

## Effect of Refrigerated and Thermal Storage on the Volatile Profile of Commercial Aseptic Korean Soymilk

Hun Kim<sup>1†</sup>, Keith R. Cadwallader<sup>1</sup>, Eun-Jeong Jeong<sup>2</sup>, and Yong-Jun Cha<sup>2</sup>

<sup>1</sup>Department of Food Science and Human Nutrition, University of Illinois, Illinois 61801, USA

<sup>2</sup>Department of Food and Nutrition, Changwon National University, Gyeongnam 641-773, Korea

### Abstract

This study determined the effect of refrigerated and thermal storage on the volatile profile of commercial aseptic soymilk. Volatile components in commercial aseptic soymilk stored either under refrigerated (4°C) or thermal (55°C) conditions for 30 days were periodically analyzed by combined solvent-assisted flavor evaporation-gas chromatography-mass spectrometry (SAFE-GC-MS). The concentrations of most of the volatile components, including aldehydes, ketones, alcohols, acids, nitrogen- and sulfur-containing compounds, alkylfurans, furan derivatives and phenolic compounds, were affected to a greater extent by thermal storage compared with refrigerated storage. Profound increases in some volatile compounds with low odor detection thresholds, such as hexanal, octanal, (*E*)-2-octenal, (*E,E*)-2,4-decadienal, 1-octen-3-ol, 3-ethyl-2,5-dimethylpyrazine, 2,3-diethyl-5-methylpyrazine, 2-pentylfuran, 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, dimethyl trisulfide, guaiacol, 4-vinylguaiacol and 4-vinylphenol, were observed in thermal stored soymilk. The volatile profile changes caused by thermal storage may influence the aroma quality of thermal-stored aseptic soymilk.

**Key words:** soymilk, thermal storage, refrigerated storage, volatile compounds, solvent-assisted flavor evaporation

### INTRODUCTION

Soymilk, called *Kong-guk* in Korea, is the aqueous extract of whole soybeans traditionally produced by grinding soaked soybeans with water (1,2). First commercialized in the early 1970's, soymilk is as good alternative for people who are allergic to or have difficulty digesting dairy milk (2,3). In the past decade the Korean soymilk market has experience consistent growth (4). This trend is most likely a result of the heightened consumer awareness of the many health benefits associated with the consumption of soy products, such as the reduction or prevention of the incidence of certain types of cancers, reduction of plasma cholesterol levels, improvement in bone health, and alleviation of symptoms of menopause (5). In addition, part of this growth can be attributed to the Korean soymilk industry's attempt at satisfying consumer demand for functional soy foods, including soymilks enhanced by addition of other functional ingredients like green tea, black sesame, black bean or ginger (1,6,7).

Over centuries, Korean consumers have developed a variety of ways in which they consume soymilk, including a preference of many people for hot or warmed soymilk (~50 to 60°C) rather than cold or room temperature soymilk. For this reason, some commercial soy-

milks packaged in cans or cartons and having a long shelf-life (more than 6 months) are stored in heating chambers on the market (especially in highway service areas). Although this practice provides the consumer with a soymilk at a desired serving temperature, there is some concern over quality degradation due to prolonged thermal storage. Thermal storage may result in changes in the sensory properties, especially flavor, due to degradation of the abundant protein, lipids and carbohydrates of soymilk (8) via Maillard reaction and lipid oxidation (9) which can occur at moderate temperatures (20~60°C) (10). Despite the potential importance of this quality degradation, no studies have fully examined the effect of thermal storage on the volatile profile of commercial soymilk.

Solvent-assisted flavor evaporation (SAFE), developed by Engel et al. (11), is versatile technique for the careful and direct isolation of volatile compounds from complex food matrices (12,13). SAFE gives higher yields of volatiles from complex fat-containing matrices compared with previous high vacuum distillation techniques (11) and can be operated under mild conditions (~35 to 40°C) thus minimizing formation of thermally-induced artifacts. SAFE combined with subsequent solvent extraction has been previously used for the isolation of volatile constituents from fresh and ultrahigh temper-

†Corresponding author. E-mail: hunkim@illinois.edu  
Phone: +1-217-333-1642, Fax: +1-217-333-1875

ature (UHT) processed soymilks (14).

The aim of the present study was to identify the effect of thermal storage compared with refrigerated storage on the volatile profile of commercial aseptic Korean soymilk. Soymilks were stored either in a refrigerator ( $\sim 4^{\circ}\text{C}$ ) or in a heating chamber ( $55^{\circ}\text{C}$ ) for 30 days, and the volatile profile periodically monitored by combined SAFE-gas chromatography-mass spectrometry (SAFE-GC-MS).

## MATERIALS AND METHODS

### Materials

Commercial aseptic soymilk packaged in Tetra Pak (200-mL) cartons was purchased from a local Korean market and was subsequently divided into two groups and stored either in an oven (thermal storage,  $55 \pm 1^{\circ}\text{C}$ ) or a refrigerator (control,  $4 \pm 0.5^{\circ}\text{C}$ ). The volatile compounds of both groups were analyzed every 10 days throughout the storage period.

Reagent grade chemicals were obtained from Fisher Scientific (Pittsburgh, PA). Reference volatile compounds and diethyl ether were purchased from Sigma-Aldrich (St. Louis, MO).

### Solvent-assisted flavor evaporation (SAFE)

Volatile compounds from soymilk were isolated using a modified SAFE (Ace Glassware, Vineland, NJ) system (14). The SAFE apparatus (consisting of a transfer head and a 5-L round-bottom flask) was connected to a receiving tube and a waste tube. The glassware was then connected to a rough pump and turbo pump as the vacuum source. The receiving tube and waste tube were held in separate Dewar flasks containing liquid nitrogen throughout the distillation period. The transfer head and round-bottom flask of SAFE system were kept at  $40^{\circ}\text{C}$  using circulating water baths. Soymilk (800 g), 100 g of sodium chloride (NaCl) and 10  $\mu\text{L}$  of internal standard solution (50.30 mg of 2-methyl-3-heptanone and 51.64 mg of 2-ethylbutyric acid in 10 mL of methanol) were loaded into the top of the transfer head and released into the flask until all of soymilk mixture had been placed under vacuum condition ( $\sim 10^{-5}$  Torr). Distillation was carried out for 4.5 hr per sample. SAFE distillation was conducted in duplicate for each sample.

### Fractionation of SAFE distillate

After SAFE distillation, the receiving tube was disconnected from the system and thawed overnight after addition of 50 mL of diethyl ether and 80 g of NaCl. The acidic and neutral-basic volatiles were extracted from the SAFE distillate as follows: distillate pH was

adjusted to 2 using aqueous 10% (v/v) HCl and extracted with ether ( $3 \times 50$  mL), pH of distillate was then adjusted to 9 using aqueous 2 M NaOH and extracted with ether ( $3 \times 50$  mL). The combined ether extract was concentrated to 50 mL in a water bath ( $43^{\circ}\text{C}$ ) using a Vigreux distillation column and 500-mL round bottom flask. The concentrated ether extract was washed three times with 20 mL of aqueous 0.5 M sodium carbonate ( $\text{Na}_2\text{CO}_3$ ). The upper ether phase was then washed twice with 10 mL of an aqueous saturated NaCl solution and then concentrated to 10 mL under a gentle  $\text{N}_2$  stream. It was then dried over anhydrous sodium sulfate (2 g), and then further concentrated to 200  $\mu\text{L}$  under  $\text{N}_2$ . The remained aqueous phases, containing acidic compounds, were combined and acidified to pH 2 by adding aqueous 4 N HCl. This solution was extracted with ether ( $3 \times 20$  mL) to recover the acidic compounds. The pooled ether extract was concentrated to 200  $\mu\text{L}$  following the procedure described above. Aroma extracts were kept at  $-70^{\circ}\text{C}$  prior to GC-MS analysis.

