

Comparison of Volatile Concentrations in Hand-Squeezed Juices of Four Different Lemon Varieties

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Fresh juices of four Italian cultivars of lemons (*Citrus limon* Burm) have been analyzed by headspace solid phase microextraction coupled to gas chromatography and gas chromatography–mass spectrometry (GC-MS). The best results have been obtained with the 2 cm 50/30 μm divinylbenzene/carboxen on poly(dimethylsiloxane) fiber, using a homogenization time of 1 h at 40 °C and a sampling period of 30 min. A total of 35 volatile compounds have been identified by GC-MS, and their relative amounts have been calculated by adding internal standard to the samples. Differences in composition of lemon juices volatile components have been observed. Verdello Siracusano lemon juice has the highest amount of volatile compounds (50.28 mg/L), followed by Interdonato (8.39 mg/L), Primo Fiore Capo d'Orlando (5.75 mg/L), and Femminello Siracusano (2.62 mg/L) juices. Volatile compounds mainly consist of mono- and sesquiterpene hydrocarbons and oxygenated molecules (aldehydes, monoterpene alcohols, and monoterpene esters). Headspace solid phase microextraction coupled to a gas chromatograph equipped with a specific sulfur detector, a sulfur chemiluminescence detector, let us detect and quantify dimethyl sulfide compound at the microgram/liter level in lemon juices.

KEYWORDS: Headspace; SPME; lemon juice; volatile compound

INTRODUCTION

Lemons (*Citrus limon* Burm; Rutaceae) are cultivated in many countries all over the world, in regions with temperate summers and mild winters, particularly in Mediterranean countries, southern California, and Argentina.

Lemon essential oils are widely used as aroma flavor enhancers for soft and alcoholic beverages and food. In pharmaceutical industries, they are used as flavoring agents to mask unpleasant tastes of drugs. In perfumery, they form the base of many compositions. Commercial oils are obtained by cold pressing (peel) or distillation (leaves). The composition of lemon peel oil has been the subject of numerous studies reviewed by Lawrence (1–3), although most of these studies concerned commercial oils for which neither the species nor the varieties were specified. A few other studies were carried out on specified varieties (4, 5), and a recent study reports on the volatile composition of nine species of lemons (6). Conversely, although lemon juices are the main commercial products of lemons, only few studies (7, 8) have been carried out on the lemon juices volatile fraction.

Recently, the solid phase microextraction (SPME) technique was introduced as an alternative to the headspace (HS) technique

as a sample preconcentration method prior to chromatographic analysis. SPME is a rapid, sensitive, and solvent-free sampling technique developed by Arthur and Pawliszyn (9) for the analysis of pollutants in water. In recent years, SPME has extended its applications to numerous other fields, such as food flavor analysis (10–13). This technique has been successfully used for the analysis of volatiles in HS citrus juice (14–18). However, data on the application of SPME in lemon juices HS analysis are not available. In the present study, SPME, followed by gas chromatography (GC) and gas chromatography–mass spectrometry (GC-MS), was evaluated for the qualitative and quantitative analysis of four varieties of Sicily lemons juices. The objective of this work was to identify the volatile components of lemon juices and to investigate the differences between volatile compounds composition inside the varieties. These data may be useful in detection of fruit juice adulteration or off-flavors relevance, as a primary objective in beverage and/or juice quality control is to ensure the authenticity of the juice products and to be able to detect adulteration. Moreover, a specific gas chromatographic sulfur detector has been applied to detect sulfur compounds in lemon juices, as Shaw (19) reports of volatile sulfides and hydrogen sulfide in different citrus juices analyzed.

MATERIALS AND METHODS

Materials and Sample Preparation. The following cultivars of early seasons lemons collected from the same Sicily region area (Siracusa,

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Italy) were investigated: Verdello Siracusano, Interdonato, Primo Fiore Capo d'Orlando, and Femminello Siracusano. Fruits were picked up from three different plants of each variety in the same ripening period. Fresh juices samples were prepared in the laboratory, squeezing by hand six washed fruits, and the ripening levels of the samples were determined by Brix analysis. Six replicate samples were performed for each variety and immediately analyzed.

