JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Effect of Cold Storage and Packaging Material on the Major Aroma Components of Sweet Cream Butter

PATRICIO R. LOZANO,† EVAN R. MIRACLE,‡ ANDREA J. KRAUSE,‡ MARYANNE DRAKE,‡ AND KEITH R. CADWALLADER*,†

Department of Food Science and Human Nutrition, University of Illinois, 1302 W. Pennsylvania Avenue, Urbana, Illinois 61801, and Department of Food Science, Southeast Dairy Foods Research Center, North Carolina State University, Raleigh, North Carolina 27695

The major aroma compounds of commercial sweet cream AA butter quarters were analyzed by GC-olfactometry and GC-MS combined with dynamic headspace analysis (DHA) and solvent-assisted flavor evaporation (SAFE). In addition, the effect of long-term storage (0, 6, and 12 months) and type of wrapping material (wax parchment paper vs foil) on the aroma components and sensory properties of these butters kept under refrigerated (4 °C) and frozen (−20 °C) storage was evaluated. The most intense compounds in the aroma of pasteurized AA butter were butanoic acid, δ-octalactone, δ-decalactone, 1-octen-3-one, 2-acetyl-1-pyrroline, dimethyl trisulfide, and diacetyl. The intensities of lipid oxidation volatiles and methyl ketones increased as a function of storage time. Refrigerated storage caused greater flavor deterioration compared with frozen storage. The intensity and relative abundance of styrene increased as a function of time of storage at refrigeration temperature. Butter kept frozen for 12 months exhibited lower styrene levels and a flavor profile more similar to that of fresh butter compared to butter refrigerated for 12 months. Foil wrapping material performed better than wax parchment paper in preventing styrene migration into butter and in minimizing the formation of lipid oxidation and hydroxyl acid products that contribute to the loss of fresh butter flavor.

KEYWORDS: Butter; flavor; aroma; storage; wrapping material; styrene migration

INTRODUCTION

The aroma constituents of butter and butter oil have been studied for more than six decades using different isolation techniques, and an extensive list of components has been reported (*1*). Most studies done prior to the 1960s were concerned with identifying the volatile constituents of butter (*2, 3*). According to Schieberle and others (*4*) and Maarse and others (*5*), 233 and 287 volatiles have been identified in butter and butter oil, respectively. Nevertheless, a reduction in the number of significant aroma compounds from hundreds to less than 50 was observed with the introduction of threshold values to determine odor–activity values (*1*). Based on this approach, as well as improvements in volatile component recovery and isolation techniques, consistent reports of the primary aroma-active contributors of butter flavor (odor value >1) were observed (*1, 6*). Most common among these compounds are diacetyl, butanoic acid, hexanoic acid, hexanal, acetaldehyde, dimethyl sulfide, δ-octalactone, δ-decalactone, decanoic acid, phenol, *p*-cresol, and skatole. Several attempts to reconstitute the flavor of butter also can be found in the literature. For instance, Urbach (*7–9*) evaluated the aroma-active compounds in butter oil and compared them against their sensory detection thresholds and odor–activity values in order to reconstitute butter odor in deodorized butter oil. However, further research into the intensity of each of the identified compounds was suggested. The use of gas chromatography–olfactometry and aroma extract dilution analysis helped solve this problem by allowing determination of the character impact compounds of butter (*1*).

The effect of storage on the aroma constituents of butter was later studied in order to understand the relation of these volatile compounds with the shelf life of butter. Widder and others (*10, 11*) identified the potent odorants in fresh butter oil and butter oil stored for 42 days at room temperature. Christensen and Holmer (*12*) studied the effect of short storage time (14 weeks) on the volatile components of butter catering sticks (10 g of butter in an extruded package of polyethylene) at two storage temperatures (−18.4 and 20 °C). Abdel-Mageed and Fadel (*13*) studied the effect of frozen storage (−18 °C) temperatures on the volatile components of butter over a long period of time (7.5 months). However, none of these studies compared the effect of refrigeration storage vs frozen storage on the aroma components of butter. Furthermore, descriptive sensory analysis has not been applied in conjunction with instrumental analyses to establish the impact of storage-related changes on specific flavors. The aim of the present study was to characterize the major aroma components of fresh sweet cream butter at 0, 6, and 12 months

* Corresponding author: telephone 217-333-5803; fax 217-333-1875; e-mail cadwlldr@uiuc.edu.
† University of Illinois.
‡ North Carolina State University.

10.1021/jf071075q CCC: $37.00
Published on Web 08/17/2007

Table 1. Sensory Terms for the Descriptive Sensory Analysis of Butter[a]

term	definition	reference[b]
diacetyl/cultured	sweet aromatic characteristic of cultured dairy products, of which diacetyl is a primary source	diacetyl, 20 ppm
milkfat/lactone	aromatic characteristic of milkfat, lactones, and coconut	heavy cream
cooked/nutty	aromatic associated with cooked milk and canned corn	1% fat milk heated in a microwave for 8 min, freshly churned (<48 h) pasteurized sweet cream butter
refrigerator/stale	stale aromatic characteristic of refrigerator with old food left in it	butter quarters (sticks) stored in a refrigerator for 18 months
painty	aromatics associated with wall paint and oxidized fats	linseed oil
salty	taste elicited by NaCl	sodium chloride solutions; 0.5% [5], 0.7% [8], 0.9% [11.5]

[a] Adapted from Krause et al. (*16*). [b] Numbers in brackets represent consensus scores for reference material.

after processing as well as to understand the effect of two wrapping materials (wax parchment paper and foil) on butter shelf life. The use of freezing temperature as well as refrigeration temperature for long periods of storage (12 months) was also compared. Descriptive sensory analysis was applied in conjunction with instrumental aroma analysis to interpret the impact of volatile changes on flavor.

MATERIALS AND METHODS

Butters. Commercially packaged pasteurized AA salted butter quarters produced in February 2005 were used in this study. Products (25 kg) were obtained directly from two production facilities (CA, USA) and received within 24 h of production packed on ice gel packs via overnight carrier. Upon receipt, products were removed from shipping containers, examined for damage, and then assigned to refrigerated (4 ± 1 °C) or frozen storage (−20 ± 1 °C) in their commercial packages as would be done by industry. The commercial samples represented duplicate production dates from the two facilities (facility 1 and facility 2) and the two common commercial wrapping materials used for butter quarters: foil (foil) and wax parchment paper (wax). At specific time points of 0, 6, and 12 months at 4 °C or 12 months at −20 °C, samples were removed for sensory and instrumental volatile analyses.

Chemicals. All solvents, internal standards and reference compounds were purchased from the Sigma-Aldrich Co. (St. Louis, MO), except for 2-acetyl-1-pyrroline which was a gift from Dr. R. Buttery (USDA, ARS, WRRC, Albany, CA); β-damascenone was provided by Firmenich Co. (Princeton, NJ), δ-octalactone, γ-nonalactone, δ-decalactone, δ-undecalactone, and δ-dodecalactone were purchased from Bedoukian Research (Danbury, CT), and (*Z*)-2-nonenal was synthesized from (*Z*)-2-nonen-1-ol (Bedoukian Research Inc., Danbury, CT) by oxidation with Dess-Martin periodinane (0.3 M in dichloromethane; Aldrich Chemical, Co.) following the procedure described by Meyer and Schreiber (*14*). Odorless deionized–distilled water was prepared by boiling glass-distilled water in an open flask until its volume was reduced by one-third of the original volume.