### Gas chromatography-mass spectrometry (GC-MS)

An Agilent 6890 GC/5973N mass selective detector (MSD) (Agilent Technologies, Palo Alto, CA) was used for the analysis aroma extracts. Separations were performed on a Stabilwax (30 m length  $\times$  0.25 mm i.d.  $\times$  0.25  $\mu\text{m}$  film) column (Restek, Bellefonte, PA) using helium as carrier gas at a constant flow rate of 1.0 mL/min. The extract (1  $\mu\text{L}$ ) was injected in duplicate in a cool on-column inlet ( $+3^{\circ}\text{C}$ , oven tracking mode). GC oven temperature was programmed from 40 to  $225^{\circ}\text{C}$  at a rate of  $4^{\circ}\text{C}/\text{min}$  with initial and final hold times of 5 min and 20 min, respectively. The MSD conditions were as follows: capillary direct interface temperature,  $280^{\circ}\text{C}$ ; ionization energy, 70 eV; mass range, 35–300 amu; EM voltage, stune+200 V; scan rate, 5 sans/s.

Compounds were identified by matching retention indices (RI) (15) and mass spectra of peaks with those of authentic standards. Tentative identifications were based on standard MS library data (NIST 0.5) (Agilent Technologies, Santa Clara, CA).

The relative concentrations of each compound were expressed by the ratio of their total ion peak area to that of internal standard (IS), 2-methyl-3-heptanone or 2-ethylbutyric acid. Relative concentrations of co-eluting compounds were calculated using the ratio of their characteristic ion peak area to that of IS.

### Statistical analysis

All statistical analyses including analysis of variance (ANOVA) and Turkey's multiple-range test were conducted using SPSS statistical program (Ver. 10.0, SPSS

Inc., Chicago, IL) at the 95% significance level.

## RESULTS AND DISCUSSION

### Volatiles in fresh soymilk

A total of 131 volatile compounds (106 positive and 25 tentative) were identified by SAFE-GC-MS in the fresh commercial soymilk (FSM) (Table 1). SAFE allows for more careful and efficient isolation of soymilk volatiles compared with other extraction methods, such as vacuum distillation, simultaneous distillation and extraction or solid-phase microextraction (SPME) for which less than 60 volatiles were previously reported (16-18). In the present study 99 volatile compounds were found in the neutral/basic fraction, 24 in the acidic fraction and eight compounds were partitioned into both fractions. The FSM volatiles were grouped into eight chemical classes, including 21 acids, 18 alcohols, 16 nitrogen-containing compounds, 12 furans and furan derivatives, 12 aldehydes, 10 sulfur-containing compounds, six phenolic compounds and 26 miscellaneous compounds.

Acids were found in high abundance in FSM. In particular, 2-ethylhexanoic, hexanoic, acetic and 3-methylbutanoic acids comprised over 82% of the total level of acids in FSM. Lozano et al. (14) reported that volatile straight-chain organic acids such as acetic, propanoic, butanoic, hexanoic and octanoic acids were responsible for vinegar-like, sweaty and cheesy odor notes in UHT-processed soymilk.

Alcohols were the second most abundant volatile constituents in FSM. Hexanol, benzenemethanol and 3-methyl-1-butanol were the major alcohols in FSM. Alcohols are generally considered as only minor contributors to food aroma because of their high odor detection thresholds (19). One notable exception is 1-octen-3-ol which has a characteristic mushroom-like odor note and an extremely low odor detection threshold of 1 ng/g (20). Toya et al. (21) reported 1-octen-3-ol as one of the most important aroma components of canned snap bean, and Lozano et al. (14) indicated 1-octen-3-ol as well as 1-penten-3-ol as odor-active alcohols in UHT-processed soymilks. In addition, Kobayashi et al. (17) and Wilkens and Lin (18) suggested that some aliphatic alcohols such as pentanol, hexanol and heptanol might also influence the intensity of the beany flavor in soymilk.

Nitrogen-containing volatiles (N-compounds) of FSM consisted of 12 pyrazines, two pyridines and two pyrroles. These compounds have not been previously reported in the other type of non-heated soy products such as soybean oil, soy protein isolates and soy flour (22). N-compounds are most likely produced in FSM during heat processing, such as during the lipoyxygenase inactivation

step or sterilization. Pyrazines and pyrroles are major volatiles formed via the Maillard reaction, and 2-alkylpyridines have been proposed to be derived from the thermal reaction of unsaturated aldehydes with ammonia (23). Among the pyrazines identified in the present study, 3-ethyl-2,5-dimethylpyrazine and 2,3-diethyl-5-methylpyrazine, which have low odor detection threshold values of 8.6 ng/g and 0.09 ng/g, respectively (20), could contribute potato-like odor notes to FSM.

Furans and furan derivatives, including furfurals and furanones, are some other well-known compounds produced by the Maillard reaction or by lipid oxidation. These compounds are mainly associated with sweet, fruity, nutty or caramel-like notes in foods (24). In the present study, 2-furanmethanol was the most abundant furan derivative in FSM, followed by 2-pentylfuran and 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (HDMF). 2-Furanmethanol, which exhibited burnt meat-like and vitamin-like odor in meat products (25), could be formed via various formation pathways such as Maillard reaction or by the deamination and dehydration of Amadori products during heating (26). 2-Pentylfuran, which is reported to be responsible for beany/grassy odor in soy products (18), was found in FSM at a concentration exceeding its odor detection threshold of 6 ng/g (20). Therefore, 2-pentylfuran accompanied by other aldehydes (mainly hexanal) may contribute to the green/beany odor of FSM. HDMF, which has a burnt sugar/caramel-like odor, was reported to be generated by the thermal treatment of sugars (27) and has been reported in heat-processed foods as well as in some dairy products (27,28).

Aldehydes and ketones are representative volatile products from the oxidation and thermal decomposition of lipids (29). Although 11 ketones were detected in FSM, their roles in the aroma of FSM may be minimal since they were found at sub-threshold concentrations. For instance the most abundant ketones in FSM, namely 2-heptanone, 2-butanone, and 2-octanone, were present at levels below their published odor detection thresholds of 140 ng/g, 50,000 ng/g, and 50 ng/g, respectively (20). Moreover, the ketones found in FSM (except for 2-heptanone) were not previously reported as odor-active compounds in fresh and UHT-processed soymilks (14).

Benzaldehyde, a Strecker degradation product from phenylalanine, was the most abundant aldehyde, followed by 2-methyl-(*E*)-2-pentenal and hexanal. The green/beany odor of soymilk has been an impediment to the widespread acceptance of soymilk in Western markets and many attempts have been made to reduce this off note in soymilk (30). Hexanal has been implicated

**Table 1.** Volatile compounds in the commercial aseptic Korean soymilk during refrigerated (4°C) and thermal (55°C) storage

