

Quantitation of Volatile Constituents in Mandarin Juices and Its Use for Comparison with Orange Juices by Multivariate Analysis

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Sixteen unpasteurized juices and one pasteurized juice from mandarin and mandarin hybrid fruit were analyzed by headspace gas chromatography (HSGC), and 42 volatile constituents were quantified in each sample. Fifteen of the mandarin juice samples had relatively low levels of volatile constituents believed important to citrus flavor when compared to comparable values in orange. The quantities were used for comparison with unpasteurized orange juices using data similarly obtained in an earlier study by HSGC. Multivariate analysis separated the mandarin and orange samples, when the first three principal components were displayed graphically.

Keywords: *Headspace gas chromatography; principal component analysis; fruit juice composition; Citrus reticulata*

INTRODUCTION

Earlier studies from this laboratory in which volatile constituents of juices from mandarin and mandarin hybrid fruit were quantified showed relatively low levels of most of the volatile constituents when compared to similar studies in orange juice (Moshonas and Shaw, 1987; Shaw and Moshonas, 1993). The small number of cultivars studied made it difficult to conclude whether the complex profile of volatile constituents in mandarin is generally weaker than that in orange. Thus, information was needed on juices from a much broader selection of mandarin and mandarin hybrid cultivars. This information would expand our knowledge of mandarin flavor and could help citrus processors in efforts to produce more stable processed mandarin juice products. Such products have been difficult to market because of certain off-flavors as well as changes in flavors that occur during storage (Shaw, 1996b).

In earlier studies, we applied multivariate analysis techniques to quantitative data obtained for volatile constituents in orange juices and juice drinks. Juice products were separated on the basis of the degree of processing (Shaw *et al.*, 1993, 1994; Moshonas and Shaw, 1997) and, in the case of juice drinks, the percentage of orange juice present (Shaw and Moshonas, 1997). These same classification techniques are applicable to quantitative data obtained by similar means from mandarin juice samples.

The current study provides basic information on amounts of volatile constituents in fresh-squeezed juice from 15 mandarin cultivars and 1 commercially processed juice and compares these values to those previously found in orange juices using computer multivariate analysis techniques.

MATERIALS AND METHODS

Fruit and Juice Samples. All fruit were harvested from trees at the Citrus Arboretum, Florida Department of Agriculture and Consumer Services, Winter Haven, FL, and were extracted by hand with a Waring commercial bar juicer unless otherwise noted (8–16 fruit per sample). The Temple orange

sample was obtained on December 21, 1994, from a dooryard tree in Winter Haven, FL. The Dancy fruit juiced on January 11, 1995, and the Murcott (Honey tangor) juiced on January 12, 1995, were purchased at a local market. The mechanically extracted sample of Robinson mandarin juice was prepared from 60 fruit in a state test house extractor (FMC Model 091B) at the Citrus Research and Education Center, Lake Alfred, FL (Florida Department of Citrus, 1996). The commercial sample of pasteurized Italian mandarin juice, not from concentrate, was purchased in Parma, Italy, on September 15, 1994, and kept frozen until analyzed on October 9, 1994.

Dynamic 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 Model PTI, Raritan, NJ). A 0.53 mm i.d. × 30 m nonpolar HP-5 capillary column with 2.65- μ m film thickness (Hewlett-Packard, Wilmington, DE) was employed, with both the FID detector and injection port at 250 °C. The temperature program was 40 °C for 6 min, increased at 6 °C/min to a final temperature of 200 °C, and held at that temperature to the end of the run (30-min total). Column flow rate was 8 mL/min. Peak areas were used for quantification 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 through 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 purge and trap sequences were fully automated.