Volatile compounds of reagent grade (ethyl isovalerate, dimethyl sulfide) were purchased from Aldrich Chemical Co. (Milan, Italy). Reference chemicals for coinjection in GC and mass spectrometry were obtained from reliable commercial sources such as Sigma-Aldrich (Milan, Italy) and Extrasynthese SA (Genay, France).

HS-SPME Procedure. A manual holder SPME (Supelco, Bellefonte, PA) was used for all of the experiments. As compared to all of the commercially available fibers, the 2 cm 50/30 μm divinylbenzene/carboxen on poly(dimethylsiloxane) Stable Flex fiber has shown the best efficiency to extract the selected compounds.

Fresh juices were centrifuged at 3000g and 4 °C for 15 min. A 25 mL supernatant aliquot was added to 25 μL of internal standard (ethyl isovalerate 1% w/w in ethanol) and transferred into a 50 mL vial. To favor the transfer of the analytes from the aqueous solution to the HS, 6 g of NaCl was added. The vials were crimp-closed with a Teflon-lined silica cap (Supelco) and equilibrated at 40 °C for 60 min with constant stirring. The SPME fiber was exposed to the sample HS at a constant depth for 30 min. The equilibration conditions used for temperature and agitation were maintained during extraction of the volatile compounds.

The fiber was then withdrawn and directly desorbed into the injection port of the GC in splitless mode for 2 min at 250 °C. Then, the split was open but the fiber was kept for 17 min in the injector for cleaning.

GC-Linear Retention Index (RI) Analysis. A Hewlett-Packard 6890 gas chromatograph (Hewlett-Packard, Palo Alto, CA), equipped with two fused-silica capillary columns (DB-1 and DB-1701, J&W: 30 m \times 0.25 mm i.d.; thickness, 0.25 μm) were installed in the same injection port, and two flame ionization detectors (FIDs) were used to analyze lemon juices samples.

Injector splitless for 2 min and detector temperatures were 250 and 280 °C, respectively. Helium carrier gas was used (1 mL min^{-1}), and the temperature program was 50 °C for 3 min followed by an increase of 5 °C/min to 280 °C for the remainder of the run. The double column signals were recorded and elaborated simultaneously. Kovats' RIs of peaks, referred to *n*-alkanes, were calculated (20) and compared with those of the authentic standards chromatographed under identical conditions on DB-1 and DB-1701 columns.

GC-MS. A Varian 3400 GC coupled with a Finnigan SSQ7000 quadrupole mass spectrometer (Thermo Finnigan, San Jose, CA) was used. The carrier gas was helium (1 mL min^{-1}). The injector temperature was 250 °C, and it was operated at splitless mode for 2 min. Analytes were separated on a DB-1 column (J&W: 30 m \times 0.25 mm i.d.; thickness, 0.25 μm) and kept at 50 °C for 3 min and then ramped to 200 °C at 5 °C/min. The transfer line was kept at 275 °C. All of the spectra were recorded in the electron impact mode at an ionization voltage of 70 eV and at the rate of 1 scan/s over the mass range (*m/z*) 33–350.

Compounds Identification and Quantification. Compounds were identified by comparison of their mass spectra and retention times with those of standard compounds or by comparison of the mass spectrum with those of the mass spectrum of NIST 98 Library. Moreover, Kovats' RIs were determined on two chromatographic capillary columns with distinct polarities and compared with RIs of the reference compounds or with those available in the literature. The relative concentrations of the compounds in the samples were calculated by comparing the peak area of each compound with that of the internal standard (ethyl isovalerate, 7.8 mg/L). The results were expressed as the average of triplicate runs for each of the six samples.