Descriptive Sensory Analysis. Flavor attributes (aromatics) of butter were evaluated retronasally at each sampling time point (**Table 1**). Eight panelists (7 females, 1 male) were selected on the basis of availability and previous experience (>75 h each) with descriptive sensory analysis of dairy products using the Spectrum method (*15*). Panelists received an additional 25 h of training to focus on identification and scaling of butter flavor attributes. During training, panelists discussed and evaluated an array of commercial butters. The Spectrum universal scale was used to scale the intensity of flavor attributes (*15*) using the language described by Krause et al. (*16*) (**Table 1**). Prior to testing, analysis of variance of panel and panelist performance on selected butters was used to determine that panelists could consistently identify and scale butter color, flavor, and texture attributes. Two weeks prior to each testing time point, panelists received an additional 3 h of refresher training and calibration, and panel and panelist performances on butter sensory attributes were once again confirmed to be consistent.

For sensory analysis, samples (7 g) were prepared with the overhead lights turned off to prevent light-induced flavor changes and placed in 2 oz. Soufflé cups (Sweetheart Cup Co. Inc., Owings, MD) and stored at 5 °C in the dark. One and a half hours before the panel session they were tempered to 19 °C. This temperature was chosen for sensory analysis since panel training sessions indicated that panelists could best identify subtle variations in butter flavor at this temperature. Panelists individually evaluated samples under white lights using paper ballots or computerized data entry (Compusense 5 v4.6, Compusense, Guelph, Canada) in individual booths in a positive air pressure room dedicated to sensory analysis. Each treatment was evaluated in duplicate by each panelist. For flavor evaluations, two warm-up samples, butters that had previously been profiled by the panel, were provided with their consensus flavor profiles along with salty taste solution references (**Table 1**). Panelists were given odorless deionized–distilled water and unsalted saltine crackers between samples for palate cleansing. To prevent temporal cues from unduly influencing panelists, at each time point, a coded fresh butter (less than 72 h old) was included among the samples evaluated.

Table 2. Sensory Profiles of Butters across Refrigerated and Frozen Storage[a]

butter sample[b]	cooked/nutty	milkfat/lactone	refrigerator/stale	salty taste
1W-fresh	4.0 (0.3)	3.0 (0.3)	ND	8.9 (0.5)
2W-fresh	3.8 (0.3)	3.0 (0.2)	ND	9.5 (0.6)
2F-fresh	3.5 (0.4)	3.1 (0.3)	ND	9.2 (0.4)
1W-6 months, 4 °C	2.1 (0.3)	2.6 (0.4)	1.0 (0.5)	8.8 (0.6)
2W-6 months, 4 °C	1.9 (0.3)	2.5 (0.3)	1.1 (0.5)	9.3 (0.5)
2F-6 months, 4 °C	2.2 (0.4)	2.7 (0.3)	ND	9.2 (0.5)
1W-12 months, 4 °C	1.5 (0.5)	2.4 (0.3)	1.9 (0.6)	8.5 (0.6)
2W-12 months, 4 °C	1.4 (0.4)	2.3 (0.4)	1.7 (0.5)	8.9 (0.6)
2F-12 months, 4 °C	1.6 (0.4)	2.4 (0.3)	1.6 (0.6)	9.1 (0.4)
1W-12 months, −20 °C	2.8 (0.4)	2.8 (0.3)	0.5 (0.5)	9.1 (0.4)
2W-12 months, −20 °C	2.7 (0.3)	2.7 (0.4)	0.5 (0.3)	9.5 (0.5)
2F-12 months, −20 °C	3.1 (0.3)	2.9 (0.3)	ND	9.5
LSD[c]	0.3	0.3	0.3	0.7 (0.5)

[a] Attributes were scaled on a 15-point universal Spectrum scale where 0 = absence of attribute and 15 = high intensity. [b] 1W and 2W are facilities 1 and 2, respectively, packaged in parchment, and 2F is facility 2 packaged in foil. [c] LSD = least significant difference. Means within an attribute that differ by more than the LSD are different ($p < 0.05$). Standard deviations of each attribute are in parentheses.

Gas Chromatography–Olfactometry (GCO). Dynamic headspace analysis in conjunction with gas chromatography–olfactometry is a method that allows efficient recovery and characterization of the majority of diverse volatiles found in dairy products, especially highly volatile aroma compounds. For dynamic headspace analysis (DHA)–GCO, a butter sample (10 g) was first equilibrated at 40 °C in a 100 mL three-neck glass purge vessel (Scientific Instrument Services, Ringoes, NJ) with constant stirring for 10 min, followed by purging of the headspace volatiles onto a Tenax TA adsorbent tube (200 mg, 60/80 mesh; Supelco, Bellefonte, PA) using ultrahigh-purity nitrogen (flow rate 50 mL/min) for 25 min. The adsorbent trap was removed from the system and dry purged (50 mL/min) for 17 min to remove moisture. The trap was then thermally desorbed for 10 min at 220 °C (TDS2, Gerstel, Germany) into a CIS4 (Gerstel, Germany) inlet held at −150 °C (solvent vent mode, 50 mL/min helium vent flow). The CIS 4 inlet was then heated (12 °C/s from −150 to 260 °C with final hold time of 10 min; purge value delay was 1.10 min) in the splitless mode, and the desorbed compounds were injected into the GC column of an Agilent 6890 GC (Palo Alto, CA) equipped with an OD2 olfactometry

Table 3. Effect of Storage Conditions on the Intensity of Aroma-Active Compounds of Butter by DHA–GCO[a]