Concentrations for each of the 42 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 peak area calibration curves (Moshonas and Shaw, 1994). The juice base was prepared by reconstitution to 11.8 °Brix of concentrated orange juice that contained no other added volatile constituents. 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 components in mandarin juice were separated for identification

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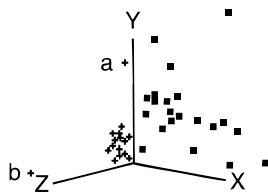


Figure 1. Principal component (PC) analysis (PC1–3) involving quantities of 42 volatile constituents in samples of 17 mandarin and mandarin hybrid juices (+) and 22 orange juices (■): point a = Murcott and point b = commercial mandarin juice samples.

by GC/MS analysis. Fifty milliliters of aqueous distillate from freshly squeezed Robinson mandarin 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 (Moshonas and Shaw, 1994). Samples (2 μ L) of the concentrated extract were used for GC/MS analysis. A Hewlett-Packard Model 5970B, MSD, GC/MS was used with a 0.32 mm \times 50 m fused silica column of cross-linked 5% phenylmethyl silicone. Column oven temperature programming was 55 $^{\circ}$ C for 9 min, raised at 7.5 $^{\circ}$ C/min to 220 $^{\circ}$ C and held there for 30 min. Injection port and ionizing source were kept at 275 $^{\circ}$ C, and the transfer line was kept at 280 $^{\circ}$ 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 the standards obtained during the quantitative determinations described above.

Multivariate Analysis. Quantitative data from headspace analyses of all juice samples were entered into a QuattroPro spreadsheet. The multivariate analysis program EinSight (Infometrix Inc., Seattle, WA) was used to calculate, tabulate, and plot results from principal component analysis (Figure 1) as described earlier (Shaw *et al.*, 1994). For these calculations, a value of 10^{-4} ppm was used for each trace component (lowest detectable level), and for components not detected, a value of 10^{-5} ppm was used (Infometrix, 1991). Eigenvalues used for principal component analysis were determined by autoscaling the data and using a correlation matrix (S. Ramos, Infometrix, Inc., Woodinville, WA, personal communication, 1997; Infometrix, 1991).

RESULTS AND DISCUSSION

Fruits from 15 different mandarin and mandarin hybrid cultivars were juiced, and the freshly expressed juices were quantitatively analyzed by dynamic HSGC along with 1 commercial pasteurized juice to compare the levels of 42 volatile constituents in the various samples studied. Table 1 summarizes the quantitative results obtained. For one cultivar, Robinson, both hand extracted and mechanically extracted juices were analyzed. The mechanically extracted sample contained higher levels of many oil-soluble constituents, including limonene, myrcene, linalool, octanal, nonanal, and decanal than did the hand extracted sample. Similar differences were reported earlier in comparing hand extracted and mechanically extracted fresh orange juices (Moshonas and Shaw, 1994). One additional mandarin sample included in this study was a commercial pasteurized Italian mandarin juice not from concentrate. This sample also contained high levels of oil-soluble constituents.

The two hand extracted juices that contained relatively high levels of volatile constituents were the Murcott (Honey tangerine) and Dancy mandarin samples

in Table 1. The Murcott is a tanger of unknown origin. The Dancy mandarin sample analyzed in this study contained higher levels of volatile constituents than reported in an earlier study from this laboratory involving fresh Dancy mandarin juice (Shaw and Moshonas, 1993). However, 13 of the 15 hand extracted juices showed relatively low levels of volatile constituents, when compared to orange juice, as shown in the last column of Table 1, which lists the mean values from 22 pasteurized orange juice samples reported earlier (Moshonas and Shaw, 1997). This column of data contains only the orange juice constituents that were also quantified in mandarin juice.

One reason for carrying out this more extensive study of volatile constituents in juices from mandarin and mandarin hybrid cultivars was to compare the quantitative and qualitative profiles of volatile constituents in mandarin and orange juices. Quantitative values from a previous study of 46 volatile constituents in fresh-squeezed orange juice samples (Moshonas and Shaw, 1994) were used as a data base for comparison with these mandarin juice samples by multivariate analysis techniques. The data for the 42 constituents common to both types of juices were tabulated in a QuattroPro spreadsheet, and then the principal components (PC) were calculated using EinSight multivariate analysis software. PC analysis involves calculation of the total variance contained in all constituents measured in all mandarin and orange juice samples. A portion of this variance can be reduced to two or three dimensions and thus plotted in conventional two- and three-dimensional graphics involving X, Y, and Z axes in space (Massart *et al.*, 1988).

