1986). [e] Salo et al. (1972) in artificial beverage imitating whiskey.

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