		RI		fresh (0 months)			6 months, 4 °C			12 months, 4 °C			12 months, −20 °C		
compound	aroma	FFAP	DB5	1W[b]	2W	2F	1W	2W	2F	1W	2W	2F	1W	2W	2F
dimethyl sulfide	sulfur	760	<500	1.8	1.5	1.0	1.5	1.0	1.0	0.0	0.0	2.0	0.5	0.8	1.0
2-methylpropanal	dark chocolate	842	–[c]	1.5	1.8	1.5	1.0	0.8	1.7	1.0	0.5	4.0	2.5	3.0	0.0
ethyl acetate	fruity	889	651	0.0	0.0	0.0	0.3	0.0	0.0	0.5	0.0	0.0	1.0	0.5	0.0
2/3-methylbutanal	dark chocolate	933	573	1.8	0.0	1.0	2.8	2.0	1.0	1.0	0.0	3.0	2.0	2.0	1.0
diacetyl	buttery	991	579	3.0	2.0	2.0	3.0	2.3	3.0	3.0	2.0	4.0	3.0	3.5	4.0
α-pinene	mint, pine oil	1003	–	0.8	0.5	1.5	1.0	0.8	1.5	2.0	0.0	0.0	0.5	0.0	0.0
ethyl butanoate	fruity, berry	1048	779	2.0	1.8	2.0	0.3	0.8	2.0	2.0	1.0	0.0	2.5	3.0	1.0
hexanal	green, grass	1069	802	1.8	1.0	0.0	1.5	1.5	1.5	2.0	2.0	1.5	1.0	2.0	1.0
1-hexen-3-one	plastic	1087	763	2.3	1.5	1.5	1.0	2.0	1.0	3.5	1.5	3.0	1.5	1.5	3.0
2-heptanone	fatty	1181	837	0.0	0.0	0.0	1.5	1.3	0.0	1.0	3.5	0.0	1.0	1.0	1.0
(*Z*)-4-heptenal	rancid, crabby	1237	888	0.5	0.8	0.0	2.3	1.5	1.5	2.0	2.0	2.0	0.0	2.0	0.0
ethenylbenzene (styrene)	styrene, plastic	1241	895	0.0	0.0	0.0	4.5	3.8	1.0	5.5	7.0	6.0	1.0	1.5	0.0
1-octen-3-one	mushroom	1299	1007	2.8	1.8	2.0	2.3	2.5	3.0	2.0	0.5	2.0	2.8	3.0	4.0
2-acetyl-1-pyrroline	popcorn	1335	928	3.0	3.5	3.0	2.0	2.5	3.0	3.5	2.0	2.0	4.0	3.0	4.0
dimethyl trisulfide	cabbage	1387	964	3.0	3.5	3.0	1.5	2.0	1.5	2.0	3.0	3.0	3.5	3.5	6.0
nonanal	mushroom	1402	1102	0.8	0.5	1.5.	0.0	0.0	0.0	0.5	1.5	1.0	0.0	1.0	0.0
acetic acid	vinegar	1435	–	0.3	2.0	0.0	3.0	2.0	1.0	1.5	2.0	2.0	0.5	0.5	1.0
decanal	green, fatty	1495	1214	0.5	0.0	0.0	0.0	0.0	0.0	3.0	0.0	0.0	0.0	0.0	0.0
(*Z*)-2-nonenal	hay, fatty	1502	–	1.0	1.3	1.0	0.5	0.0	0.0	1.0	1.0	0.0	1.5	1.5	2.0
(*E*)-2-nonenal	hay	1515	–	1.0	1.5	0.5	1.8	2.3	1.0	1.0	3.0	2.0	1.5	1.5	0.0
(*E,Z*)-2,6-nonadienal	cucumber	1596	–	0.0	0.3	0.0	2.3	0.8	0.5	0.5	1.0	2.0	2.0	0.5	0.0
butanoic acid	fecal, cheesy	1625	–	2.0	2.5	2.0	3.0	1.0	1.0	1.0	1.5	2.0	2.0	2.5	2.0
3-methylbutanoic acid	sweaty	1645	–	1.0	2.5	0.0	2.3	1.5	2.5	1.0	1.0	1.0	0.0	0.5	2.0
2-acetyl-2-thiazoline	cooked	1759	1097	0.5	0.0	0.0	2.0	1.0	1.0	1.0	0.0	0.0	0.5	0.5	0.0
β-damascenone	apple sauce	1818	–	0.8	1.0	0.5.	0.5	1.0	1.0	1.5	0.0	2.0	1.5	1.0	2.0
hexanoic acid	doughy, sweaty	1854	–	0.0	0.0	0.0	0.8	0.0	1.0	0.0	0.0	0.0	2.0	1.0	4.0
δ-octalactone	herbaceous	1988	1302	1.8	0.8	1.0	1.0	0.8	2.0	2.0	2.0	1.	3.0	3.0	2.0
γ-nonalactone	peachy	2012	1350	2.5	2.3	1.5	0.0	0.0	0.0	0.5	0.0	0.0	1.5	1.0	0.0
δ-decalactone	waxy, sweet	2189	1463	2.0	2.5	2.0	3.0	3.3	1.5	0.8	1.0	0.0	1.5	1.0	1.0
δ-undecalactone	coconut, butter	2264	–	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
δ-dodecalactone	coconut, cheesy	2426	>1700	0.8	1.3	0.0	2.3	2.5	2.0	1.8	0.5	1.0	1.5	2.0	1.0
skatole	skatole	2516	–	2.3	1.0	0.0	1.8	1.8	1.0	2.0	0.0	1.5	0.5	0.0	0.0

[a] DHA-GCO average intensity ($n = 2$) on an 8-point scale, where 0 = none, 7 = very strong. [b] 1W and 2W are facilities 1 and 2, respectively, packaged in parchment, and 2F is facility 2 packaged in foil. [c] –, not detected.

port (Gerstel, Germany). Two different polarity columns were employed to obtain retention indexes (*17*): DB-5MS (15 m × 0.32 mm i.d. × 0.5 μm film) (J&W Scientific, Folsom, CA) and Stabilwax DA (FFAP) (15 m × 0.32 mm i.d. × 0.5 μm film) (Restek, Bellefonte, PA). The GC oven was programmed from 35 to 225 °C at 10 °C/min with initial and final holding times of 5 and 20 min, respectively. Helium was used as a carrier gas at 2.2 mL/min. Two experienced sniffers evaluated each sample using an 8-point intensity scale as previously described by Cadwallader et al. (*18*).

Solvent-assisted flavor evaporation (SAFE)–GCO was chosen as an effective method to extract and analyze the main intermediate and semivolatile aroma components of butter from their lipid nonvolatile matrix (*19*). One quarter (112 g) of butter was combined with 440 mL of diethyl ether in a 1000 mL flask. The mixture of ether and butter was stirred for 5 min at 30 °C, allowing the butter to dissolve into the ether. After cooling the mixture to room temperature (25 °C), 10 μL of internal standard solution (containing 46.5 mg of 2-ethylbutyric acid and 51.2 mg of 2-methyl-3-heptanone in 10 mL of methanol) was added. Over a period of 30 min the mixture was fed in dropping aliquots into the SAFE system (distillation head at 35 °C, sample flask at 40 °C, and vacuum at less than 1×10^{-4} Torr). Then the SAFE unit was kept at 10^{-5} Torr for 2 h prior to removal of the extract from the system. This extract was thawed for ~2 h (no ice present) before being transferred to a 500 mL round-bottom flask and then concentrated to 25 mL by distillation at 42 °C using a Vigreaux column. Anhydrous sodium sulfate (1 g) was added directly to the extract to remove water. The remaining liquid extract was filtered through another 1 g of anhydrous sodium sulfate anhydrous and concentrated to 100 μL under a gentle stream of nitrogen gas. The concentrated extract was injected (1 μL) using a cool on-column method (+3 °C; oven tracking mode) into a GCO system operated under the same conditions described for GCO–DHA. Average odor intensity of each compound was based on results of two experienced panelists as described for DHA–GCO.