Figure 1 is a plot of the 17 mandarin samples from Table 1 and 22 fresh orange juice samples from an earlier study (Moshonas and Shaw, 1997), using the first three principal components calculated from quantitative data for 42 volatile constituents common to both mandarin and orange juice samples. This three-dimensional plot of principal components 1–3 represents 65% of the total variance provided by the 42 constituents quantified (Shaw *et al.*, 1994). The plot shown in this figure was obtained by rotation of the axes on the computer screen until maximum separation of orange and mandarin samples was achieved, which afforded separation of the mandarin and orange juice samples into two groups. In a two-dimensional plot of principal components 1 and 2 (figure not shown), the mandarin juice samples a and b in Figure 1 were not separated from the orange juice samples. Principal components 1 and 2 represented 51% of the total variance. Nikdel and Fishback (1989) had used the Einsight program to separate orange juices by country of origin on the basis of mineral content and found similar results (51% of total variance for PC1 and PC2 and 65% for PC1–3).

Most of the mandarin and mandarin hybrid samples were clustered in a small area of Figure 1, while the orange juice samples were more widely dispersed but separated from mandarin samples. The one commercial mandarin juice analyzed (sample b in Figure 1) was well-separated from the fresh mandarin juices, perhaps because of its higher level of peel oil and thus of major peel oil components, including α -pinene, myrcene, limonene, and (*E*)-linalool oxide (Table 1).

Loading values obtained from the EinSight program (Table 2) help explain the separations illustrated in Figure 1. Loading values indicate the relative extent to which each constituent contributes to the variance

Table 1. Quantities (Milligrams per Liter) of Volatile Components in Mandarin and Mandarin Hybrid Juices