GC/Sulfur Chemiluminescence Detector (SCD)/FID. A Hewlett-Packard 6890 gas chromatograph was equipped with two equal fused-silica capillary columns DB-1 (J&W: 30 m \times 0.25 mm i.d.; thickness, 1 μm) installed in the same injector port maintained at 250 °C and connected, respectively, to a FID detector (280 °C) and a SCD Antek 8040 (Antek Instruments, PAC Company, Houston, TX) operated with

a combustion chamber at 1050 °C and a vacuum fixed on 190 Torr. Capillary GC separation was carried out under conditions analogous to those reported in the previous paragraph.

Calibration Curve Dimethyl Sulfide. Reference solutions of 1, 10, and 100 $\mu\text{g/L}$ dimethyl sulfide in water were prepared by adding 25 mL of water and 6 g of NaCl to a 2.5 μL solution of 10, 100, and 1000 mg/L dimethyl sulfide in heptane; they were transferred into 50 mL vials sealed by a Teflon septum and an aluminum cap and analyzed under conditions analogous to those reported in the SPME procedure by a gas chromatograph equipped with both SCD and FID detectors. Peak areas obtained on SCD detector were related to the area of heptane on an FID detector to reduce the variability of the single injection. The calibration line of dimethyl sulfide was finally obtained by plotting corrected peak areas vs different concentrations. The limit of detection, calculated according IUPAC guidelines (21), was 0.01 $\mu\text{g/L}$ considering a signal higher than three times the background noise, and the limit of quantification (21) was 0.1 $\mu\text{g/L}$.

To avoid possibly a matrix effect, 25 mL of each of the four lemon juices, added to 6 g of NaCl, was spiked with 2.5 μL of a solution of 10, 100, and 1000 mg/L dimethyl sulfide in heptane according to the method of standard additions. The resulting calibration curves are given.

Quantification of Dimethyl Sulfide in Lemon Juices. Aliquots of 25 mL of lemon juices prepared as described on SPME procedure were added to 2.5 μL of heptane and analyzed by a gas chromatograph equipped with SCD and FID detectors. Corrected areas of dimethyl sulfide were reported to the calibration curve. The results were expressed as the average of triplicate runs for each of the six samples. RSD% was less than 5%.

Statistical Analysis. The concentrations of the components detected in lemon juices were statistically tested by univariate analysis and multivariate analysis, performed using Mathematica 5.2 software (Wolfram Research Inc.) and R 2.1.1 software (free language and environment for statistical computing and graphics developed by an international community). These analyses showed significant differences ($P < 0.05$) among the average values for all of the variables. Principal component analysis was performed by Mathematica 5.2 software integrated with self-written programming language and plot results.

RESULTS AND DISCUSSION

Composition of Volatile Compounds in Lemon Juices. A total of 35 components (10 monoterpene hydrocarbons, four sesquiterpene hydrocarbons, seven monoterpene alcohols, seven aldehydes, five esters, and two miscellaneous) were identified in lemon juices by HS-SPME coupled to GC-FID and GC-MS. The identified volatile compounds and their relative amounts in mg/L are given in **Table 1**. The components are listed in order of their elution on the DB-1 column. A classification based on functional groups is summarized in **Table 2**. Components semiquantitative evaluation was carried out by comparing the peak area of each compound with that of the internal standard, assuming a response factor equal to 1.

Verdello Siracusano lemon juice has the highest amount of volatile compounds (50.28 mg/L) followed by Interdonato (8.39 mg/L), Primo Fiore Capo d'Orlando (5.75 mg/L), and Femminello Siracusano (2.62 mg/L). Volatile compounds mainly consist of mono- and sesquiterpene hydrocarbons and oxygenated molecules.

Monoterpene hydrocarbons (limonene, α -thujene, α -pinene, camphene, β -pinene, β -myrcene, α -terpinene, β -ocimene, γ -terpinene, and terpinolene) constitute the main volatile group, representing 70–90% of total volatiles in all four juices. Their amounts are, respectively, 45.42 mg/L for Verdello Siracusano, 6.54 mg/L for Interdonato, 4.89 mg/L for Primo Fiore Capo d'Orlando, and 1.81 mg/L for Femminello Siracusano. Limonene is by far the most abundant monoterpene in all juices (54.1–68.8% of total), followed by γ -terpinene (6.8–11.4%), β -pinene (2.6–6.5%), terpinolene (1–1.7%), and β -myrcene (1–1.5%).