Identification and Quantification of Volatiles. The main odorants of salted AA butter were identified by GC–MS. Two microliters of each organic extract obtained by SAFE was transferred into an Agilent 6890N GC/5973N mass selective detector (MSD) system by cold on-column injection (+3 °C; oven tracking mode). Two columns with the same dimensions (30 m × 0.25 mm i.d. × 0.25 μm film thickness) but having different polarities (FFAP or DB-5MS) (J&W Scientific, Folsom, CA) were employed. The oven was programmed from 35 to 225 °C at a rate of 10 °C/min with initial and final hold times of 5 and 25 min, respectively. Helium was used as carrier gas at a constant rate of 1.0 mL/min. MSD conditions were as follows: MS transfer line heater, 280 °C; ionization energy, 70 eV; mass range, 40–300 amu; scan rate, 5 scans/s. For better sensitivity the electron multiplier voltage was 200 V above the autotune setting.

Compound Identification. Compound identification was based on matching retention indices (on two different column phases) and mass spectra of unknown with those of authentic standards. An *n*-alkane series was used for the determination of retention indices (*17*).

Semiquantitation of Compounds. A semiquantitative estimate of the concentration of each positively identified odorant was based on its area response ratio [total ion chromatogram (TIC) area of compound/TIC area of internal standard, 2-methyl-3-heptanone] as described by Baek and Cadwallader (*20*) and Zhou et al. (*21*). The results were expressed in ppb or μg/kg of butter.

Quantitative Analysis of Selected Benzene Derivatives. Selected storage-related volatile compounds in butter were quantified using solid phase microextraction (SPME). Butter (10 g) plus a Teflon-coated octagonal stir bar (8 mm o.d. × 13 mm length) (Fisher Scientific, Pittsburgh, PA) was transferred into a precleaned 40 mL amber vial equipped with a screw cap and Teflon-lined silicon septum (Supelco, Bellefonte, PA). Internal standard solution (2-methyl-3-heptanone in methanol) was added to all sample vials to control for analysis variability at a final concentration of 81 ppb. The sample was allowed

Table 4. Effect of Storage Conditions on the Intensity of Aroma-Active Compounds of Butter by SAFE-GCO[a]

		RI		fresh (0 months)			6 months, 4 °C			12 months, 4 °C			12 months, −20 °C		
compound	aroma	FFAP	DB5	1W[b]	2W	2F	1W	2W	2F	1W	2W	2F	1W	2W	2F
dimethyl sulfide	sulfurous	760	<500	0.0	0.0	0.0	0.0	0.0	1.0	0.0	0.0	0.0	0.0	0.0	0.0
2-methyl propanal	dark chocolate	842	–[c]	0.5	0.0	0.0	0.0	0.0	1.0	0.5	1.0	2.0	2.0	1.5	1.0
ethyl acetate	fruity	889	651	2.0	0.0	4.0	2.5	1.5	3.0	3.0	0.0	0.0	2.0	0.5	0.0
2/3-methylbutanal	dark chocolate	933	573	2.0	1.0	2.0	0.0	1.0	1.0	0.5	0.0	0.0	0.0	0.0	0.0
diacetyl	buttery	991	579	1.0	2.0	2.0	2.0	1.0	1.0	0.5	1.5	1.0	2.5	1.0	1.0
α-pinene	mint	1003	–	2.0	0.0	1.0	0.5	0.5	1.0	0.0	0.0	0.0	1.0	0.5	0.0
ethyl butanoate	raspberry	1048	779	1.0	0.0	0.0	0.5	0.0	2.0	0.5	0.5	0.0	1.5	2.0	1.0
hexanal	green, grass	1069	802	2.0	2.5	2.0	2.0	1.5	2.0	3.5	4.5	4.0	1.5	0.0	0.0
1-hexen-3-one	plastic	1087	763	0.5	0.0	0.0	3.5	1.0	1.0	3.0	3.5	1.0	1.5	0.0	0.0
2-heptanone	fatty	1181	837	1.5	0.0	3.0	2.5	2.0	1.0	1.0	1.5	0.0	1.5	0.0	0.0
(*Z*)-4-heptenal	rancid, crabby	1237	888	2.5	1.0	2.0	3.5	3.0	3.0	2.5	3.5	3.0	3.5	3.0	0.0
ethenylbenzene (styrene)	styrene, plastic	1241	895	1.0	0.0	0.0	6.0	5.0	5.0	7.0	7.0	2.0	5.0	3.0	3.0
1-octen-3-one	mushroom	1299	1007	2.0	1.5	4.0	2.5	2.5	3.0	4.0	2.0	2.0	1.0	4.0	1.0
2-acetyl-1-pyrroline	popcorn	1335	928	4.0	5.0	6.0	2.5	2.0	5.0	3.5	3.5	1.0	4.0	2.5	1.0
dimethyl trisulfide	cabbage	1387	964	1.0	1.0	4.0	4.0	0.0	1.0	4.0	4.0	3.0	2.0	3.5	2.0
nonanal	mushroom	1402	1102	1.0	3.5	2.0	1.0	0.0	0.0	2.0	3.0	5.0	4.0	4.5	3.0
acetic acid	vinegar	1435	–	2.0	1.5	0.0	4.0	2.0	1.0	4.0	4.5	1.0	1.5	4.0	2.0
decanal	green, fatty	1495	1214	3.5	1.0	2.0	0.5	0.0	0.0	2.5	1.0	0.0	2.5	1.0	4.0
(*Z*)-2-nonenal	hay, fatty	1502	–	0.0	1.5	3.0	1.0	0.0	0.0	4.0	2.0	0.0	2.5	1.5	2.0
(*E*)-2-nonenal	hay	1515	–	2.5	0.0	0.0	2.0	3.0	3.0	0.5	2.5	2.0	1.0	1.5	0.0
(*E,Z*)-2,6-nonadienal	cucumber	1596	–	1.0	0.0	2.0	1.5	0.5	2.0	1.0	1.0	2.0	2.0	0.5	0.0
butanoic acid	fecal, cheesy	1625	–	5.0	5.5	6.0	3.0	4.0	6.0	2.0	1.5	0.0	2.0	5.5	2.0
3-methylbutanoic acid	sweaty, feet	1645	–	2.5	3.0	0.0	0.5	1.0	3.0	2.0	3.0	2.0	1.0	1.0	2.0
2-acetyl-2-thiazoline	cooked	1759	1097	4.0	2.5	3.0	1.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0
β-damascenone	apple sauce	1818	–	3.0	3.0	1.0	2.0	0.0	3.0	3.0	2.0	2.0	2.0	2.0	2.0
hexanoic acid	doughy, sweaty	1854	–	0.0	0.0	2.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
δ-octalactone	herbaceous	1988	1302	5.0	3.0	2.0	1.5	0.0	2.0	3.0	2.0	3.0	3.5	3.0	4.0
γ-nonalactone	peachy	2012	1350	5.0	3.5	1.0	0.0	0.0	0.0	1.5	0.0	0.0	0.5	0.5	0.0
δ-decalactone	waxy, sweet	2189	1463	2.5	1.0	0.0	2.5	1.0	2.0	2.0	1.5	2.0	2.0	1.0	0.0
δ-undecalactone	coconut, butter	2264	–	2.0	0.0	2.0	1.5	2.0	0.0	1.5	2.0	2.0	2.0	1.5	0.0
δ-dodecalactone	coconut, cheesy	2426	>1700	0.0	0.0	0.0	1.5	2.0	1.0	0.0	0.0	0.0	1.5	1.5	0.0
skatole	skatole	2516	–	2.0	1.0	0.0	0.0	0.5	0.0	0.5	0.0	1.0	0.0	0.0	0.0