component ^a	Robinson ^{fg}												orange ^h					
	Comm ^b	Clem ^c	Dancy ^c	Eller ^c	Fair ^c	Fall ^d	Frem ^c	Iyo ^e	Mon ^c	Mur ^e	Ortan ^e	Page ^f		hand	mech	Sats ^c	Sunb ^f	Temple ^e
methanol ^j	125	1.4	75	3.3	4.1	4.2	1.2	1.1	2.6	124	4.6	0.56	2.2	11.1	2.7	1.9	2.7	50
ethanol	562	tr ^j	520	tr	tr	tr	tr	tr	tr	2128	tr	tr	tr	tr	tr	tr	tr	576
1-propanol	0.27	nd ^k	0.14	tr	tr	tr	tr	tr	tr	0.65	tr	nd	tr	tr	tr	tr	tr	0.30
ethyl acetate	1.2	0.062	0.14	0.064	0.075	0.071	nd	0.065	0.063	0.74	0.13	0.064	0.068	0.063	0.065	0.066	0.067	0.17
2-methylpropanol	0.12	nd	0.035	nd	nd	0.017	nd	0.004	nd	0.34	nd	nd	nd	0.0005	nd	tr	nd	0.04
butanol	0.018	nd	0.004	nd	nd	tr	nd	nd	nd	0.053	nd	nd	tr	0.0005	tr	tr	0.0005	0.04
1-penten-3-ol	0.009	tr	tr	0.004	0.013	nd	0.0006	tr	0.0049	0.009	0.004	0.010	0.021	0.017	0.0069	nd	0.039	0.07
1-penten-3-one	0.010	nd	0.003	0.010	tr	0.0003	nd	nd	tr	0.0014	0.010	0.002	0.0067	0.0008	tr	0.0022	tr	0.035
2-pentanol	0.12	0.007	0.014	0.024	0.024	0.0079	0.0088	0.007	0.026	0.049	0.024	0.028	0.11	0.0075	0.020	0.0079	0.053	0.15
ethyl propionate	0.023	nd	0.011	nd	tr	tr	nd	nd	nd	0.028	nd	nd	tr	nd	nd	nd	nd	0.014
methyl butanoate	0.0002	nd	nd	tr	nd	nd	nd	nd	nd	nd	tr	nd	nd	nd	nd	nd	0.0005	0.016
3-methylbutanol	0.22	nd	0.054	nd	nd	nd	nd	nd	nd	0.97	nd	nd	nd	nd	nd	tr	nd	0.15
2-methylbutanol	0.048	nd	0.004	nd	tr	tr	nd	nd	nd	0.12	nd	nd	tr	nd	tr	nd	nd	0.022
1-pentanol	0.019	nd	0.0007	tr	tr	tr	nd	nd	nd	0.0006	tr	tr	tr	tr	tr	tr	0.0025	0.029
3-methyl-2-buten-1-ol	0.13	0.018	0.13	nd	nd	0.034	nd	0.0078	0.019	0.062	0.011	nd	nd	0.030	0.021	nd	nd	0.038
hexanal	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
(E)-2-hexenal	0.0032	nd	tr	tr	0.0097	0.0061	nd	tr	tr	0.020	nd	tr	tr	tr	tr	tr	tr	0.023
(Z)-3-hexen-1-ol	0.041	0.016	nd	0.013	nd	nd	nd	0.015	0.015	0.079	0.041	0.012	0.030	0.015	0.051	0.040	0.34	0.38
hexanol	0.067	tr	tr	tr	tr	tr	tr	tr	tr	0.026	tr	tr	tr	tr	tr	tr	tr	0.12
heptanal	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	0.0033	tr	tr	tr	tr	0.0007
α -pinene	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.43
sabinene	0.015	0.30	0.075	0.063	0.035	0.023	0.021	0.032	0.10	0.028	0.099	0.039	0.044	0.11	0.031	0.058	0.17	0.08
myrcene ^l	4.2	1.1	0.43	1.6	0.63	0.45	0.31	0.49	0.22	0.49	1.9	0.49	0.66	1.4	0.90	0.52	0.31	1.54
ethyl hexanoate	2.2	nd	nd	nd	nd	nd	nd	nd	0.26	nd	nd	nd	nd	nd	nd	nd	nd	0.10
octanal	0.088	0.0057	0.050	0.0052	0.0038	0.0016	0.0023	0.0037	0.0045	0.026	0.0037	0.0075	0.012	0.14	0.0063	0.0032	0.0049	0.13
α -phellandrene	0.058	0.010	0.016	0.016	0.0085	0.0075	0.0068	0.018	0.0091	0.0077	0.016	0.0080	0.0082	0.12	0.010	0.008	0.007	0.015
δ -3-carene	0.0087	0.016	nd	0.093	nd	0.0089	nd	0.0084	0.014	nd	nd	0.0086	0.011	nd	nd	nd	nd	0.023
limonene	226	44	94	81	34	23	20	110	44	28	104	28	38	73	40	29	19	70
γ -terpinene	10.8	nd	0.60	0.013	0.0027	nd	0.0033	0.71	nd	0.077	0.013	0.0029	0.0037	0.0023	0.20	0.003	0.003	0.007
octanol	nd	0.10	nd	nd	nd	nd	nd	nd	0.13	nd	nd	nd	0.087	0.13	nd	nd	nd	0.15
(E)-linalool oxide	10.3	0.049	0.67	0.28	0.015	0.012	0.014	nd	0.056	0.019	0.044	0.017	0.017	nd	0.012	0.015	0.016	0.08
linalool	1.5	0.23	1.5	0.11	0.058	0.32	0.013	2.2	0.16	0.20	0.12	0.14	0.43	1.0	0.14	0.16	0.12	0.61
nonanal	0.024	0.007	0.039	0.008	0.009	0.009	0.0020	nd	0.008	0.12	0.004	0.0067	nd	0.015	0.021	0.005	0.0058	0.02
ethyl 3-hydroxyhexanoate	2.4	nd	0.23	0.28	nd	nd	nd	0.23	nd	nd	nd	nd	nd	0.29	0.37	0.24	nd	0.29
terpinen-4-ol	0.22	0.29	0.084	nd	nd	nd	nd	nd	0.18	nd	0.063	nd	0.10	0.080	0.10	nd	0.063	0.11
decanal	0.17	nd	0.28	0.044	0.034	0.019	0.016	0.029	nd	0.051	0.056	0.054	0.038	0.24	0.081	0.027	0.030	0.14
α -terpineol	tr	nd	0.48	nd	nd	nd	nd	tr	tr	nd	nd	nd	tr	1.04	tr	nd	nd	0.4
neral	0.0085	nd	0.0032	0.006	nd	nd	nd	0.0068	nd	0.0006	nd	nd	0.0017	0.005	nd	nd	nd	0.008
carvone	0.04	tr	tr	0.017	nd	nd	tr	tr	tr	tr	nd	tr	0.023	0.020	nd	tr	0.011	0.04
geraniol	tr	nd	0.0024	0.006	nd	nd	nd	0.0098	nd	0.0057	nd	nd	0.0007	0.003	tr	tr	0.01	
perillaldehyde	0.20	0.016	0.070	0.012	0.0036	nd	0.0015	0.090	0.017	0.0014	0.008	0.0029	0.015	0.040	0.029	0.0052	0.0011	0.03
valencene	0.73	nd	0.48	nd	0.46	1.8	0.51	0.44	nd	0.61	0.69	0.67	1.0	0.51	0.96	0.99	0.64	4.0