Table 1. Volatile Components Amounts mg/L Detected in Four Lemon Juice Varieties

peak no.	components	RI ^a		method of identification	lemon juice			
		DB-1	DB-1701		Verdello Siracusano	Interdonato	Primo Fiore Capo d'Orlando	Femminello Siracusano
1	ethanol	<500	553	RI, MS ^b	0.157	0.130	0.001	0.116
2	acetic acid	530	640	RI, MS ^b	0.004	ND	ND	ND
3	α -thujene	926	938	RI, MS ^c	0.083	0.004	0.003	ND
4	α -pinene	933	943	RI, MS ^b	0.285	0.033	0.023	0.007
5	camphene	945	970	RI, MS ^b	0.029	0.003	0.003	ND
6	β -pinene	972	999	RI, MS ^b	3.280	0.270	0.170	0.070
7	β -myrcene	985	1016	RI, MS ^b	0.741	0.116	0.076	0.026
8	α -terpinene	1012	1036	RI, MS ^b	0.027	0.009	0.091	0.028
9	limonene	1032	1065	RI, MS ^b	34.597	5.449	3.839	1.421
10	β -ocimene	1043	1075	RI, MS ^b	0.017	0.010	0.008	0.004
11	γ -terpinene	1055	1090	RI, MS ^b	5.731	0.567	0.598	0.252
12	terpinolene	1081	1113	RI, MS ^b	0.502	0.083	0.081	0.045
13	nonanal	1084	1189	RI, MS ^b	0.012	0.004	0.017	ND
14	linalool	1086	1192	RI, MS ^b	0.021	0.027	0.051	0.064
15	fenchol	1097	1218	RI, MS ^b	0.062	0.005	0.029	0.013
16	borneol	1150	1283	RI, MS ^b	0.117	0.28	0.071	0.06
17	4-terpineol	1163	1266	RI, MS ^b	0.372	0.115	0.157	0.098
18	α -terpineol	1175	1298	RI, MS ^b	0.846	0.242	0.274	0.215
19	decanal	1186	1287	RI, MS ^b	0.064	0.003	0.005	ND
20	octyl acetate	1194	1287	RI, MS ^c	0.014	0.002	0.002	ND
21	nerol	1210	1316	RI, MS ^b	0.024	0.010	0.016	0.021
22	neral	1214	1365	RI, MS ^b	0.014	0.017	0.030	0.013
23	geraniol	1236	1332	RI, MS ^b	0.070	0.007	0.020	0.035
24	9-methyldecanal	1236	1331	RI, MS ^c	0.032	ND	ND	ND
25	geranial	1244	1399	RI, MS ^b	0.037	0.042	0.080	0.027
26	undecanal	1286	1390	RI, MS ^b	0.014	ND	0.002	ND
27	nonyl acetate	1294	1387	RI, MS ^b	0.003	ND	ND	ND
28	citronellyl acetate	1336	1428	RI, MS ^b	0.030	0.045	0.007	0.003
29	neryl acetate	1347	1438	RI, MS ^b	0.284	0.281	0.011	0.068
30	geranyl acetate	1363	1454	RI, MS ^b	0.054	0.101	0.005	0.020
31	dodecanal	1387	1492	RI, MS ^b	0.042	0.014	0.002	ND
32	β -caryophyllene	1428	1471	RI, MS ^b	0.186	0.028	0.01	0.003
33	α -bergamotene	1435	1466	RI, MS ^c	0.966	0.206	0.024	0.006
34	α -zingiberene	1493	1531	RI, MS ^c	0.028	ND	0.003	ND
35	β -bisabolene	1503	1540	RI, MS ^c	1.414	0.288	0.03	0.008

^a RIs were calculated from GC-FID data. ^b Compounds were identified by comparison with the authentic standards by RI on DB-1 and DB-1701 columns and mass spectra in the electron impact mode. Positive identifications indicate that mass spectral data were compared with authentic standards. ^c Compounds were tentatively identified when reference compounds were not available; ND, not detected.