[a] SAFE-GCO average intensity ($n = 2$) on an 8-point scale, where 0 = none and 7 = very strong. [b] 1W and 2W are facilities 1 and 2, respectively, packaged in parchment, and 2F is facility 2 packaged in foil. [c] –, not detected.

to equilibrate in the vial at 40 °C for 30 min using a Reacti-Therm heating/stirring module (Pierce, Rockford, IL). Volatile compounds were extracted by exposing a 1 cm–50/30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) StableFlex SPME fiber (Supelco, Bellefonte, PA) to the headspace of each sample at 40 °C for 30 min at a depth of 2 cm. The fiber was desorbed by splitless injection at 250 °C for 5 min in the injection port fitted with a SPME inlet liner (Supelco, Bellefonte, PA) at a depth of 3 cm into an Agilent 6890N GC/5973N MSD system. Separations were performed on a fused silica capillary column (DB-5MS, 30 m length × 0.25 mm i.d. × 0.25 μm df) (J&W Scientific, Folsom, CA). Helium gas was used as a carrier at a constant flow of 1 mL/min. The oven temperature was programmed from 35 to 200 °C at a rate of 10 °C /min with initial and final hold times of 3 and 10 min, respectively. MSD conditions were the same as previously described. Duplicate analyses were performed on each sample.

The concentration of a compound was determined from its area response ratio (TIC area of compound/TIC area of internal standard). Individual response factors were determined for styrene, ethylbenzene, and toluene at four levels using fresh butter free of these compounds as a reference control matrix (styrene levels 10–2000 ng/g (ppm), ethylbenzene levels 1–100 ppb, and toluene levels 10–200 ppb). Calculations were performed as described by Zhou et al. (*21*).

Threshold Determination of Selected Benzene Derivatives. Best estimate thresholds of styrene, ethylbenzene, and toluene were determined using the ASTM ascending forced choice method of limits procedure E679-79 (*22*). All thresholds were determined orthonasally in vegetable oil. Stock solutions were prepared in methanol. Aliquots of these stock solutions were placed into vegetable oil. Panelists ($n = 35$) were given these concentrations in a series with two vegetable oil blanks containing the same volume of added methanol. Seven ascending series were tested. Series were presented in ascending concentration. The individual best estimate threshold was calculated as the geometric mean of the last concentration with an incorrect response and the first concentration with a correct response. The group best estimate threshold (BET) was calculated as the geometric mean of the individual best estimate thresholds.

Statistical Analysis. Sensory and instrumental data were evaluated by analysis of variance with means separation. Principal components analysis was also applied to determine how butter flavors and volatile compounds changed with storage time. All analyses were conducted using SAS (version 9.1; Cary, NC).

RESULTS AND CONCLUSIONS

Flavor of Butter. Fresh butters were characterized by cooked/nutty and milkfat flavors with high salty taste intensities (**Table 2**). These butters were not manufactured from cultured cream, so they were not expected to display overt diacetyl notes although the panel was trained to identify and scale this attribute. Freshly churned salted butter is characterized by an intense cooked/nutty flavor which likely comes from the high heat treatment that the cream receives prior to churning (*23*). This flavor is known to rapidly dissipate in butter (*23*). In general, cooked/nutty flavor decreased more rapidly in butters across storage compared to milk fat flavor while salty taste was unchanged with storage time.

Beginning at 6 months for refrigerated butter, low intensities of a "stale" flavor were documented by the panel (**Table 2**). This attribute was described as "plastic-like" and "chemical" before the panel agreed on the descriptor stale/refrigerator since the flavor was best typified by butter that had been stored at 4 °C for more than 1 year. This flavor increased in intensity in refrigerated butters between 6 and 12 months of storage. After 12 months of storage at −20 °C, this flavor was present at sensory threshold levels in the two butters packaged in wax

Table 5. Semiquantitative Concentrations (μg/kg) of Selected Aroma Compounds in Butter

	fresh (0 months)			6 months, 4 °C			12 months, 4 °C			12 months, −20 °C		
compounds	1W[b]	2W	2F	1W	2W	2F	1W	2W	2F	1W	2W	2F
ethyl acetate	2.7 d[a]	8.4 d	0 d	56.9 bc	130.0 a	105.4 ab	123.7 a	94.8 ab	45.5 bcd	16.7 cd	88.7 ab	50.0 bcd
diacetyl	0.8 c	0 c	0 c	0 c	7.4 b	0 c	1.8 bc	4.7 bc	7.5 b	3.4 bc	0 c	78.2 a
α-pinene	0 b	2.8 b	4.7 b	7.2 b	36.2 a	0 b	0.5 b	1.2 b	0.9 b	7.0 b	2.5 b	1.4 b
toluene	512.8 bc	386.4 bc	528.6 abc	141.9 c	282.7 bc	129.9 c	2159.0 a	728.9 abc	712.0 abc	114.7 c	1791.7 ab	2606.8 a
hexanal	1.3 c	3.6 c	0 c	0 c	36.0 b	64.39 a	0 c	0 c	0 c	0 c	0 c	0 c
2-heptanone	23.7 bc	38.9 abc	26.5 abc	19.9 bc	51.0 ab	51.4 ab	34.7 abc	57.5 a	19.0 bc	7.2 c	9.1 c	6.1 c
limonene	67.6 a	147.3 a	124.8 a	34.8 a	179.4 a	83.8 a	171.2 a	20.3 a	156.6 a	9.8 a	21.5 a	184.5 a
styrene	22.7 d	39.1 d	0 d	372.4 bcd	673.4 b	174.7 cd	1164.9 a	1173.5 a	276.7 bcd	606.6 bc	322.3 bcd	100.5 d
acetic acid	293.3 ab	322.2 ab	488.1 a	319.6 ab	97.5 b	32.9 b	355.9 ab	417.9 a	349.3 ab	442.7 a	542.9 a	281.4 ab
butanoic acid	273.3 b	222.8 b	329.0 b	374.2 b	258.2 b	239.2 b	939.6 a	1059.4 a	665.6 ab	801.9 a	328.5 b	279.2 b
3-methylbutanoic acid	29.2 b	184.2 a	59.8 b	28.1 b	15.3 b	5.5 b	54.9 b	25.9 b	34.2 b	41.6 b	23.5 b	31.9 b
hexanoic acid	330.3 ab	0 d	376.0 ab	228.2 abc	199.7 bc	63.1 cd	381.4 ab	406.7 a	276.7 abc	339.5 ab	286.9 abc	156.5 bcd
δ-hexalactone	0 b	0 b	0 b	8.4 b	32.1 ab	35.7 ab	53.7 a	53.5 a	43.6 ab	47.0 a	49.5 a	29.6 ab
δ-octalactone	42.4 b	62.6 b	36.6 b	14.8 b	42.3 b	10.2 b	41.1 b	45.9 b	913.4 a	40.9 b	27.1 b	16.9 b
δ-decalactone	46.9 bc	59.8 bc	35.1 bc	34.7 c	61.8 abc	34.5 c	88.3 ab	74.4 abc	89.9 ab	103.2 a	48.7 bc	35.2bc
δ-dodecalactone	10.3 d	12.2 d	2.7 d	29.9 c	48.4 b	70.0 a	4.1 d	5.5 d	8.8 d	9.0 d	1.1 d	4.2 d
skatole	0.6 a	0.5 a	0.2 ab	0 b	0 b	0 b	0 b	0 b	0 b	0 b	0 b	0 b
ethyl butanoate	0 b	0 b	0 b	0 b	0 b	0 b	0 b	0 b	0 b	1.8 a	0 b	0 b
propanoic acid	0 b	0 b	0 b	0 b	0 b	0 b	0 b	0 b	0 b	24.8 a	0 b	0 b
2-methylpropanic acid	0 c	0 c	0 c	0 c	0 c	0 c	61.2 a	22.8 bc	42.2 ab	0 c	0 c	0 c
octanoic acid	0 c	0 c	0 c	0 c	0 c	0 c	0 c	0 c	0 c	175.3 a	112.3 b	95.2 b