^a Listed in increasing retention order on a nonpolar capillary GC column. ^b Pasteurized juice not from concentrate purchased in Italy. ^c Mandarins: Clem, Clementine; Ellen, Ellendale; Fair, Fairchild; Frem, Fremont; Mon, Monreal Clementine; Sats, Satsuma (Saunt, 1990). ^d Fall, Fallglo, a mandarin \times tangelo hybrid (Hearn, 1987). ^e Orange-mandarin hybrid: Mur, Murcott, or honey tangerine; Ortan, Ortanique (Hodgson, 1967; Saunt, 1990). ^f A tangelo \times mandarin hybrid: Sunb, Sunburst (Hodgson, 1967). ^g Hand, hand extracted; mech, mechanically extracted juice. ^h Data are from 22 unpasteurized orange juice samples reported earlier (Moshonas and Shaw, 1997). ⁱ A minor amount of acetaldehyde coeluted with this component. ^j tr, trace, detected but too small to quantify. ^k nd, not detected. ^l A trace of β -pinene was present in the myrcene peak.

Table 2. Relative Contributions of 42 Constituents to Variance in Principal Components 1–3 Based on Loading Values

constituent	packing order in ^a		
	PC1 ^b (34%)	PC2 ^c (17%)	PC3 ^d (14%)
α-pinene	1	29	14
myrcene	2	17	27
limonene	3	13	22
decanal	4	14	38
neral	5	18	31
carvone	6	39	33
nonanal	7	11	41
octanal	8	10	37
α-phellandrene	9	27	6
geranial	10	21	24
hexanal	11	34	8
octanol	12	42	15
perillaldehyde	13	28	7
linalool	14	22	30
valencene	15	41	13
δ-carene	16	19	29
heptanal	17	26	32
methyl butanoate	18	25	12
sabinene	19	8	26
methanol	20	4	25
1-penten-3-one	21	40	17
ethyl propionate	22	31	28
ethanol	23	6	39
(E)-2-hexenal	24	16	18
1-pentanol	25	7	20
(Z)-3-hexenol	26	32	11
2-pentanol	27	12	21
3-methyl-2-butenol	28	33	9
hexanol	29	9	19
propanol	30	5	40
ethyl 3-hydroxyhexanoate	31	30	5
α-terpineol	32	24	23
butanol	33	15	34
ethyl acetate	34	23	4
2-methylbutanol	35	2	35
2-methylpropanol	36	1	36
3-methylbutanol	37	3	42
ethyl hexanoate	38	35	3
(E)-linalool oxide	39	36	2
γ-terpinene	40	38	1
1-penten-3-ol	41	20	16
4-terpineol	42	37	10

