

Volatile Constituents in Fresh and Processed Juices from Grapefruit and New Grapefruit Hybrids

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Forty-five volatile constituents of juices from grapefruit and grapefruit hybrids were quantified by headspace gas chromatography. The three types of grapefruit juice analyzed include pasteurized juice not from concentrate, reconstituted single strength juice from concentrate, and fresh, unpasteurized juice. Principal component and discriminant analyses were carried out using 48 grapefruit juice samples, and the samples were classified into the three types of juice based on degree of processing. Discriminant analysis was superior to principal component analysis for this purpose. Juices from two recently developed grapefruit hybrids were classified similarly to unpasteurized grapefruit juices from commercial cultivars.

Keywords: *Headspace gas chromatography; principal component analysis; discriminant analysis*

INTRODUCTION

Traditionally, commercial grapefruit juice has been prepared by diluting frozen concentrated grapefruit juice to single strength juice prior to retail packaging. Recently, grapefruit juice not from concentrate is being sold in increasing amounts and is considered a premium quality product (Brown, 1995). Unpasteurized grapefruit juice is also a commercial product but is sold in very small quantities compared to the other grapefruit juice products. There is a need for objective methods to evaluate quality of these various grapefruit products in order to help processors distinguish the characteristics of each type to produce higher quality and more consistent products for the consumer. New hybrid grapefruit cultivars which have recently been developed need to be evaluated objectively for their resemblance to commercial cultivars in overall flavor quality. Both of these needs could be met if objective methods for evaluating flavor components were sensitive enough to distinguish the various types of commercial grapefruit juice and also classify grapefruit hybrids regarding their similarity to the flavor quality of grapefruit cultivars used commercially.

Earlier studies to determine differences in flavor quality among grapefruit juice products have involved attributes contributed by nonvolatile flavor constituents such as bitterness, juice acidity, astringency, and sweetness (Sinclair, 1972). Later studies have focused on specific volatile flavor constituents such as nootkatone (Stevens et al., 1970; Shaw and Wilson, 1980) as well as *p*-menthenethiol and other trace volatile sulfur compounds (Demole et al., 1982; Shaw and Wilson, 1982). Pino and co-workers (1986) have used multiple linear regression analysis to correlate several volatile flavor components with the quality of grapefruit flavor. They

also determined relative flavor intensity values for some volatile components of grapefruit based on the flavor threshold of these compounds in water (Pino, 1982). Rouseff and co-workers (1988) used multivariate analysis to help correlate selected volatile and nonvolatile flavor components with sensory ratings of several pasteurized not from concentrate grapefruit juice products.

In the current study, the quantities of up to 45 volatile constituents present in various commercial types of grapefruit juice and in juice from two newly developed citrus hybrid fruit were determined. The results were used for multivariate analyses to classify these various types of grapefruit juice and hybrids based on these data.

MATERIALS AND METHODS

Juice Samples. Commercial grapefruit juice samples were obtained from citrus processing plants in Florida during the 1995–96 season and kept at -18°C until analyzed. The 37 commercial juice samples used in this study included 16 pasteurized juices not from concentrate and 21 reconstituted juices from concentrate. These samples were available for study as part of a larger group collected for a continuing nutritional database study (Fellers et al., 1991). The 11 juice samples from fresh grapefruit and eight samples from hybrids were obtained either by halving and hand juicing samples of 3–8 fruit using a kitchen juicer or, in two cases for fresh grapefruit juice, by purchasing the mechanically squeezed unpasteurized juice from local fruit stands adjacent to citrus groves on the day the juice was extracted from the fruit.