[a] In rows, means ($n = 2$) followed by different letters are significantly different to $p < 0.05$. [b] 1W and 2W are facilities 1 and 2, respectively, packaged in parchment, and 2F is facility 2 packaged in foil.

Table 6. Concentrations (μg/kg) of Selected Benzene Derivatives in Butter after 12 months of Storage[a]

butter sample[b]	styrene	ethyl benzene	toluene
1W-12 months, −20 °C	261 ± 34.0	60 ± 0.2	76 ± 6.6
2W-12 months, −20 °C	129 ± 22.3	41 ± 0.0	64 ± 2.9
2F-12 months, −20 °C	9 ± 1.0	2 ± 0.2	13 ± 2.9
1W-12 months, 4 °C	1298 ± 128	49 ± 14	107 ± 45
2W-12 months, 4 °C	840 ± 55.2	88 ± 7.3	149 ± 92
2F-12 months, 4 °C	53 ± 1.0	5 ± 0.2	58 ± 2.9

[a] Average ± standard deviation ($n = 2$). [b] 1W and 2W are facilities 1 and 2, respectively, packaged in parchment, and 2F is facility 2 packaged in foil.

paper. In general, sensory changes (loss of cooked/nutty and milkfat flavor and evolution of refrigerator/stale flavor) were more evident in refrigerated butters compared to frozen butters, and within each storage condition, butter packaged in foil wrappers maintained fresh flavors better than quarters wrapped in wax parchment paper ($p < 0.05$).

Main Aroma Components of Butter. Thirty-two and 27 aroma-active compounds were identified by SAFE-GCO (**Table 3**) and DHA–GCO (**Table 4**), respectively. These two complementary extraction methods were expected to provide different results. For example with SAFE, highly volatile compounds such as dimethyl sulfide (bp 37 °C) may be lost during workup and concentration of the extract. This compound was only detected in foil wrapped butter at 6 months refrigeration by SAFE but was recovered in nearly all samples by DHA. In contrast, less volatile compounds such as lactones are well-recovered by SAFE and only poorly recovered by DHA. **Tables 3** and **4** show that butanoic acid, 1-octen-3-one, diacetyl (2,3-butanedione), 2-acetyl-1-pyrroline, dimethyl trisulfide, δ-octalactone, and δ-decalatone were the main contributors to fresh butter aroma. These findings are in agreement with previous studies conducted on butter oil and butter (*1,4,6,10,13,24*). The contribution of diacetyl has been controversial, and several authors (*1, 4, 7*) suggest that its contribution is only marginal. The disagreement among researchers might be due to inherent differences among the types of butters analyzed. Cultured butter, made from cream that has been cultured or fermented by lactic acid bacteria, would be expected to display diacetyl notes. Sensory results in the present study suggest that diacetyl is of minor importance in sweet cream butter since it was not detected as a distinct sensory attribute of the butter. Other sensory studies with cultured butter have indicated that diacetyl plays a key role in the flavor of cultured butters (*16*).

Compounds identified in the present study that were not previously identified in sweet cream butter are guaiacol, 2-acetyl-1-pyrroline, and 2-acetyl-2-thiazoline. Pasteurization of the cream in the production of butter might be responsible for the formation of these three odorants (*25, 26*). Compounds such as methylpropanal, 3-methylbutanal, hexanal, 1-hexen-3-one, δ-octalactone, and skatole were detected with medium intensities (ratings between 2 and 3 on an 8-point scale) (**Tables 3** and **4**). The remaining compounds identified were considered background odorants (intensities < 2); however, their contribution should not be overlooked, as in the case of (*E*)- and (*Z*)-2-nonenal and acetic and hexanoic acids, which have been previously reported in butter oil and butter at concentrations exceeding their odor detection thresholds (*4, 10*).

Effect of Storage on the Major Aroma Compounds in Salted Butter. The effect of storage time (6 and 12 months) as well as temperature of storage (4 and −20 °C) is shown in **Tables 3** and **4**. Sulfur-containing compounds such as dimethyl sulfide (*27*) and dimethyl trisulfide (*1, 25*) have been mentioned as possible contributors to butter aroma. Dimethyl sulfide showed a considerable decrease in intensity and seems to contribute to butter aroma only for a short period following manufacture, possibly due to loss by evaporation since it has a low boiling point of 37.4 °C (*28*). This result agrees with the sensory results which indicate a loss of cooked/nutty flavor with refrigerated storage. Dimethyl sulfide is possibly a large contributor to the characteristic, intense, and elusive cooked/nutty flavor of fresh butter.

The main changes detected in aroma-active volatile compounds were an increase in the intensity of lactones (δ-octalactone, δ-decalactone, and δ-dodecalactone) as well as an increase in lipid-derived compounds [(*E*)-2-nonenal, 2-heptanone, (*Z*)-4-heptenal, (*E,Z*)-2,6-nonadienal] with storage time. (*E*)-2-Nonenal was reported to cause a cardboard-like off-flavor in butter oil stored at room temperature (*10, 11*). During refrigeration storage, the odor intensity of hexanal did not