^a Listed in decreasing order of their contribution to the variance of that PC based on absolute value. Percent values in parentheses are percent contribution of that PC to the total variance. ^b Loading values range from 0.019 to 0.236. ^c Loading values range from 0.002 to 0.298. ^d Loading values range from 0.039 to 0.400.

contained in each principal component (Rouseff and Nagy, 1987; Moshonas and Shaw, 1997). As listed in Table 2, the seven constituents that contributed most to principal component 2 were all C₁–C₅ alcohols. In decreasing order of their contributions, they were 2-methylpropanol, 2-methylbutanol, 3-methylbutanol, methanol, propanol, pentanol, and ethanol. The average values for these alcohols were relatively high in the orange juice samples as well (Table 1) and, thus, contribute to their separation from mandarin samples by this method. The juice sample from Murcott (point a) was separated from the other fresh mandarin juices in Figure 1. The Murcott sample contained higher levels of many water-soluble volatile constituents than did the other hand-expressed samples, especially the lower molecular weight alcohols containing from one to five carbons (Table 1).

Loading values for PC1 show that the first 10 constituents listed in Table 2 were primarily oil constituents. For 7 of these 10 constituents, higher levels were found in orange than in mandarin juices when average

values were considered, as shown in Table 1. This indicates the importance of oil constituents to PC1 in the separation of orange from mandarin juices, as well as the separation discussed above involving the one commercial mandarin sample, which had high oil content, from the other mandarin juice samples.

For PC3, loading values in Table 2 show that 9 of the first 10 constituents ranked were oil constituents, with only ethyl acetate considered primarily a water-soluble constituent (Maarse and Visscher, 1989; Shaw, 1979, 1996a). Thus, loading values showed that oil-soluble compounds contributed most to PC1 and PC3, while water-soluble constituents were the major contributors to PC2. In PC analysis, after determination of PC1, each succeeding PC is calculated to contain the highest amount of variance unrelated (orthogonal) to the preceding PC (Massart *et al.*, 1988). Thus, the constituents most important to one principal component (PC1) are, in general, relatively unimportant to the adjacent principal component (PC2).

This study provides a more extensive database of volatile constituents and their amounts present in mandarin and mandarin hybrid juices than heretofore available (Maarse and Visscher, 1989; Shaw 1996a). Visual inspection of amounts of individual constituents in Table 1 shows very low levels in most juices of esters, aldehydes, and oxygenated terpenes expected to be important in the delicate and desirable flavor of mandarin juice (Shaw, 1996a). All except the Dancy, Murcott, and commercial juice samples had amounts of most volatile constituents at the low end of the range previously reported for three mandarin and mandarin hybrid samples (Shaw, 1996a; Shaw and Moshonas, 1993). It is especially noteworthy that ethanol and many volatile constituents believed important to the delicate, fruity flavor of orange, including methyl butanoate, ethyl hexanoate, octanal, decanal, and α-pinene (Ahmed *et al.*, 1978), were present in relatively low quantities when compared to amounts present in fresh orange juice. Another volatile constituent important to orange flavor is ethyl butanoate, which was not detected in the mandarin juice samples used in this study, even by mass spectral analysis. This compound was detected in some, but not all, of the mandarin juice samples analyzed in earlier studies at this laboratory (Moshonas and Shaw, 1987; Shaw and Moshonas, 1993).

Mandarin flavor, just as found for orange flavor, is a result of many volatile components present in the proper proportions needed to produce the delicious, aromatic, and highly prized flavor of this fruit (Shaw, 1991). This study corroborates and extends our earlier knowledge of the volatile components present and their low amounts relative to their amounts present in orange. This study involves quantification of volatile constituents in juices from a wider selection of mandarin and mandarin hybrid cultivars than heretofore reported. Because of the nature of the HSGC technique used, it focuses on the more volatile constituents that can be quantified. Less volatile oil-soluble constituents believed to contribute to mandarin flavor (methyl *N*-methylantranilate, thymol, and α-sinensal) are not volatile enough to be quantified by this method at the levels present in juice (Shaw, 1991). Thus, their variation among cultivars and contribution to the profile of volatile constituents were not monitored in this study.

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