The new grapefruit hybrids developed by author Hearn are designated hybrids 1 and 2 in Table 1. Hybrid 1 is a cross between Nakon pummelo (male parent) and Shamouti orange (seed parent). It looks and tastes like grapefruit and is useful as a seed parent for cross-breeding with other grapefruit varieties. It is the first successful hybridization of citrus leading to a grapefruit. Numerous attempts with pummelo as the seed parent and orange as the male parent during the past 100 years failed to result in a hybrid that would be recognized as a grapefruit. Hybrid 2 is a cross between either Duncan or Marsh grapefruit (male) and Pearl tangelo (seed). It matures early in Florida (September), has less acidity and bitterness

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Table 1. Quantities (Parts per Million) of Volatile Components in Grapefruit Juices from Grapefruit and Grapefruit Hybrids

component ^a	pasteurized		reconstituted		unpasteurized		hybrid 1		hybrid 2	
	mean ^b	range	mean ^b	range	mean ^b	range	mean	range	mean	range
methanol ^{c,d}	31.5a	15.8–55.4	7b	2.2–31.7	29.6a	2.7–73.5	18.9	5.0–27.7	6.7	2.7–12.7
ethanol ^d	486a	313–826	194b	22–447	260b	21–787	165	56–207	174	173–176
1-propanol	0.24a	0.15–0.37	0.026b	nd ^e –0.094	0.099b	0.025–0.12	0.11	0.034–0.16	nd	nd
ethyl acetate ^d	1.44a	0.36–6.6	0.24b	0.009–0.81	0.27b	tr–1.45	0.091	0.032–0.16	0.001	nd–0.005
2-methylpropanol	0.012a	0.003–0.058	0.001b	nd–0.009	0.003b	tr–0.017	0.004	nd–0.007	nd	nd
butanol	0.039a	0.015–0.10	0.01b	nd–0.023	0.005b	tr–0.017	0.015	0.005–0.023	nd	nd
1-penten-3-ol	0.013a	0.003–0.022	0.002b	nd–0.017	0.013a	nd–0.040	0.09	nd–0.18	0.026	nd–0.069
1-penten-3-one	0.019a	0.014–0.029	0.011b	nd–0.016	0.011b	tr–0.020	0.032	0.020–0.041	0.012	nd–0.018
2-pentanol	tr ^f a	tr	0.005a	nd–0.11	0.002a	tr–0.024	0.1	0.053–0.15	0.044	tr–0.18
ethyl propionate	0.021a	0.015–0.048	0.004b	nd–0.02	0.007b	nd–0.028	nd	nd	0.011	nd–0.015
methyl butanoate ^d	0.007a	0.005–0.013	0.002b	nd–0.007	0.003b	nd–0.010	0.007	0.004–0.011	nd	nd
3-methylbutanol ^d	0.083a	0.020–0.35	0.009b	nd–0.055	0.031ab	nd–0.017	0.008	nd–0.030	0.004	nd–0.015
2-methylbutanol	0.01a	nd–0.052	0.004a	nd–0.036	0.003a	nd–0.029	tr	nd–0.001	nd	nd
1-pentanol	0.008a	nd–0.017	0.037a	nd–0.46	0.011a	nd–0.033	0.017	tr–0.046	0.015	0.005–0.029
3-methyl-2-buten-1-ol	0.006a	nd–0.054	0.026b	nd–0.077	0.012ab	nd–0.059	nd	nd	nd	nd
hexanal ^{d,g}	0.32a	0.23–0.50	0.2b	0.015–0.28	0.23ab	0.02–0.52	0.42	0.37–0.46	0.36	0.27–0.42