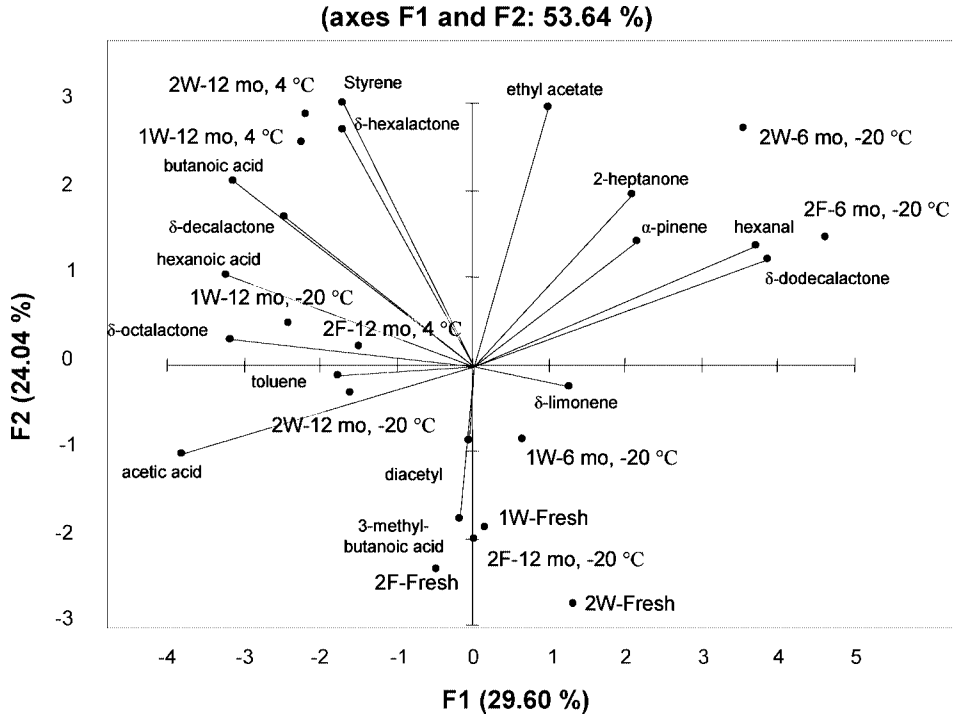

Figure 1. Principal Component Biplot of the Average Relative Concentrations ($n = 2$) of Selected Aroma Compounds in Butter

increase for up to 6 months, but this odorant showed a marked increase in intensity after 12 months. This result is in agreement with previous studies that reported no significant ($p < 0.05$) change in hexanal concentrations when butter was stored for up to 14 weeks at 4 °C (*12*). Acidic odorants, such as butanoic and acetic acids, increased in intensity after 6 and 12 months of refrigeration storage. Meanwhile, hexanoic acid, diacetyl, and 1-octen-3-one underwent no considerable change in intensity after 6 and 12 months of refrigeration storage.

The observed increase in odor intensities of δ-octalactone and δ-decalactone as a function refrigeration storage may have been caused by the hydrolysis of 4-/5-hydroxy fatty acids or oxygen attack on fatty acids at positions 4 or 5 followed by a hydrolysis (*12*). Meanwhile, γ-nonalactone was identified only in fresh butters and butters stored at −20 °C. This suggests that γ-nonalactone is stable at frozen storage conditions, but its oxidation may occur at refrigeration temperatures. In butters stored for 12 months at −20 °C, modest increases in the odor intensities of most of the lactones and lipid oxidation products were observed, and the intensity of dimethyl sulfide declined. At this low temperature, slower rates for conversion of δ-hydroxy products and for lipid oxidation reactions would be expected, and evaporation of highly volatile compounds such as dimethyl sulfide should be retarded. Intensities of butyric and hexanoic acids increased after 12 months of frozen storage. However, on the basis of relatively low odor intensities of these compounds, they are not expected to make a significant contribution to the overall flavor of butter in accordance with other reports (*24*).

Relative concentrations of selected aroma compounds in fresh and stored butters are show in **Table 5**. In agreement with GCO results, levels of δ-decalactone, δ-octalactone, and acetic and butanoic acids increased slightly after 12 months of refrigerated storage. δ-Dodecalactone initially increased after 6 months of refrigeration storage and then decreased to levels originally found in the fresh butters. Meanwhile, frozen storage did not affect the levels of δ-dodecalactone. Skatole decreased to below its detection limit during both refrigeration and frozen storage. Terpenes, such as α-pinene and limonene, may have undergone autoxidation at refrigeration temperatures, as proposed by Schrader et al. (*29*), and their levels decreased after 6 and 12 months of refrigeration storage.

Impact of Package Material. Packaging material impacted sensory perception of butter flavor as well as instrumental results (**Tables 2**–**6**). By both isolation techniques, butters packaged in wax paper showed higher intensities of styrene than butter packaged in foil at 6 and 12 months of refrigeration storage. Even under frozen storage the intensity of styrene was higher compared to fresh butters (**Table 6**). To eliminate the possibility of styrene migration from the storage environment (refrigerator or freezer), the paper board from fresh butter packaging as well as parchment and foil along with Styrofoam (refrigerator insulation) were analyzed (data not shown). Ethylbenzene was selected because of the high levels present in foam insulation and its presence in literature as a packaging off flavor in butter (*30*, *31*). This pattern suggests that styrene migration from the package may occur even at low temperatures and that frozen storage can decrease the rate. Similar studies involving yogurts and chocolate desserts (*30*), commercial cheese blocks (20 kg) (*31*), and domestic use Cheddar cheese (*32*) kept at refrigeration temperatures have also reported the migration of styrene and styrene derivatives from the packaging material. In our case, aluminum foil seems to be the best packaging material to minimize styrene migration into the final product. Butter frozen for 12 months in foil is more similar to fresh butters than refrigerated butters (**Figure 1**).

Sensory results also confirm these findings. Refrigerator/stale flavor was not detected in foil-wrapped butter after 6 months at 4 °C while this flavor was detected in wax paper wrapped quarters following 6 months at 4 °C (**Table 2**). After 12 months of frozen storage, refrigerator/stale flavor was detected at very low intensities and was significantly lower compared to butters stored at 4 °C. Other fresh butter flavors (cooked/nutty and milkfat) were also higher in frozen butter than in refrigerated butters. Odor detection thresholds determined in oil for styrene (3.4 ± 0.6 mg/kg), ethylbenzene (4.1 ± 0.6), and toluene (94.7 ± 0.5) exceeded the levels of these compounds found in the butters. Therefore, these

benzene derivatives alone do not seem to be responsible for refrigerator/stale flavor. It is possible that an additive effect is at work and that subthreshold levels are detectable in butter when levels of multiple benzene derivatives increase and/or when fresh flavor compounds decrease and other storage-related flavor compounds increase (as observed during storage at refrigeration temperature). Furthermore, thresholds are matrix dependent. Detection thresholds for highly nonpolar compounds in water would be lower than in oil (*33*). Butter composition is ~80% fat and 20% water. Since our thresholds were conducted in oil, thresholds for these compounds in butter would presumably be lower, and this might also account for the thresholds being higher than concentrations found in stored butter.

The main aroma constituents identified in this study agreed well with previous investigations on butter and butter oil (*1*, *4*, *6*, *10*, *13*, *24*). Three compounds not previously reported in uncultured butter were found (2-acetylpyrroline, dimethyl trisulfide, and guaiacol). These compounds may be formed during the pasteurization process of the cream during manufacture. Intensities of several lipid oxidation products, including (*E*)-2-nonenal, (*Z*)-4-heptenal, and (*E,Z*)-2,6-nonadienal, were higher at refrigeration storage of butter than at frozen conditions. In addition, styrene migration from the package to butter was observed. This pattern was reduced by the use of freezing temperatures and aluminum foil as wrapping material.

LITERATURE CITED

(1) Peterson, D. G.; Reineccius, G. A. Characterization of the volatile compounds that constitute fresh sweet butter aroma. *Flav. Fragr. J.* **2003**, *18*, 215–220.