Compound	RI <sup>1)</sup>	FR/IS <sup>2)</sup>	Concentration (ng/g) <sup>3)</sup>						
			FSM <sup>4)</sup>	RSM (4°C)			TSM (55°C)		
				10D	20D	30D	10D	20D	30D
<b>Aldehydes (12)</b>									
2-Methylbutanal	916	NB/IS-1	3.9 <sup>a</sup>	3.3 <sup>a</sup>	4.2 <sup>a</sup>	3.7 <sup>a</sup>	7.1 <sup>b</sup>	9.8 <sup>c</sup>	10.9 <sup>c</sup>
3-Methylbutanal	919	NB/IS-1	3.0 <sup>a</sup>	3.6 <sup>a</sup>	4.1 <sup>a</sup>	4.4 <sup>a</sup>	3.5 <sup>a</sup>	12.8 <sup>b</sup>	13.1 <sup>b</sup>
Pentanal	978	NB/IS-1	1.5 <sup>a</sup>	1.6 <sup>a</sup>	1.6 <sup>a</sup>	1.7 <sup>a</sup>	2.7 <sup>ab</sup>	3.6 <sup>b</sup>	3.3 <sup>b</sup>
Hexanal	1073	NB/IS-1	12.7 <sup>a</sup>	12.8 <sup>a</sup>	9.9 <sup>a</sup>	11.0 <sup>a</sup>	17.4 <sup>b</sup>	17.6 <sup>b</sup>	20.5 <sup>c</sup>
2-Methyl-2-butenal (isomer)*	1086	NB/IS-1	0.6 <sup>a</sup>	1.2 <sup>b</sup>	1.0 <sup>b</sup>	1.3 <sup>bc</sup>	1.1 <sup>b</sup>	1.6 <sup>c</sup>	2.4 <sup>d</sup>
2-Methyl-2-pentenal (isomer)*	1151	NB/IS-1	13.1 <sup>a</sup>	24.6 <sup>ab</sup>	31.3 <sup>bcd</sup>	41.3 <sup>cd</sup>	25.1 <sup>abc</sup>	45.9 <sup>d</sup>	47.5 <sup>d</sup>
Heptanal	1181	NB/IS-1	0.9 <sup>bc</sup>	1.1 <sup>cd</sup>	0.5 <sup>ab</sup>	0.3 <sup>a</sup>	1.3 <sup>cd</sup>	1.5 <sup>de</sup>	1.8 <sup>e</sup>
Octanal	1284	NB/IS-1	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	2.0 <sup>b</sup>	2.1 <sup>b</sup>	2.8 <sup>b</sup>
Nonanal	1387	NB/IS-1	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.1 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>
(E)-2-Octenal	1420	NB/IS-1	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.9 <sup>b</sup>	1.8 <sup>c</sup>	1.8 <sup>c</sup>
Benzaldehyde	1510	NB/IS-1	51.2 <sup>ab</sup>	49.4 <sup>a</sup>	57.5 <sup>bc</sup>	60.3 <sup>cd</sup>	65.4 <sup>de</sup>	68.2 <sup>e</sup>	72.7 <sup>e</sup>
(E,E)-2,4-Decadienal	1797	NB/IS-1	0.8 <sup>a</sup>	1.2 <sup>ab</sup>	0.3 <sup>a</sup>	0.9 <sup>ab</sup>	1.9 <sup>b</sup>	3.0 <sup>c</sup>	3.0 <sup>c</sup>
<b>Ketones (12)</b>									
2-Butanone	908	NB/IS-1	4.3 <sup>a</sup>	16.9 <sup>b</sup>	19.7 <sup>b</sup>	19.2 <sup>b</sup>	15.1 <sup>b</sup>	19.7 <sup>b</sup>	25.9 <sup>c</sup>
2-Pentanone	976	NB/IS-1	1.5 <sup>ab</sup>	1.2 <sup>a</sup>	0.8 <sup>a</sup>	1.5 <sup>ab</sup>	1.2 <sup>a</sup>	2.3 <sup>b</sup>	2.2 <sup>b</sup>
2-Hexanone	1071	NB/IS-1	0.4 <sup>a</sup>	0.4 <sup>a</sup>	0.7 <sup>ab</sup>	0.8 <sup>ab</sup>	1.5 <sup>bc</sup>	2.1 <sup>c</sup>	2.3 <sup>c</sup>
(E)-3-Penten-2-one	1118	NB/IS-1	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	1.5 <sup>b</sup>	1.8 <sup>c</sup>	1.6 <sup>bc</sup>
2-Heptanone	1178	NB/IS-1	8.9 <sup>a</sup>	9.7 <sup>a</sup>	10.4 <sup>a</sup>	9.0 <sup>a</sup>	14.0 <sup>b</sup>	18.0 <sup>c</sup>	19.8 <sup>d</sup>
2-Octanone	1279	NB/IS-1	4.0 <sup>a</sup>	3.8 <sup>a</sup>	2.4 <sup>a</sup>	3.6 <sup>a</sup>	6.2 <sup>b</sup>	7.7 <sup>b</sup>	11.4 <sup>c</sup>
6-Methyl-5-hepten-2-one (isomer)*	1328	NB/IS-1	0.6 <sup>ab</sup>	1.2 <sup>b</sup>	1.4 <sup>b</sup>	2.7 <sup>c</sup>	3.4 <sup>cd</sup>	4.1 <sup>d</sup>	3.3 <sup>c</sup>
2-Nonanone	1382	NB/IS-1	0.3 <sup>a</sup>	0.4 <sup>a</sup>	0.4 <sup>a</sup>	0.4 <sup>a</sup>	0.5 <sup>a</sup>	0.6 <sup>b</sup>	1.0 <sup>c</sup>
3-Octen-2-one (isomer)*	1394	NB/IS-1	0.3 <sup>a</sup>	0.2 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	2.0 <sup>b</sup>	2.9 <sup>c</sup>	2.7 <sup>bc</sup>
3-Nonen-2-one (isomer)*	1497	NB/IS-1	— <sup>5)a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	0.5 <sup>b</sup>	0.8 <sup>b</sup>	5.4 <sup>c</sup>
3,5-Octadien-2-one (isomer)*	1560	NB/IS-1	1.5 <sup>a</sup>	2.6 <sup>bc</sup>	3.0 <sup>c</sup>	3.0 <sup>c</sup>	1.8 <sup>ab</sup>	2.0 <sup>ab</sup>	1.4 <sup>a</sup>
6-Methyl-3,5-heptadien-2-one (isomer)*	1586	NB/IS-1	0.3 <sup>a</sup>	0.6 <sup>ab</sup>	0.4 <sup>a</sup>	0.5 <sup>ab</sup>	0.3 <sup>a</sup>	0.5 <sup>ab</sup>	1.0 <sup>b</sup>
<b>Alcohols (18)</b>									
Ethanol	934	NB/IS-1	2.7 <sup>c</sup>	0.9 <sup>a</sup>	0.9 <sup>a</sup>	0.6 <sup>a</sup>	2.1 <sup>b</sup>	2.6 <sup>c</sup>	2.8 <sup>c</sup>