(E)-2-hexenal	0.46a	nd–0.68	nd b	nd	0.24ab	nd–0.67	nd	nd	0.18	nd–0.722
(Z)-3-hexenal	0.11a	0.087–0.18	0.016b	nd–0.15	0.25c	0.14–0.49	0.57	0.38–0.76	0.48	0.43–0.53
hexanol	0.025a	nd–0.082	0.006a	nd–0.044	0.06b	0.010–0.16	0.08	0.034–0.15	0.091	0.079–0.105
heptanal	0.012a	0.000–0.052	0.01a	tr–0.052	0.004a	tr–0.029	0.039	0.010–0.064	0.051	tr–0.164
α-pinene ^d	0.25a	0.14–0.44	0.37ab	0.045–1.06	0.57b	0.055–1.0	0.09	0.076–0.12	0.022	nd–0.046
sabinene ^d	0.004a	nd–0.011	0.38a	tr–7.9	0.029a	0.003–0.096	0.032	0.009–0.051	0.003	0.001–0.008
myrcene ^d	1.4a	0.52–3.1	2.7a	0.006–11	1.1a	0.23–3.1	0.21	0.091–0.32	0.022	tr–0.055
ethyl hexanoate	0.037a	0.021–0.058	0.039a	nd–0.25	0.018a	nd–0.036	0.023	0.019–0.028	nd	nd
octanal ^d	0.31ab	0.22–0.67	0.38a	0.18–0.81	0.21b	0.17–0.42	0.18	0.17–0.20	0.13	nd–0.18
α-phellandrene	0.06a	0.038–0.11	0.17b	0.040–0.49	0.032a	nd–0.077	0.034	0.027–0.042	nd	nd
limonene ^d	39a	17–86	81a	0.96–312	23a	2.8–83	5.6	3.6–7.5	0.97	tr–2.27
(E)-ocimene	6.2a	nd–14.7	10.2a	0.58–34.6	3a	nd–16	0.17	nd–0.39	0.96	nd–2.22
γ-terpinene	0.02a	nd–0.047	0.013a	nd–0.046	0.018a	nd–0.061	nd	nd	0.012	nd–0.048
octanol	0.61a	0.53–0.78	0.62a	0.480–1.09	0.44b	nd–0.75	1.2	0.63–2.7	0.12	nd–0.47
(Z)-linalool oxide	0.68a	0.10–2.4	1.6b	0.23–2.9	0.008c	nd–0.072	nd	nd–nd	nd	nd–nd
(E)-linalool oxide	0.85a	0.39–1.6	2.9b	nd–10.0	0.26a	0.011–1.2	0.28	nd–0.79	nd	nd
linalool ^d	0.22a	0.16–0.41	0.26a	0.12–0.58	0.11b	0.090–0.18	0.18	nd–0.53	nd	nd
nonanal ^d	0.036ab	0.018–0.10	0.051a	nd–0.19	0.009b	0.001–0.038	0.005	tr–0.011	0.004	nd–0.008
ethyl 3-hydroxyhexanoate	0.035ab	nd–0.12	0.076a	nd–0.25	0.015b	nd–0.062	0.041	nd–0.11	nd	nd
ethyl octanoate	0.062ab	0.026–0.11	0.082a	nd–0.20	0.019b	nd–0.11	0.011	0.003–0.016	nd	nd
terpinen-4ol	0.13a	nd–0.24	0.01b	nd–0.11	0.032b	nd–0.23	0.017	nd–0.068	nd	nd
decanal ^d	0.31a	tr–1.0	0.36a	0.008–0.98	0.082a	nd–0.41	0.026	0.009–0.047	0.001	tr–0.004
α-terpineol ^d	1.68a	nd–11.5	1.2a	nd–18.8	tr a	nd–tr	nd	nd	nd	nd
neral ^d	0.011a	nd–0.049	0.011a	nd–0.038	0.024a	nd–0.089	nd	nd	nd	nd
carvone ^d	0.027a	0.004–0.052	0.017ab	nd–0.057	0.003b	nd–0.013	0.005	nd–0.009	nd	nd
geranial ^d	0.004a	nd–0.037	0.006a	nd–0.059	0.019a	nd–0.096	nd	nd	nd	nd
perillaldehyde	0.01ab	0.003–0.032	0.013a	nd–0.037	0.002b	nd–0.010	nd	nd	nd	nd
caryophyllene	3.6a	3.0–4.5	1.5b	0.72–4.0	3.5a	0.66–8.7	0.34	nd–0.65	0.17	0.16–0.17
valencene	0.33a	0.080–0.530	0.15b	nd–0.270	0.019c	nd–0.160	0.095	nd–0.28	0.84	0.044–1.44