(2) Ramshaw, E. H. Volatile components of butter and their relevance to its desirable flavour. *Aust. J. Dairy Technol.* **1974**, *29*, 110–115.

(3) Kinsella, J. E. Butter flavor. *Food Technol.* **1975**, *29*, 82–98.

(4) Schieberle, P.; Gassenmeier, K.; Guth, H.; Sen, A.; Grosch, W. Character impact odor compounds of different kinds of butter. *Lebensm. Wiss. Technol.* **1993**, *26*, 347–356.

(5) Maarse, H.; Visscher, C. A. Volatile compounds in foods. In *Qualitative and Quantitative Data*; Maarse, H., Visscher, C. A., Willemsens, L. C., Nijssen, L. M., Boelens, M. H., Eds.; TNO Nutrition and Food Research Institute: Zeist, The Netherlands, 1994; sp5.

(6) Siek, T. J.; Lindsay, R. C. Semiquantitative analysis of fresh sweet-cream butter volatiles. *J. Dairy Sci.* **1969**, *53*, 700–703.

(7) Urbach, G.; Stark, W.; Forss, D. A. Flavour and flavour thresholds of lactones, fatty acids, phenols, indole and skatole in deodorized butter. *J. Dairy Res.* **1972**, *39*, 35–47.

(8) Stark, W.; Urbach, G.; Hamilton, J. S. Volatile compounds of butter oil. IV. Quantitative estimation of free fatty acids and free-δ-lactones in butter oil cold-finger molecular distillation. *J. Dairy Res.* **1976**, *43*, 469–477.

(9) Forss, D. A.; Stark, W.; Urbach, G. Volatile compounds in butter oil. I. Lower boiling points. *J. Dairy Res.* **1967**, *34*, 131–136.

(10) Widder, S.; Sen, A.; Grosch, W. Changes in the flavor of butter oil during storage - identification of potent odorants. *Z. Lebensm. Unters. Forsch.* **1991**, *193*, 32–35.

(11) Widder, S.; Grosch, W. Precursors of 2-nonenals causing the cardboard off-flavour in butter oil. *Nahrung-Food* **1997**, *41*, 42–45.

(12) Christensen, T. C.; Holmer, G. GC/MS analysis of volatile aroma components in butter during storage in different catering packaging. *Milchwissenschaft* **1996**, *51*, 134–139.

(13) Abdel-Mageed, M.; Fadel, H. M. Effect of freeze storage on the volatiles of butter. *Grasas y Aceites* **1995**, *46*, 165–168.

(14) Meyer, S. D.; Schreiber, S. L. Acceleration of the Dess-Martin oxidation by water. *J. Org. Chem.* **1994**, *59*, 7549–7552.

(15) Meilgaard, M.; Civille, G.; Carr, B. Descriptive analysis techniques. In *Sensory Evaluation Techniques*, 2nd ed.; Meilgaard, M., Civille, G., Carr, B., Eds.; CRC Press: Boca Raton, FL, 1999; pp 173–183.

(16) Krause, A. J.; Lopetcharat, K.; Drake, M. A. Identification of the characteristics that drive consumer liking of butter and vegetable oil spreads. *J. Dairy Sci.* **2007**, in press.

(17) van den Dool, H.; Kratz, P. D. A generalization of the retention index system including linear temperature programmed gas liquid partition chromatography. *J. Chromatogr.* **1963**, *11*, 463–471.

(18) Cadwallader, K. R. C.; Lopez, J. R.; Menke, S. D.; Surakarnkul, R. Aroma components of wines from chardonel: A French-American hybrid grape. In *Nutraceutical Beverages. Chemistry, Nutrition and Health Effects*; Shahidi, F., Weerasinghe, D. K., Eds.; *ACS Symposium Seies 871*; American Chemical Society: Washington, DC, 2004; pp 365–379.

(19) Engel, W.; Bahr, W.; Schieberle, P. Solvent assisted flavour evaporation- a new and versatile technique for the careful and direct isolation of aroma compounds from complex food matrices. *Eur. Food Res. Technol.* **1999**, *209*, 237–241.

(20) Baek, H. H.; Cadwallader, K. R. Roasted chicory aroma evaluation by gas chromatography/mass spectrometry/olfactometry. *J. Food Sci.* **1998**, *63*, 234–237.

(21) Zhou, Q.; Wintersteen, C. L.; Cadwallader, K. R. Identification and quantification of aroma-active components that contribute to the distinct malty flavor of buckwheat honey. *J. Agric. Food Chem.* **2002**, *50*, 2016–2021.

(22) ASTM. 679-79; Standard practice for determination of odor and taste thresholds by a forced-choice ascending concentration series method of limits, 1992.

(23) Bodyfelt, F. W.; Tobias, J.; Trout, G. M. Sensory evaluation of butter. In *The Sensory Evaluation of Dairy Products*; Van Nostrand Reinhold: New York, 1988; pp 376–417.

(24) Peterson, D. G.; Reineccius, G. A. Determination of the aroma impact compounds in heated sweet cream butter. *Flav. Fragr. J.* **2003**, *18*, 320–324.

(25) Lee, S.; Macku, C. Shibamoto, T. Isolation and identification of headspace volatiles formed in heated butter. *J. Agric. Food Chem.* **1991**, *39*, 1972–1975.

(26) Colahan-Sederstrom, P.; Peterson, G. D. Inhibition of key aroma compounds generated during ultrahigh-temperature processing of bovine milk via Epicatechin addition. *J. Agric. Food Chem.* **2005**, *53*, 398–402.

(27) Povolo, M.; Contarini, G. Comparison of solid-phase microextraction and purge-and-trap methods for the analysis of the volatile fraction of butter. *J. Chromatogr.* **2003**, *95*, 117–125.

(28) Shooter, D.; Jayatiss, N.; Renner, N. Volatile reduced sulphur compounds in butter by solid phase microextraction. *J. Dairy Res.* **1999**, *66*, 115–123.

(29) Schrader, W.; Geiger, J.; Klockow, D. Degradation of pinene on Tenax during sample storage: effects of daylight radiation and temperature. *Environ. Sci. Technol.* **2001**, *35*, 2717–2720.

(30) Ehret-Henry, J.; Ducruet, V.; Luciani, A.; Feigenbaum, A. Styrene and ethylbenzene migration from polystyrene into dairy-products by dynamic purge-and-trap gas-chromatography. *J. Food Sci.* **1994**, *59*, 990–992.

(31) Bendall, J. G. Food contamination with styrene dibromide via packaging migration of leachate from polystyrene cold-storage insulation. *J. Food Protect.* **2007**, *70*, 1037–1040.

(32) Fleming-Jones, M. E.; Smith, R. E. Volatile organic compounds in foods: a five year study. *J. Agric. Food Chem.* **2003**, *51*, 8120–8127.

(33) van Gemert, L. J. *Odour thresholds: compilations of odour thresholds values in air water and other media*; Oliemans Punter & Partners BV: The Netherlands, 2003.

Received for review April 12, 2007. Revised manuscript received July 20, 2007. Accepted July 21, 2007. Funding was provided in part by the California Dairy Research Foundation.

JF071075Q