2-Butanol	1027	NB/IS-1	0.8 <sup>b</sup>	0.5 <sup>a</sup>	0.5 <sup>a</sup>	0.5 <sup>a</sup>	0.8 <sup>b</sup>	1.0 <sup>c</sup>	0.6 <sup>a</sup>
2-Methyl-1-propanol	1090	NB/IS-1	1.1 <sup>a</sup>	1.3 <sup>a</sup>	1.8 <sup>a</sup>	1.2 <sup>a</sup>	7.3 <sup>b</sup>	7.4 <sup>b</sup>	9.8 <sup>c</sup>
3-Pentanol	1104	NB/IS-1	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.2 <sup>a</sup>	0.7 <sup>b</sup>	0.6 <sup>b</sup>
1-Butanol	1142	NB/IS-1	7.0 <sup>ab</sup>	6.0 <sup>a</sup>	6.7 <sup>a</sup>	7.4 <sup>ab</sup>	7.4 <sup>ab</sup>	9.1 <sup>b</sup>	7.6 <sup>ab</sup>
1-Penten-3-ol	1158	NB/IS-1	3.9 <sup>a</sup>	4.8 <sup>a</sup>	4.5 <sup>a</sup>	4.7 <sup>a</sup>	7.9 <sup>b</sup>	9.0 <sup>b</sup>	10.2 <sup>b</sup>
3-Methyl-1-butanol	1205	NB/IS-1	30.8 <sup>a</sup>	35.1 <sup>a</sup>	32.0 <sup>a</sup>	32.2 <sup>a</sup>	64.3 <sup>b</sup>	63.5 <sup>b</sup>	65.1 <sup>b</sup>
1-Pentanol	1248	NB/IS-1	25.4 <sup>b</sup>	24.9 <sup>b</sup>	18.7 <sup>a</sup>	17.0 <sup>a</sup>	30.5 <sup>c</sup>	35.3 <sup>d</sup>	33.5 <sup>cd</sup>
1-Hexanol	1350	NB/IS-1	55.1 <sup>a</sup>	58.7 <sup>ab</sup>	63.6 <sup>b</sup>	59.7 <sup>ab</sup>	60.6 <sup>ab</sup>	63.8 <sup>b</sup>	58.4 <sup>ab</sup>
3-Octanol*	1390	NB/IS-1	3.7 <sup>ab</sup>	3.8 <sup>ab</sup>	3.3 <sup>a</sup>	4.1 <sup>ab</sup>	6.0 <sup>b</sup>	14.9 <sup>c</sup>	16.7 <sup>c</sup>
2-Octanol*	1416	NB/IS-1	1.1 <sup>b</sup>	<0.1 <sup>a</sup>	0.2 <sup>a</sup>	0.3 <sup>a</sup>	1.3 <sup>bc</sup>	1.5 <sup>cd</sup>	1.7 <sup>d</sup>
1-Octen-3-ol	1447	NB/IS-1	29.8 <sup>a</sup>	33.8 <sup>ab</sup>	36.9 <sup>b</sup>	35.1 <sup>ab</sup>	35.2 <sup>ab</sup>	46.0 <sup>d</sup>	42.1 <sup>c</sup>
1-Heptanol	1450	NB/IS-1	5.3 <sup>b</sup>	0.8 <sup>a</sup>	1.2 <sup>a</sup>	1.0 <sup>a</sup>	5.7 <sup>b</sup>	4.8 <sup>b</sup>	6.1 <sup>b</sup>
1-Octanol	1552	NB/IS-1	5.7 <sup>cd</sup>	0.4 <sup>a</sup>	1.2 <sup>b</sup>	1.0 <sup>ab</sup>	5.4 <sup>c</sup>	6.3 <sup>d</sup>	6.1 <sup>d</sup>
Benzenemethanol	1864	BF/IS-B	48.2 <sup>a</sup>	49.2 <sup>a</sup>	53.8 <sup>ab</sup>	53.1 <sup>ab</sup>	53.8 <sup>ab</sup>	54.0 <sup>ab</sup>	56.8 <sup>b</sup>
2-Phenylethanol	1897	NB/IS-1	38.8 <sup>a</sup>	37.6 <sup>a</sup>	35.7 <sup>a</sup>	36.8 <sup>a</sup>	43.1 <sup>b</sup>	45.4 <sup>b</sup>	44.2 <sup>b</sup>
1-Dodecanol	1965	NB/IS-1	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	5.0 <sup>b</sup>	6.8 <sup>c</sup>	7.6 <sup>d</sup>
1-Tetradecanol	2162	NB/IS-1	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	2.4 <sup>b</sup>	2.4 <sup>b</sup>	1.9 <sup>b</sup>
<b>Acids (21)</b>									
Acetic acid	1451	AC/IS-2	174.7 <sup>a</sup>	168.2 <sup>a</sup>	230.6 <sup>a</sup>	217.9 <sup>a</sup>	271.3 <sup>ab</sup>	368.1 <sup>b</sup>	670.1 <sup>c</sup>
Propanoic acid	1533	AC/IS-2	19.6 <sup>a</sup>	23.3 <sup>a</sup>	23.3 <sup>a</sup>	20.8 <sup>a</sup>	24.8 <sup>a</sup>	27.4 <sup>a</sup>	60.6 <sup>b</sup>
2-Methylpropanoic acid	1562	AC/IS-2	25.3 <sup>a</sup>	28.6 <sup>abc</sup>	26.1 <sup>a</sup>	27.4 <sup>ab</sup>	33.7 <sup>bc</sup>	35.5 <sup>c</sup>	51.8 <sup>d</sup>
2,2-Dimethylpropanoic acid*	1572	AC/IS-2	29.1 <sup>c</sup>	21.9 <sup>b</sup>	7.9 <sup>a</sup>	5.9 <sup>a</sup>	28.3 <sup>c</sup>	6.9 <sup>a</sup>	5.4 <sup>a</sup>
Butanoic acid	1621	AC/IS-2	37.0 <sup>ab</sup>	34.0 <sup>a</sup>	32.7 <sup>a</sup>	31.6 <sup>a</sup>	38.4 <sup>ab</sup>	41.7 <sup>b</sup>	43.6 <sup>b</sup>
2-Propenoic acid (isomer)*	1631	AC/IS-2	1.5 <sup>a</sup>	1.7 <sup>a</sup>	1.3 <sup>a</sup>	1.3 <sup>a</sup>	2.1 <sup>a</sup>	2.0 <sup>a</sup>	3.7 <sup>b</sup>
3-Methylbutanoic acid	1666	AC/IS-2	170.0 <sup>a</sup>	160.1 <sup>a</sup>	158.2 <sup>a</sup>	169.6 <sup>a</sup>	216.5 <sup>b</sup>	229.7 <sup>b</sup>	261.6 <sup>c</sup>
2-Methyl-2-propenoic acid (isomer)*	1682	AC/IS-2	5.1 <sup>a</sup>	5.5 <sup>a</sup>	5.6 <sup>a</sup>	6.4 <sup>a</sup>	6.0 <sup>a</sup>	5.2 <sup>a</sup>	8.9 <sup>b</sup>
Pentanoic acid	1731	AC/IS-2	48.6 <sup>a</sup>	44.0 <sup>a</sup>	44.8 <sup>a</sup>	46.1 <sup>a</sup>	48.5 <sup>a</sup>	47.1 <sup>a</sup>	56.3 <sup>b</sup>