^a Listed in increasing order on a nonpolar capillary GC column. Data are means of three to five replications. ^b Means followed by the same letter in the same row are not significantly different at the 95% confidence level. ^c A minor amount of acetaldehyde coeluted with this component. ^d Reprted by Pino et al. (1986) to contribute to grapefruit flavor. ^e nd, not detected. ^f tr, trace, detected but too small to quantify. ^g A minor amount of ethyl butanoate coeluted with this component.

than major commercial grapefruit cultivars, and has potential commercial use as an early-season grapefruit-type fruit to supply to fresh grapefruit markets in Japan and Europe.

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, 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, then increased at 6 °C/min to 200 °C final temperature. Column flow rate was 8 mL/min. To purge the headspace above the juice sample, 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 15 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 purge and trap sequences were fully automated. Peak areas were used for quantitation.

Concentrations for each of the 45 compounds were calculated with regression equations, determined using standard solutions prepared by injecting four different concentrations of each compound added to a bland-tasting juice base which had almost no volatile constituents 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 grapefruit juice components were separated for identification by GC-MS using samples of juice from Duncan and Marsh grapefruit and from hybrids 1 and 2. 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 by 50 m fused silica column of cross-linked 5% phenylmethyl silicone. Column oven

temperature programming was 55 °C for 9 min, then 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 with authentic samples followed by analysis using the headspace GC system described above.

Data Analyses. Quantitative values for the 45 volatile constituents monitored in each of the 56 juice samples were entered into a Quattro Pro spreadsheet program. When a constituent was present in a sample, but too low to quantitate, it was entered as 10^{-4} in the spreadsheet (shown as tr in Table 1); if it was undetected in a sample, it was entered as 10^{-5} , or nd in Table 1. The multivariate analysis programs used were all programs contained in Statistica software (Statsoft, Inc., Tulsa, OK). ANOVA was used for calculations of significant differences between sample means in Table 1.

RESULTS AND DISCUSSION

The quantities of 45 volatile constituents determined by HSGC in 56 samples of commercial and fresh juices from grapefruit and two new grapefruit hybrids were determined. The mean and range of values for juices from each of the five types of grapefruit and hybrid juice analyzed are listed in Table 1. All of the constituents listed except caryophyllene had previously been quantified in orange juice by this method (Moshonas and Shaw, 1994; Shaw et al., 1994).

The three types of grapefruit juice listed in Table 1 are pasteurized juice not from concentrate, juice reconstituted from frozen concentrate, and unpasteurized, fresh-squeezed juice. Pasteurized juice undergoes less thermal processing than juice reconstituted from concentrate, which is pasteurized prior to concentration to produce frozen concentrate and then repasteurized after reconstitution to single strength juice prior to retail packaging (Carter, 1983). Unpasteurized juice undergoes the least processing of the three types and is considered to have the most fresh-flavor, followed by pasteurized juice not from concentrate, with reconstituted juice having the least fresh flavor notes (Shaw et al., 1995). The data in Table 1 for individual constituents show significant differences between some mean values among the three groups of 48 total juice samples analyzed, even though a wide range of values was found for each constituent in each group. Of the 45 constituents monitored, 14 showed no significant difference among the three juice types, 28 showed one type different from the other two, and only three constituents showed significant differences among all three types.

Multivariate analysis using quantities of volatile constituents present in each juice was able to separate the three types of juice samples as shown in Figures 1 and 2. Principal component (PC) analysis in Figure 1 shows the general separation achieved for the three types of juice using the first three principal components calculated from quantities of 45 volatile constituents monitored. PC analysis affords an unsupervised representation of the information obtained from the data and is useful for sample-to-sample comparisons (Burgard and Kuznicki, 1990). However, discriminant analysis, which involves assigning classes to all samples, is necessary for classification of selected sets of variables, as shown in Figure 2. In this figure, the ellipse surrounding each group of juice samples represents the 95% confidence level, showing that all three groups were statistically separated from each other by this method.