Table 2. Constitution of Functional Groups and Relative Amounts (mg/L) in the HS of Four Lemon Juice Varieties

functional group	lemon juice			
	Verdello Siracusano	Interdonato	Primo Fiore Capo d'Orlando	Femminello Siracusano
monoterpene hydrocarbons	45.294	6.544	4.892	1.853
sesquiterpene hydrocarbons	2.594	0.522	0.067	0.017
monoterpene alcohols	1.512	0.686	0.618	0.506
aldehydes	0.215	0.080	0.136	0.040
esters	0.385	0.429	0.025	0.091
miscellaneous	0.161	0.130	0.001	0.116
total	50.287	8.391	5.739	2.623

Sesquiterpene hydrocarbons (β -caryophyllene, α -bergamotene, α -zingiberene, and β -bisabolene) are the second most important group in Verdello Siracusano juice (2.59 mg/L) but not for the others, with β -bisabolene representing 0.3–3.4% and α -bergamotene representing 0.2–1.9% of the total.

Oxygenated compounds are mainly aldehydes (nonanal, decanal, 9-methyldecanal, undecanal, dodecanal, neral, and geranial), monoterpene alcohols (linalool, fenchol, borneol, 4-terpineol, α -terpineol, nerol, and geraniol), and esters (octyl acetate, nonyl acetate, citronellyl acetate, neryl acetate, and geranyl acetate). Oxygenated compounds have a percentage distribution higher in Femminello Siracusano, (24.28%), followed by Interdonato (15.79%), Primo Fiore Capo d'Orlando (13.59%), and Verdello Siracusano (4.20%). The monoterpene

alcohols show a similar trend of 19.29, 8.17, 10.77, and 3.00% of the volatile compounds in the four juices, respectively. Aldehydes are prevalent on Verdello Siracusano (0.22 mg/L) and Primo Fiore Capo d'Orlando (0.14 mg/L) juices while esters are mainly present on Interdonato juice (0.43 mg/L) and Verdello Siracusano juice (0.39 mg/L).

The PCA revealed that over 99% of the point variability could be explained by two components (**Figure 1**). The Verdello variety is the most separated cluster even though more spread than the others. **Figure 2** could explain the significance of all compounds in the calculation of PCA where limonene causes a left shift of Verdello's cluster. Other relevant compounds are the monoterpenes hydrocarbons β -pinene and γ -terpinene, and

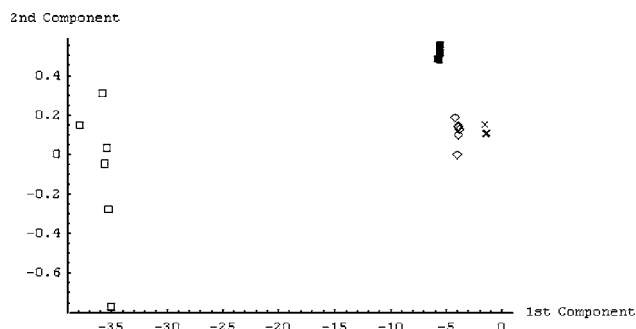


Figure 1. PCA of volatile components of four varieties of lemons (□, Verdello; ■, Interdonato; ◇, Capo d'Orlando; and ×, Femminello).