**Table 1.** Continued

Compound	RI <sup>1)</sup>	FR/IS <sup>2)</sup>	Concentration (ng/g) <sup>3)</sup>						
			FSM <sup>4)</sup>	RSM (4°C)			TSM (55°C)		
				10D	20D	30D	10D	20D	30D
<b>Acids (21)</b>									
3-Methyl-2-butenic acid (isomer)*	1789	AC/IS-2	4.9 <sup>a</sup>	4.8 <sup>a</sup>	5.0 <sup>a</sup>	5.2 <sup>a</sup>	5.4 <sup>ab</sup>	5.0 <sup>a</sup>	6.3 <sup>b</sup>
4-Methylpentanoic acid (isomer)*	1796	AC/IS-2	7.5 <sup>ab</sup>	7.7 <sup>ab</sup>	6.6 <sup>a</sup>	8.1 <sup>ab</sup>	8.5 <sup>b</sup>	9.8 <sup>c</sup>	10.1 <sup>c</sup>
3-Methyl-3-butenic acid (isomer)*	1806	AC/IS-2	1.4 <sup>a</sup>	2.4 <sup>a</sup>	1.4 <sup>a</sup>	2.1 <sup>a</sup>	2.3 <sup>a</sup>	2.6 <sup>a</sup>	4.8 <sup>b</sup>
2-Ethyl-2-methylbutanoic acid*	1823	AC/IS-2	5.4 <sup>b</sup>	5.1 <sup>b</sup>	7.5 <sup>c</sup>	8.4 <sup>d</sup>	4.9 <sup>ab</sup>	4.2 <sup>a</sup>	4.9 <sup>ab</sup>
Hexanoic acid	1839	AC/IS-2	284.1 <sup>a</sup>	295.6 <sup>a</sup>	304.0 <sup>ab</sup>	304.8 <sup>ab</sup>	312.3 <sup>ab</sup>	313.8 <sup>ab</sup>	334.5 <sup>b</sup>
2-Ethylhexanoic acid	1949	AC/IS-2	1,007.6 <sup>ab</sup>	1,054.5 <sup>bc</sup>	1,092.1 <sup>bcd</sup>	1,116.2 <sup>cd</sup>	1,186.7 <sup>d</sup>	1,095.8 <sup>bcd</sup>	938.0 <sup>a</sup>
Octanoic acid	2052	AC/IS-2	44.1 <sup>abc</sup>	39.9 <sup>a</sup>	45.0 <sup>abc</sup>	46.2 <sup>bcd</sup>	41.8 <sup>ab</sup>	49.9 <sup>cd</sup>	51.0 <sup>d</sup>
Nonanoic acid	2158	AC/IS-2	21.9 <sup>d</sup>	14.0 <sup>a</sup>	16.5 <sup>c</sup>	15.4 <sup>bc</sup>	14.7 <sup>ab</sup>	14.3 <sup>ab</sup>	14.5 <sup>ab</sup>
Decanoic acid	2264	AC/IS-2	14.3 <sup>c</sup>	7.3 <sup>ab</sup>	7.1 <sup>ab</sup>	6.0 <sup>a</sup>	9.4 <sup>b</sup>	6.4 <sup>a</sup>	6.3 <sup>a</sup>
Undecanoic acid	2370	AC/IS-2	3.8 <sup>c</sup>	2.7 <sup>bc</sup>	2.0 <sup>ab</sup>	2.0 <sup>ab</sup>	2.5 <sup>bc</sup>	2.8 <sup>bc</sup>	1.1 <sup>a</sup>
Benzoic acid	2413	AC/IS-2	50.8 <sup>bc</sup>	43.3 <sup>ab</sup>	31.5 <sup>a</sup>	27.6 <sup>a</sup>	55.7 <sup>bc</sup>	50.7 <sup>bc</sup>	59.6 <sup>c</sup>
Dodecanoic acid	2475	AC/IS-2	23.8 <sup>c</sup>	20.0 <sup>bc</sup>	18.6 <sup>bc</sup>	15.2 <sup>ab</sup>	13.9 <sup>ab</sup>	13.9 <sup>ab</sup>	9.0 <sup>a</sup>
<b>Nitrogen-containing compounds (17)</b>									
Pyrazine	1205	NB/IS-1	0.4 <sup>ab</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.5 <sup>b</sup>	0.8 <sup>c</sup>	0.8 <sup>c</sup>
2-Methylpyrazine	1257	NB/IS-1	10.3 <sup>a</sup>	12.7 <sup>a</sup>	15.8 <sup>ab</sup>	17.0 <sup>b</sup>	16.8 <sup>b</sup>	27.3 <sup>c</sup>	32.9 <sup>d</sup>
2,5-Dimethylpyrazine	1314	NB/IS-1	312.4 <sup>a</sup>	329.4 <sup>a</sup>	327.2 <sup>a</sup>	340.9 <sup>a</sup>	408.2 <sup>b</sup>	437.5 <sup>bc</sup>	467.4 <sup>c</sup>
2,6-Dimethylpyrazine	1319	NB/IS-1	114.7 <sup>a</sup>	105.3 <sup>a</sup>	122.6 <sup>a</sup>	120.4 <sup>a</sup>	172.0 <sup>b</sup>	220.6 <sup>c</sup>	227.1 <sup>c</sup>
Ethylpyrazine	1320	NB/IS-1	0.3 <sup>a</sup>	0.4 <sup>a</sup>	0.3 <sup>a</sup>	0.5 <sup>a</sup>	5.7 <sup>b</sup>	7.1 <sup>c</sup>	7.8 <sup>c</sup>
2,3-Dimethylpyrazine	1331	NB/IS-1	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.2 <sup>a</sup>	3.5 <sup>b</sup>	4.5 <sup>b</sup>	4.3 <sup>b</sup>
2-Ethyl-6-methylpyrazine	1374	NB/IS-1	2.5 <sup>a</sup>	3.1 <sup>a</sup>	3.8 <sup>a</sup>	3.4 <sup>a</sup>	5.2 <sup>b</sup>	9.0 <sup>c</sup>	9.2 <sup>c</sup>
2-Ethyl-5-methylpyrazine	1380	NB/IS-1	11.1 <sup>ab</sup>	10.6 <sup>a</sup>	11.8 <sup>ab</sup>	11.5 <sup>ab</sup>	13.4 <sup>b</sup>	16.5 <sup>c</sup>	17.6 <sup>c</sup>
2-Vinylpyrazine*	1421	NB/IS-1	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.8 <sup>b</sup>
3-Ethyl-2,5-dimethylpyrazine	1434	NB/IS-1	47.7 <sup>a</sup>	49.4 <sup>a</sup>	47.5 <sup>a</sup>	53.0 <sup>ab</sup>	58.5 <sup>b</sup>	73.2 <sup>d</sup>	67.4 <sup>c</sup>
2-Methyl-6-vinylpyrazine	1473	NB/IS-1	1.1 <sup>a</sup>	0.6 <sup>a</sup>	0.6 <sup>a</sup>	0.5 <sup>a</sup>	3.7 <sup>b</sup>	8.6 <sup>c</sup>	10.8 <sup>d</sup>
2-Methyl-5-vinylpyrazine	1479	NB/IS-1	0.2 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.4 <sup>b</sup>	0.5 <sup>bc</sup>	0.6 <sup>c</sup>
2,3-Diethyl-5-methylpyrazine	1502	NB/IS-1	0.3 <sup>a</sup>	0.6 <sup>a</sup>	0.6 <sup>a</sup>	0.6 <sup>a</sup>	4.3 <sup>c</sup>	3.5 <sup>b</sup>	5.9 <sup>d</sup>
2-Acetylpyridine	1589	NB/IS-1	2.4 <sup>a</sup>	2.2 <sup>a</sup>	1.8 <sup>a</sup>	1.9 <sup>a</sup>	3.4 <sup>b</sup>	4.3 <sup>c</sup>	5.7 <sup>d</sup>
2-Acetyl-4-methylpyridine*	1616	NB/IS-1	2.6 <sup>a</sup>	3.2 <sup>a</sup>	2.9 <sup>a</sup>	3.5 <sup>a</sup>	3.7 <sup>a</sup>	4.9 <sup>a</sup>	4.9 <sup>a</sup>
2-Acetylpyrrole	1958	NB/IS-1	2.2 <sup>a</sup>	2.7 <sup>a</sup>	3.0 <sup>a</sup>	2.2 <sup>a</sup>	8.3 <sup>b</sup>	13.6 <sup>c</sup>	13.3 <sup>c</sup>
2-Formylpyrrole*	2001	NB/IS-1	1.6 <sup>a</sup>	1.9 <sup>a</sup>	1.9 <sup>a</sup>	2.0 <sup>a</sup>	2.7 <sup>b</sup>	3.4 <sup>c</sup>	3.8 <sup>c</sup>
<b>Furans and furan derivatives (13)</b>									
2-Methylfuran	878	NB/IS-1	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.3 <sup>a</sup>	0.9 <sup>b</sup>	1.4 <sup>b</sup>	10.4 <sup>c</sup>
2-Ethylfuran	951	NB/IS-1	6.0 <sup>a</sup>	5.8 <sup>a</sup>	7.6 <sup>abc</sup>	6.5 <sup>ab</sup>	7.1 <sup>ab</sup>	9.5 <sup>cd</sup>	11.0 <sup>d</sup>
2-Ethyl-5-methylfuran	1025	NB/IS-1	0.3 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.4 <sup>a</sup>	2.6 <sup>b</sup>	2.6 <sup>b</sup>
2-Vinylfuran*	1067	NB/IS-1	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	0.1 <sup>a</sup>	0.6 <sup>c</sup>
2-Butylfuran	1130	NB/IS-1	0.3 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.4 <sup>a</sup>	0.5 <sup>a</sup>	0.6 <sup>a</sup>
2-Pentylfuran	1228	NB/IS-1	13.9 <sup>a</sup>	12.1 <sup>a</sup>	14.0 <sup>a</sup>	13.1 <sup>a</sup>	12.3 <sup>a</sup>	21.4 <sup>b</sup>	23.4 <sup>b</sup>
Furfural	1458	AC/IS-2	6.1 <sup>ab</sup>	6.6 <sup>b</sup>	6.4 <sup>ab</sup>	5.3 <sup>a</sup>	10.1 <sup>c</sup>	15.7 <sup>d</sup>	16.6 <sup>d</sup>
2-Acetylfuran	1495	BF/IS-B	3.9 <sup>ab</sup>	5.3 <sup>b</sup>	1.9 <sup>a</sup>	2.0 <sup>a</sup>	5.2 <sup>b</sup>	10.1 <sup>c</sup>	9.2 <sup>c</sup>
5-Methylfurfural	1564	NB/IS-1	0.2 <sup>a</sup>	0.2 <sup>ab</sup>	0.2 <sup>ab</sup>	0.2 <sup>ab</sup>	0.5 <sup>b</sup>	0.9 <sup>c</sup>	1.8 <sup>d</sup>
2-Furanmethanol	1655	BF/IS-B	70.0 <sup>a</sup>	72.0 <sup>a</sup>	74.1 <sup>a</sup>	75.5 <sup>a</sup>	183.8 <sup>b</sup>	280.4 <sup>c</sup>	390.7 <sup>d</sup>
3-Furanmethanol	1673	BF/IS-B	0.6 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	1.7 <sup>b</sup>	3.5 <sup>c</sup>	9.7 <sup>d</sup>
5-Ethyl-2(5H)-furanone*	1743	NB/IS-1	1.8 <sup>ab</sup>	2.1 <sup>abc</sup>	1.5 <sup>a</sup>	1.6 <sup>a</sup>	2.1 <sup>abc</sup>	2.6 <sup>bc</sup>	3.0 <sup>c</sup>
4-Hydroxy-2,5-dimethyl-3(2H)-furanone (HDMF)	2022	AC/IS-2	6.1 <sup>a</sup>	6.7 <sup>a</sup>	7.0 <sup>a</sup>	6.8 <sup>a</sup>	13.1 <sup>b</sup>	15.3 <sup>b</sup>	32.8 <sup>c</sup>
<b>Sulfur-containing compounds (12)</b>									
Thiophene	1014	NB/IS-1	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	— <sup>a</sup>	0.2 <sup>b</sup>	1.4 <sup>c</sup>
Dimethyl disulfide	1060	NB/IS-1	0.3 <sup>a</sup>	0.6 <sup>ab</sup>	1.2 <sup>abc</sup>	1.9 <sup>c</sup>	1.5 <sup>bc</sup>	1.9 <sup>c</sup>	1.9 <sup>c</sup>
2-Methyl-1-thiophene	1085	NB/IS-1	0.3 <sup>a</sup>	0.2 <sup>a</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	1.0 <sup>a</sup>	1.3 <sup>a</sup>	3.3 <sup>b</sup>
3-Methyl-1-thiophene	1112	NB/IS-1	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.3 <sup>ab</sup>	0.3 <sup>ab</sup>	0.4 <sup>b</sup>
Thiazole	1238	NB/IS-1	0.1 <sup>a</sup>	0.1 <sup>ab</sup>	0.2 <sup>ab</sup>	0.2 <sup>ab</sup>	0.1 <sup>ab</sup>	0.2 <sup>b</sup>	0.7 <sup>c</sup>
Dimethyl trisulfide	1365	NB/IS-1	0.2 <sup>a</sup>	0.1 <sup>a</sup>	0.2 <sup>a</sup>	0.2 <sup>a</sup>	1.0 <sup>a</sup>	2.1 <sup>b</sup>	2.3 <sup>b</sup>
Dihydro-3(2H)-thiophenone*	1541	NB/IS-1	0.2 <sup>a</sup>	0.3 <sup>ab</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.5 <sup>b</sup>	24.5 <sup>c</sup>	31.5 <sup>d</sup>