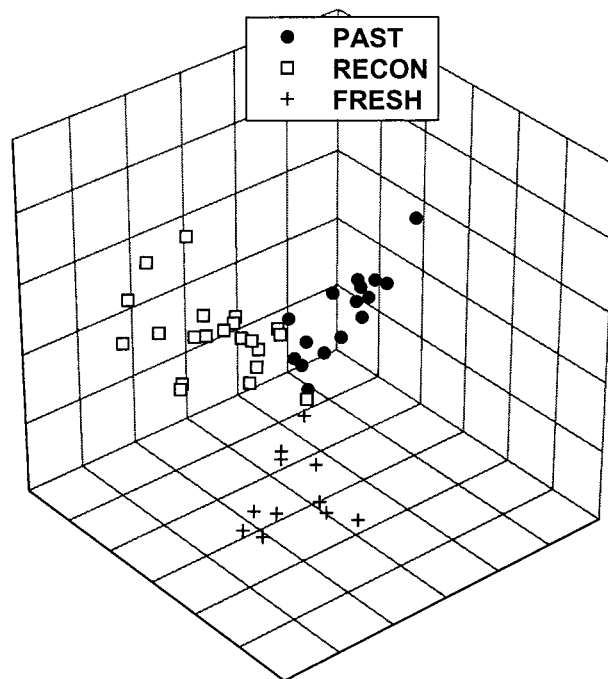


Figure 1. Principal component analysis of 48 grapefruit juice samples where PAST indicates pasteurized juice not from concentrate, RECON is juice reconstituted to single strength from frozen concentrate, and FRESH is unpasteurized juice.

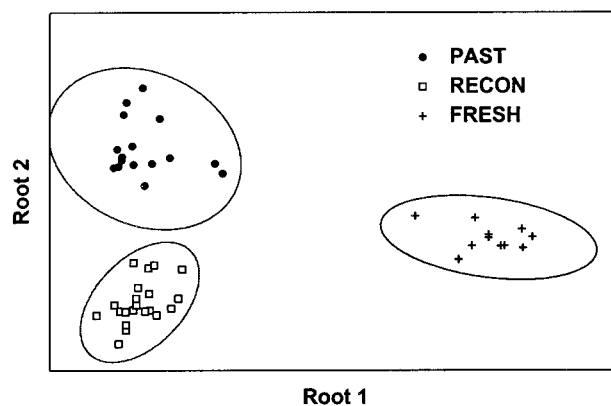


Figure 2. Forward stepwise discriminant analysis of 48 grapefruit juice samples as defined in Figure 1 legend.

The separations achieved in Figures 1 and 2 are comparable to those found earlier for three types of orange juice, similarly analyzed (Shaw et al., 1999).

In discriminant analysis, root 1 provides the most overall discrimination between the groups, and root 2 the second most discrimination. Root 1 represents 74.5% of the variance, and root 2 represents the remaining 24.5%. With forward stepwise analysis of 48 samples, 16 variables (steps) were chosen, since the number of variables chosen should not be more than one-third the number of samples analyzed (Burgard and Kuznicki, 1990). The 16 volatile constituents chosen were, in decreasing order of their contribution to root 1, (*Z*)-3-hexenol, valencene, octanol, α -pinene, octanal, caryophyllene, geranial, 1-penten-3-ol, 1-penten-3-one, ethylhexanoate, terpinen-4-ol, ethyl propionate, α -terpineol, 3-methyl-2-butenol, 1-propanol and 3-methylbutanol. (*Z*)-3-Hexenol and valencene are two of the three constituents in Table 1 which show significant differences in mean values for all three juice types. The contribution of the 16 variables to root 2 in decreasing

order are 1-propanol, terpinen-4-ol, ethyl propionate, valencene, 1-penten-3-one, (*Z*)-3-hexenol, caryophyllene, 3-methylbutanol, 1-penten-3-ol, 3-methyl-2-butenol, α -pinene, octanal, α -terpineol, geranial, octanol, and ethylhexanoate.