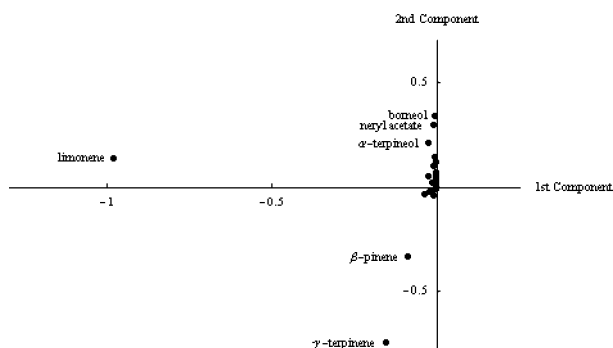
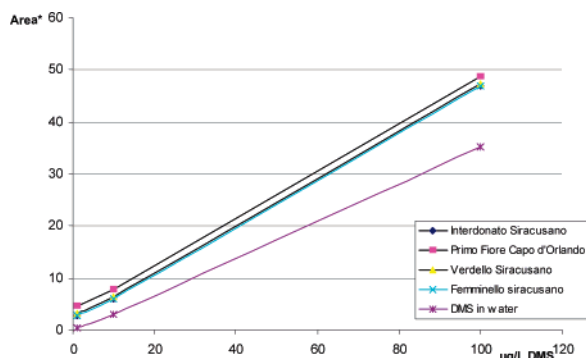


Figure 2. Plot of the substance weight.

Table 3. Calibration Curves of Dimethyl Sulfide (DMS)



*The area counts are corrected according heptane area counts.

this means that this functional group distinguishes lemon's variety more than other groups.

Quantification of Dimethyl Sulfide in Lemon Juices. As few studies (19) report of volatile sulfides on citrus juices, a specific gas chromatographic SCD has been applied for the first time to the study of fresh lemon juices by HS-SPME.

The SCD detector detected just one sulfur compound on lemon juices, and this was attributed to dimethyl sulfide and confirmed by adding it to the sample. To estimate the concentration of dimethyl sulfide in the different lemon juices, first, a calibration curve of dimethyl sulfide in water and salt system was prepared and the linear regression line and R^2 obtained are shown. Second, calibration curves with the standard addition method for each juice were also prepared and reported together in **Tables 3** and **4**. Therefore, raw juices were used and salt was added to help release volatile compounds as completely as possible, all together to minimize the matrix effect. **Table 5** reports the amounts of dimethyl sulfide in different lemon juices: Primo Fiore Capo d'Orlando has the highest amount of dimethyl sulfide (10.1 $\mu\text{g/L}$), followed by

Table 4. Data of the Calibration Curves of DMS Obtained by Standard Addition of 1, 10, and 100 $\mu\text{g/L}$ to Lemon or Water

lemon juice	calibration curve	R^2
Interdonato	$y = 0.4501x + 1.9289$	$R^2 = 0.9997$
Primo Fiore Capo d'Orlando	$y = 0.4495x + 3.6967$	$R^2 = 0.9997$
Verdello Siracusano	$y = 0.4502x + 2.3189$	$R^2 = 0.9997$
Femminello Siracusano	$y = 0.45x + 1.8756$	$R^2 = 0.9997$
DMS in water	$y = 0.3541x - 0.2839$	$R^2 = 0.9998$

Table 5. Dimethyl Sulfide Amounts ($\mu\text{g/L}$) in Lemon Juice Varieties

lemon juices	dimethyl sulfide
Primo Fiore Capo d'Orlando	10.1
Verdello Siracusano	7.2
Femminello Siracusano	5.9
Interdonato	6.5

Verdello Siracusano (7.2 $\mu\text{g/L}$), Interdonato (6.5 $\mu\text{g/L}$), and Femminello Siracusano (5.9 $\mu\text{g/L}$).

The results obtained show that HS-SPME followed by GC or GC-MS can be successfully used in the analysis of volatile components of lemon juices. Differences in the composition of volatile molecules from various cultivars were observed, and these data could be used for the characterization of each varieties. Differences in dimethyl sulfide amounts at very low levels ($\mu\text{g/L}$) were also outlined by using a specific detector.