**Table 1.** Continued

Compound	RI <sup>1)</sup>	FR/IS <sup>2)</sup>	Concentration (ng/g) <sup>3)</sup>						
			FSM <sup>4)</sup>	RSM (4°C)			TSM (55°C)		
				10D	20D	30D	10D	20D	30D
Sulfur-containing compounds (12)									
4-Methylthiazole	1624	NB/IS-1	2.6 <sup>a</sup>	3.2 <sup>a</sup>	2.8 <sup>a</sup>	2.7 <sup>a</sup>	4.4 <sup>b</sup>	5.3 <sup>c</sup>	6.4 <sup>d</sup>
2-Acetylthiazole	1634	NB/IS-1	1.5 <sup>a</sup>	1.6 <sup>ab</sup>	1.6 <sup>a</sup>	1.5 <sup>a</sup>	2.1 <sup>bc</sup>	2.4 <sup>c</sup>	2.5 <sup>c</sup>
2-Acetyl-4-methylthiazole <sup>*</sup>	1681	NB/IS-1	4.4 <sup>a</sup>	4.7 <sup>ab</sup>	4.9 <sup>b</sup>	4.7 <sup>ab</sup>	5.1 <sup>b</sup>	5.1 <sup>b</sup>	4.8 <sup>ab</sup>
3-(Methylthio)-1-propanol	1706	NB/IS-1	4.4 <sup>c</sup>	0.4 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	3.8 <sup>c</sup>	2.8 <sup>b</sup>	3.9 <sup>c</sup>
2-Hydroxymethylthiophene <sup>*</sup>	1924	NB/IS-1	4.4 <sup>a</sup>	9.1 <sup>c</sup>	15.4 <sup>de</sup>	14.1 <sup>d</sup>	6.4 <sup>b</sup>	16.1 <sup>c</sup>	13.9 <sup>d</sup>
Phenolic compounds (6)									
2-Methoxyphenol (guaiacol)	1847	NB/IS-1	1.5 <sup>a</sup>	1.2 <sup>a</sup>	1.5 <sup>a</sup>	1.5 <sup>a</sup>	2.2 <sup>b</sup>	4.9 <sup>c</sup>	7.6 <sup>d</sup>
Phenol	1994	BF/IS-B	1.5 <sup>a</sup>	2.6 <sup>a</sup>	4.9 <sup>b</sup>	5.0 <sup>b</sup>	2.3 <sup>a</sup>	6.2 <sup>b</sup>	6.2 <sup>b</sup>
Eugenol	2152	NB/IS-1	1.2 <sup>ab</sup>	2.2 <sup>bc</sup>	2.6 <sup>c</sup>	2.1 <sup>bc</sup>	1.4 <sup>a</sup>	2.8 <sup>c</sup>	2.5 <sup>c</sup>
4-Vinyl-2-methoxyphenol (4-vinylguaiacol)	2174	BF/IS-B	2.0 <sup>a</sup>	0.7 <sup>a</sup>	1.1 <sup>a</sup>	0.8 <sup>a</sup>	23.8 <sup>b</sup>	52.6 <sup>c</sup>	75.9 <sup>d</sup>
4-Vinylphenol	2370	BF/IS-B	0.8 <sup>a</sup>	0.8 <sup>a</sup>	0.8 <sup>a</sup>	0.8 <sup>a</sup>	10.9 <sup>b</sup>	22.8 <sup>c</sup>	25.3 <sup>d</sup>
Vanillin	2543	AC/IS-2	12.9 <sup>ab</sup>	12.8 <sup>abc</sup>	13.7 <sup>ab</sup>	12.4 <sup>a</sup>	14.6 <sup>abc</sup>	15.1 <sup>bc</sup>	15.7 <sup>c</sup>
Miscellaneous compounds (26)									
Octane	812	NB/IS-1	0.7 <sup>a</sup>	0.9 <sup>a</sup>	1.4 <sup>ab</sup>	1.3 <sup>ab</sup>	2.1 <sup>ab</sup>	2.7 <sup>ab</sup>	3.6 <sup>b</sup>
2,4-Dimethylpentane <sup>*</sup>	819	NB/IS-1	3.0 <sup>a</sup>	3.4 <sup>a</sup>	3.9 <sup>a</sup>	6.2 <sup>a</sup>	3.9 <sup>a</sup>	3.9 <sup>a</sup>	5.9 <sup>a</sup>
4-Methyloctane <sup>*</sup>	862	NB/IS-1	3.2 <sup>a</sup>	2.8 <sup>a</sup>	3.1 <sup>a</sup>	3.7 <sup>ab</sup>	3.7 <sup>ab</sup>	4.2 <sup>b</sup>	4.6 <sup>b</sup>
Ethyl acetate	899	NB/IS-1	62.5 <sup>ab</sup>	63.0 <sup>ab</sup>	59.8 <sup>a</sup>	65.1 <sup>ab</sup>	59.5 <sup>a</sup>	69.4 <sup>b</sup>	64.2 <sup>ab</sup>
Decane	1002	NB/IS-1	3.7 <sup>a</sup>	3.6 <sup>a</sup>	3.6 <sup>a</sup>	5.1 <sup>ab</sup>	6.3 <sup>ab</sup>	6.2 <sup>ab</sup>	6.8 <sup>b</sup>
Toluene	1034	NB/IS-1	113.8 <sup>b</sup>	36.7 <sup>a</sup>	38.6 <sup>a</sup>	39.2 <sup>a</sup>	103.9 <sup>b</sup>	57.4 <sup>a</sup>	56.8 <sup>a</sup>
Butyl acetate	1065	NB/IS-1	1.7 <sup>c</sup>	0.6 <sup>a</sup>	0.2 <sup>a</sup>	0.5 <sup>a</sup>	1.3 <sup>bc</sup>	0.5 <sup>a</sup>	0.8 <sup>ab</sup>
$\beta$ -Pinene	1097	NB/IS-1	0.4 <sup>ab</sup>	0.3 <sup>a</sup>	0.3 <sup>a</sup>	0.6 <sup>bc</sup>	0.4 <sup>a</sup>	0.6 <sup>c</sup>	0.6 <sup>c</sup>
Ethylbenzene	1117	NB/IS-1	1.0 <sup>a</sup>	1.0 <sup>a</sup>	1.5 <sup>ab</sup>	1.8 <sup>bc</sup>	2.4 <sup>c</sup>	1.7 <sup>abc</sup>	1.7 <sup>abc</sup>
<i>p</i> -Xylene	1127	NB/IS-1	0.6 <sup>b</sup>	0.6 <sup>b</sup>	0.4 <sup>a</sup>	0.4 <sup>a</sup>	1.7 <sup>d</sup>	1.1 <sup>c</sup>	1.0 <sup>c</sup>
<i>m</i> -Xylene	1133	NB/IS-1	1.0 <sup>a</sup>	0.9 <sup>a</sup>	1.1 <sup>ab</sup>	1.2 <sup>abc</sup>	1.5 <sup>bc</sup>	1.6 <sup>c</sup>	1.6 <sup>c</sup>
Butyl propanoate	1139	NB/IS-1	1.1 <sup>ab</sup>	1.0 <sup>a</sup>	1.8 <sup>d</sup>	1.8 <sup>d</sup>	1.2 <sup>ab</sup>	1.5 <sup>c</sup>	1.3 <sup>bc</sup>
<i>o</i> -Xylene	1176	NB/IS-1	0.8 <sup>a</sup>	0.8 <sup>a</sup>	0.9 <sup>ab</sup>	1.2 <sup>ab</sup>	0.8 <sup>a</sup>	1.5 <sup>b</sup>	0.8 <sup>a</sup>
Limonene	1188	NB/IS-1	1.3 <sup>a</sup>	1.1 <sup>a</sup>	1.7 <sup>a</sup>	1.8 <sup>a</sup>	1.4 <sup>a</sup>	1.6 <sup>a</sup>	1.7 <sup>a</sup>
$\beta$ -Phellandrene	1196	NB/IS-1	0.5 <sup>ab</sup>	0.5 <sup>a</sup>	0.4 <sup>a</sup>	0.6 <sup>abc</sup>	0.6 <sup>abc</sup>	0.8 <sup>c</sup>	0.7 <sup>bc</sup>
Butyl butyrate	1215	NB/IS-1	1.4 <sup>ab</sup>	0.8 <sup>ab</sup>	0.8 <sup>ab</sup>	0.2 <sup>a</sup>	1.5 <sup>ab</sup>	1.8 <sup>b</sup>	2.0 <sup>b</sup>
<i>p</i> -Cymene	1261	NB/IS-1	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.1 <sup>a</sup>	0.7 <sup>b</sup>	0.1 <sup>a</sup>	0.6 <sup>b</sup>
Trimethylbenzene (isomer) <sup>*</sup>	1271	NB/IS-1	1.1 <sup>a</sup>	0.6 <sup>a</sup>	1.3 <sup>a</sup>	1.1 <sup>a</sup>	1.0 <sup>a</sup>	1.4 <sup>a</sup>	1.1 <sup>a</sup>
Tridecane	1302	NB/IS-1	2.7 <sup>c</sup>	1.0 <sup>b</sup>	0.1 <sup>ab</sup>	<0.1 <sup>a</sup>	2.9 <sup>c</sup>	3.0 <sup>c</sup>	2.8 <sup>c</sup>
Tetradecane	1402	NB/IS-1	20.4 <sup>abc</sup>	15.7 <sup>a</sup>	19.3 <sup>abc</sup>	22.4 <sup>bc</sup>	17.1 <sup>ab</sup>	16.7 <sup>ab</sup>	23.2 <sup>c</sup>
$\alpha$ -Isophorone <sup>*</sup>	1570	NB/IS-1	2.6 <sup>a</sup>	2.9 <sup>a</sup>	2.7 <sup>a</sup>	2.2 <sup>a</sup>	4.4 <sup>b</sup>	5.1 <sup>b</sup>	6.1 <sup>c</sup>
Hexadecane	1601	NB/IS-1	2.9 <sup>ab</sup>	2.8 <sup>ab</sup>	2.0 <sup>a</sup>	2.3 <sup>a</sup>	2.3 <sup>a</sup>	3.1 <sup>b</sup>	7.6 <sup>c</sup>
$\gamma$ -Hexalactone	1685	NB/IS-1	4.9 <sup>a</sup>	4.8 <sup>a</sup>	6.9 <sup>ab</sup>	7.2 <sup>ab</sup>	5.1 <sup>a</sup>	8.7 <sup>c</sup>	9.0 <sup>c</sup>
Naphthalene	1721	NB/IS-1	4.9 <sup>a</sup>	5.4 <sup>ab</sup>	5.2 <sup>a</sup>	4.9 <sup>a</sup>	6.8 <sup>b</sup>	11.5 <sup>c</sup>	14.1 <sup>d</sup>
3-Hydroxy-2-methyl-4 <i>H</i> -pyran-4-one (maltol)	1957	AC/IS-2	1,060.9 <sup>a</sup>	1,090.9 <sup>a</sup>	1,059.5 <sup>a</sup>	1,036.5 <sup>a</sup>	1,074.0 <sup>a</sup>	1,045.9 <sup>a</sup>	1,050.2 <sup>a</sup>
$\gamma$ -Nonalactone	2011	NB/IS-1	12.5 <sup>a</sup>	15.2 <sup>ab</sup>	15.9 <sup>ab</sup>	16.1 <sup>ab</sup>	16.0 <sup>ab</sup>	21.3 <sup>b</sup>	19.7 <sup>b</sup>