Pino et al. (1986) monitored 32 volatile constituents in canned grapefruit juice and determined the relative importance of flavor contribution for 28 of them. Eighteen of the 45 constituents quantified in this study were among those monitored in their study as well. Footnote d in Table 1 indicates those 18 constituents, and six of them are among the 16 constituents listed above as contributing to the separation achieved by discriminant analysis. Most of these 16 constituents are considered as important contributors to citrus flavors (Shaw, 1991). The 12 remaining constituents evaluated by Pino et al. (1986) included nootkatone, a known flavor impact compound in grapefruit. Nootkatone is among the lower half of the 28 constituents they studied with regard to relative flavor contribution. In the current study, nootkatone was present at levels too small to be quantified by the HSGC technique employed.

Grapefruit Hybrids. Two new grapefruit hybrids were included in this study to determine how closely the profile of volatile constituents matched those for commercial grapefruit. Hybrid 2 has potential for commercial development because it matures very early and has a relatively low bitter flavor, while hybrid 1 has potential as a parent for the development of other new grapefruit hybrids. Quantities of up to 45 volatile constituents identified in juice from four samples of each hybrid fruit are listed in Table 1 for comparison with unpasteurized grapefruit juice. Nine of the 45 volatile constituents were not detected in hybrid 1 and 19 were not detected in hybrid 2. The average values for 14 other constituents were lowest in hybrid 2. The relatively mild grapefruit flavor noted in juice from this hybrid fruit is reflected in these low or undetected levels. On the basis of the volatile constituents, hybrid 1 resembles commercial grapefruit more than does hybrid 2.

Multivariate analysis of 19 unpasteurized grapefruit and hybrid juice samples was carried out after elimination of all components (19) undetected in at least one of the three unpasteurized juice types, as indicated by a mean value of nd in Table 1. This adjustment was necessary for the discriminant analysis program to function. Principal component analysis of the 26 remaining constituents quantified in juice from the grapefruit and both hybrid fruit types did not show as clear of a separation between the main groups of juices. This is as expected since 19 variables were not used to perform the PCA analysis of any one juice type from the others. The hybrid samples did not show a clear association with any one group, but could be seen to be closest to the fresh category.

Mahalanobis distances can be used for classification of unknown samples, hybrids 1 and 2 in this instance (Statistica Manual, 1995). Each of the three known types of grapefruit juice discussed herein have different group centroids in discriminant analysis (Table 2). The Mahalanobis distances were calculated by discriminant analysis using all 56 samples, where the eight hybrid samples were not assigned to one of the three classes. Seven of the eight samples were found to be closest to the group centroid corresponding to fresh grapefruit juice. The remaining sample (H1C) was classified as

Table 2. Classification of Hybrid Fruit Juice Samples as Unknowns Using Discriminant Analysis Mahalanobis Distances

discriminant analysis case ^a	squared Mahalanobis distances from group centroids		
	past ^b	recon ^b	fresh ^b
H1A	565	559	282
H1B	268	255	159
H1C	761	692	813
H1D	297	274	130
H2A	413	415	137
H2B	543	576	265
H2C	1080	1128	896
H2D	1240	1233	881

^a Classified as unknown groups. ^b Known groups where past indicates pasteurized juice not from concentrate, recon is juice reconstituted to single strength from frozen concentrate, and fresh is unpasteurized juice.

slightly closer to the centroid for reconstituted juice from concentrate than for that of fresh or pasteurized juices.

This study extends the application of our earlier studies with grapefruit juice by showing the ability of multivariate analysis programs to classify citrus juices by degree of processing used, based on amounts of volatile constituents present. Principal components analysis is useful for sample-to-sample comparisons and to verify the correctness of class assignments, while discriminant analysis is capable of categorizing and determining the prediction capability of a group of variables. These techniques have potential for use in quality control, product development, and evaluation and comparison of juice from new citrus hybrids.

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JF0001076