LITERATURE CITED

- Lawrence, B. M. Lemon oil. *Essential Oils 1979–1980*; Allured Publishing: Carol Stream, IL, 1981; p 30. (b) Lawrence, B. M. *Essential Oils 1988–1991*; Allured Publishing: Carol Stream, IL, 1983; pp 61–69.
- Lawrence, B. M. Lemon oil. *Essential Oils 1992–1994*; Allured Publishing: Carol Stream, IL, 1995; p 30. (b) Lawrence, B. M. *Essential Oils 1988–1991*; Allured Publishing: Carol Stream, IL, 1983; pp 1–4, 114–117, 119–121, 123–125, 158–162.
- Lawrence, B. Progress in essential oils, lemon oil. *Perfum. Flavor* **1996**, 21 (1–2), 41–45.
- Dellacassa, E.; Lorenzo, D.; Moyna, P.; Verzera, A.; Mondello, L.; Dugo, P. Uruguayan essential oils. Part VI. Composition of lemon oil. *Flavour Fragrance J.* **1997**, 12, 247–255.
- Starrantino, A.; Terranova, G.; Dugo, P.; Bonaccorsi, I.; Mondello, L. On the genuineness of Citrus essential oils. Part II. Chemical composition of the essential oil of new hybrids of lemon obtained in Sicily. *Flavour Fragrance J.* **1997**, 12, 153–161.
- Lota, M. L.; de Rocca Serra, D.; Tomi, F.; Jacquemond, C.; Casanova, J. Volatile components of peel and leaf oils of lemon and lime species. *J. Agric. Food Chem.* **2002**, 50, 796–805.
- Moshonas, M. G.; Shaw, P. E. Analysis of flavor constituents from lemon and lime essence. *J. Agric. Food Chem.* **1972**, 20, 1029–1030.
- Shaw, P. E. Citrus fruits. In *Volatile Compounds in Food and Beverages*; Dekker: New York, 1991; Vol. 44, pp 305–327.
- Arthur, C. L.; Pawliszyn, J. Solid-phase microextraction with thermal desorption using fused silica optical fibers. *Anal. Chem.* **1990**, 62, 2145–2148.
- Yang, X.; Peppard, T. Solid-phase microextraction for flavor analysis. *J. Agric. Food Chem.* **1994**, 42, 1925–1930.
- Penton, Z. Flavor volatiles in a fruit beverage with automated SPME. *Food Test Anal.* **1996**, 6, 16–18.
- Field, J. A.; Nickerson, G.; James, D. D.; Heider, C. Determination of essential oils in hops by headspace solid-phase microextraction. *J. Agric. Food Chem.* **1996**, 44, 1768–1772.
- Roberts, D. D.; Pollien, P.; Milo, C. Solid-phase microextraction method development for headspace analysis of volatile flavour compounds. *J. Agric. Food Chem.* **2000**, 48, 2430–2437.

- (14) Rega, B.; Fournier, N.; Guichard, E. Solid phase microextraction (SPME) of orange juice flavour: Odor representativeness by direct gas chromatography olfactometry (D-GC-O). *J. Agric. Food Chem.* **2003**, *51*, 7092–7099.
- (15) Rega, B.; Fournier, N.; Nicklaus, S.; Guichard, E. Role of pulp in flavor release and sensory perception in orange juice. *J. Agric. Food Chem.* **2004**, *52*, 4204–4212.
- (16) Jordan, M.; Tillman, T. N.; Mucci, B.; Laencina, J. Using HS-SPME to determine the effects of reducing insoluble solids on aromatic composition of orange juice. *Lebens.-Wiss. Technol.* **2001**, *34*, 244–250.
- (17) Jia, M. Y.; Zhang, Q. H.; Min, D. B. Optimization of solid-phase microextraction analysis for headspace flavor compounds of orange juice. *J. Agric. Food Chem.* **1988**, *36*, 2744–2747.
- (18) Steffen, A.; Pawliszyn, J. Analysis of flavor volatiles using headspace solid-phase microextraction. *J. Agric. Food Chem.* **1996**, *44*, 2187–2193.
- (19) Shaw, P. E.; Wilson, C. W. Volatile sulfides in headspace gases of fresh and processed citrus juices. *J. Agric. Food Chem.* **1982**, *30*, 685–688.
- (20) Kovats, E. Gas chromatography characterization of organic compounds. *Helv. Chim. Acta* **1958**, *41*, 1915–1932.
- (21) IUPAC Compendium of Chemical Technology, 1985.

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