<sup>1)</sup>RI=retention indices on Stabilwax column (30 m length  $\times$  0.25 mm i.d.  $\times$  0.25  $\mu$ m film thickness, Restek, USA).

<sup>2)</sup>FR=organic fraction in which volatiles were detected (AC, acidic fraction; NB, neutral and basic fraction; BF, found in both fractions). IS=internal standard (IS) used for calculating relative concentration; IS-1, 2-methyl-3-heptanone for NB; IS-2, 2-ethylbutyric acid for AC; IS-B, both ISs (combined concentration after calculating each relative concentration using IS-1 (portion in NB) or IS-2 (portion in AC) in case when the compound partitioned into both fractions).

<sup>3)</sup>Mean relative concentration (ng/g) ( $n=4$ ).

<sup>4)</sup>Days of refrigerated (4°C, RSM) and thermal (55°C, TSM) storage (FSM, fresh soymilk; 10D, 10 days; 20D, 20 days; 30D, 30 days). Mean relative concentrations having different letters (a-f) in each row are significantly different ( $p < 0.05$ ).

<sup>5)</sup>Not detected.

\*Compound tentatively identified by MS data only.

as the main contributor to the green/cut-grass odor of soymilk due to its low odor detection threshold (4.5 ng/g) (20,22) and relatively high abundance. However, Ho et al. (31) reported that 2,4-decadienal having fatty note, has the most influence on the flavor of soymilk rather than hexanal. In addition to the above aldehydes, the malty/chocolate smelling compounds, 2-methylbutanal and 3-methylbutanal, as well as pentanal (butter-like), nonanal (fatty, fruity), (*E*)-2-octenal (raw peanut-like) were also reported as odor-active compounds in UHT-processed soymilk (14).

Eleven sulfur-containing volatiles (S-compounds) were identified in FSM, including four thiophenes, four thiazoles and two sulfides (dimethyl disulfide and dimethyl trisulfide), and all were detected at a concentration less than 5 ng/g. Heterocyclic S-compounds, such as thiophenes and thiazoles, have been reported as components of Maillard model systems containing sugars and sulfur-containing amino acids (23). These compounds play important roles in developing desirable meaty or cooked aromas (32). Among the S-compounds detected in FSM, only dimethyl trisulfide, with a cabbage-like odor note, was found at a concentration exceeding its odor detection threshold (0.01 ng/g) (20). Boatright (33) reported that dimethyl trisulfide is an important off-flavor in soymilk, and demonstrated that its intensity in soymilk could be reduced by addition of gallic acid.

Among the six phenolic compounds identified in FSM, guaiacol and 4-vinylguaiacol were previously reported as key aroma compounds in UHT-processed soymilk (14).

Twenty-six miscellaneous compounds were detected in FSM, consisting of eight aromatic hydrocarbons, seven straight hydrocarbons, four esters, four terpenes, two lactones and maltol. Maltol, having a caramel/burnt sugar-like odor note, was the most abundant compound in FSM and along with  $\gamma$ -nonalactone was indicated as odor-active constituent of UHT-processed soymilk (14). In general, aromatic and straight chain hydrocarbons are known to make only a light contribution to the overall flavor of foods (34). The other miscellaneous compounds also were considered not to be important odorants due to their high odor detection thresholds and/or low concentrations in FSM.

#### **Effect of refrigerated and thermal storage on volatile profiles of soymilk**

During 30 days of refrigerated storage (4°C), no significant change was observed for most of the volatile compounds in the soymilk. Compounds that increased ( $p < 0.05$ ) in concentration in refrigerated soymilk (RSM)

included three aldehydes (2-methyl-2-(*E*)-butenal, 2-methyl-2-(*E*)-pentenal, and benzaldehyde), three ketones (2-butanone, 6-methyl-5-hepten-2-one, and 3,5-octadien-2-one), two acids (2-ethyl-2-methylbutanoic and 2-ethylhexanoic acids), and two S-compounds (dimethyl disulfide and 2-hydroxymethylthiophene) (Table 1). On the other hand, numerous compounds decreased ( $p < 0.05$ ) during storage. These included seven alcohols (ethanol, 1-pentanol, 1-heptanol, 1-octanol, 2-butanol, 2-octanol, and 3-(methylthio)-1-propanol), six acids (nonanoic, decanoic, undecanoic and dodecanoic, 2,2-dimethylpropanoic, and benzoic acid), two aromatic hydrocarbons (toluene and *p*-xylene), butyl acetate and tridecane. Lozano et al. (14) indicated that cold storage (4.4°C) for 7 days caused a significant decrease in S-compounds in UHT soymilk, but this trend was not observed in the present study. Even though some compounds significantly increased or declined during refrigerated storage, these may not necessarily affect the overall flavor of the soymilk due to the relative high odor detection thresholds of these compounds. This is in agreement with the findings of Park et al. (35) who reported no difference between fresh and stored soymilks (at 5°C for 45 days) by electronic nose analysis.

Thermal storage had a great impact on the volatile profile of soymilk compared with refrigerated storage (Table 1). Six volatile compounds, namely 2-vinylfuran, 2-vinylpyrazine, thiophene, 3-nonen-2-one, 1-dodecanol, and 1-tetradecanol, were not found in FSM but were formed in thermal stored soymilk (TSM). The first three compounds have been reported to be formed by thermal reaction between glucose and phenylalanine (2-vinylfuran), glucose and lysine (2-vinylpyrazine) or 2,4-decadienal and cysteine (thiophene) (36-38). The other ketone listed (3-nonen-2-one) and the alcohols (1-dodecanol and 1-tetradecanol) could have been produced by lipid oxidation during storage (29). 3-Nonen-2-one was also found in fruits, and was reported to undergo carbon-carbon double reduction to yield the saturated nine-carbon ketone, 2-nonanone (39).

The major compounds of each class of volatiles identified in FSM, such as 2-hexanoic acid, 3-methyl-1-butanol, 2,6-dimethylpyrazine, 2-furanmethanol, benzaldehyde, 2-butanone, 2-hydroxymethylthiophene and maltol were also predominant in TSM. The concentrations of these compounds as well as some other volatiles were significantly affected by the thermal storage.

Among the N-compounds, four pyrazines (ethyl-, 2,3-dimethyl-, 2-methyl-6-vinyl-, and 2,3-diethyl-5-methylpyrazines) and 2-acetylpyrrole increased by over 5 fold in TSM ( $p < 0.05$ ). N-Containing heterocyclic compounds

are generally formed through thermal interactions between reducing sugars and amino acids, and are recognized as important aroma contributors (roasted nut-like odor) to thermal processed foods (38). Pyrazines, nevertheless, were not considered as odor active component in the soymilk in a previous study (14). However, based on the relative levels and odor detection thresholds of the pyrazines detected in the present study, 3-ethyl-2,5-dimethylpyrazine and 2,3-diethyl-5-methylpyrazine were expected to contribute characteristic potato-like notes to the aromas of both FSM and TSM (20). The contribution may be even greater in TSM, because of the increase in these compounds during thermal storage ( $p < 0.05$ ).

The relative concentrations of 2-acetylfuran, furfural, 5-methylfurfural, 2- and 3-furanmethanol and HDMF increased throughout thermal storage (10~30 days) ( $p < 0.05$ ). 2-Methylfuran showed a dramatic increase (over 40-fold higher than FSM) after 30 days of storage. The increase in furans and furan derivatives in TSM might be caused by Maillard reaction and lipid oxidation (mainly 2-alkylfurans) during thermal storage (40). In general, these compounds are characterized by burnt meat-like/caramel-like/burnt sugar-like odors, and are considered as desirable aromas in heat processed foods (41). Especially important may be the increase of furans which have low odor detection thresholds. These included 2-pentylfuran (6 ng/g, odor detection threshold) (20) and HDMF (25 ng/g, odor detection threshold) (20) which could provide green bean-like and bunt sugar-/caramel-like odors, respectively, to the overall flavor of the TSM.

Among the S-compounds derived from the thermal breakdown of S-containing amino acids (40), 2-methyl-1-thiophene, dimethyl trisulfide and dihydro-3(2*H*)-thiophenone were detected at significantly higher (>10-fold) levels in TSM ( $p < 0.05$ ). The increase of dimethyl trisulfide could lead to a cabbage-like odor in TSM. This is supported by the results of Lozano et al. (14) who reported dimethyl trisulfide to be an important odor-active compound in UHT-processed soymilk.

All six phenolic compounds significantly increased during thermal storage. In particular, the marked increases were observed for 4-vinylguaiacol, 4-vinylphenol and guaiacol. Phenolic compounds have been reported to be formed by thermal decarboxylation of specific acids, such as ferulic acid and vanillin acid, which are the precursors of 4-vinylguaiacol and guaiacol, respectively (42,43). In addition, these compounds were reported to contribute to the roasted and smoky odor of UHT-processed soymilk (14). Therefore, guaiacol,

4-vinylguaiacol and 4-vinylphenol which have relatively low odor detection thresholds (3 ng/g, 3 ng/g and 10 ng/g, respectively) (20) may play key roles in developing roasted or cooked odor of TSM.

Besides the volatiles described above, other classes of volatiles associated with the oxidative degradation of unsaturated fatty acids, such as aldehydes, ketones, alcohols and acids (41,44), also increased in TSM. In the case of ketones, the contents of 2-butanone, 2-hexanone, 3-penten-2-one, 6-methyl-5-hepten-2-one and 3-octen-2-one increased by over 5-fold during thermal storage. However, these ketones may not be important contributors to the aroma of TSM, because they were present at levels below their odor detection thresholds (20). Lozano et al. (14) did not indicate any of these ketones as odor-active compounds in UHT-processed soymilk.

Benzaldehyde, 2-methyl-(*E*)-2-pentenal and hexanal were the predominant aldehydes in TSM. As mentioned early, hexanal and (*E,E*)-2,4-decadienal were previously reported as the main contributors to the green/beany odor of soymilk. In addition to these aldehydes, octanal, characterized by a green/orange peel-like odor, also significantly increased during thermal storage, and its concentration in TSM exceeded its odor detection threshold (0.7 ng/g) (20) after 10 days of storage period. Thus, considering their significant increase and low odor detection thresholds, hexanal, octanal and (*E,E*)-2,4-decadienal might contribute to the typical aroma of TSM.

In general alcohols and acids underwent only a slight increase in concentration during thermal storage. Notable exceptions include 2-methyl-1-propanol (9.3-fold increase), acetic acid (3.8-fold increase), propanoic acid (3.1-fold increase), and 3-methyl-(*E*)-3-butenic acid (3.4-fold increase). In a previous study acids were not detected in soymilk stored at 38°C for 12 weeks (45). However, in that study the volatiles were isolated by SPME which is not an effective method for the analysis of low volatility acids. In the present study, some volatile fatty acids (nonanoic, decanoic, undecanoic, and dodecanoic acids) underwent a slight decrease during both thermal and refrigerated storage. Most of acids and alcohols detected in TSM were present at concentrations below their odor detection thresholds (except for 1-octen-3-ol) (20). Therefore, these compounds might be only minor contributors to overall aroma of TSM.

In conclusion, thermal storage (55°C) of commercial aseptic soymilk resulted in greater volatile profile changes than refrigerated storage. Profound changes were observed for volatiles formed by Maillard and thermal degradation reactions and by lipid oxidation/degradation. Especially important were increases in com-

pounds having relatively low odor detection thresholds, such as hexanal, octanal, (*E*)-2-octenal, (*E,E*)-2,4-decadienal, 1-octen-3-ol, 3-ethyl-2,5-dimethylpyrazine, 2,3-diethyl-5-methylpyrazine, 2-pentylfuran, HDMF, dimethyl trisulfide, guaiacol, 4-vinylguaiacol, and 4-vinylphenol. The increase of these compounds may influence the intensities of green/fatty, mushroom-like, sulfur and cooked/nutty aroma notes in TSM. However, by considering only the relative concentrations of the volatiles, this prediction cannot be solidly supported. Therefore, further studies such as accurate quantification of volatiles as well as GC-olfactometry analysis, including aroma extract dilution analysis, are required to clearly identify the key aroma compounds in TSM.

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