Solid-Phase Extraction of Less Volatile Flavor Compounds from Ultrahigh-Temperature Processed Milk†

Kelegoun Coulibaly and Ike J. Jeon*

Department of Animal Sciences and Industry, Kansas State University, Manhattan, Kansas 66506

A rapid and sensitive, solid-phase extraction method was developed to isolate relatively nonvolatile flavor compounds from ultrahigh-temperature (UHT) milk. Various solid-phase materials were investigated including activated carbons, C_{18} reversed-phase silica, Florisil, and silica gel. The solid-phase materials packed in small columns (5-mL glass syringes) were treated with various solvents and loaded with the milk samples. Then, the compounds retained in the columns were eluted with methylene chloride and analyzed by gas chromatography or gas chromatography–mass spectrometry. Results indicated that the combination of activated carbon and C_{18} reversed-phase silica was the most effective in isolating relatively nonvolatile flavor compounds from UHT milk. Recoveries of the lactones added to milk were as low as 30 ppb, and reproducibility of the method appeared to be acceptable according to the coefficients of variation.

INTRODUCTION

The literature indicates that many different methods have been applied for the isolation of volatile flavor compounds from milk and milk products. However, the type of method employed is extremely important in the isolation of chemical compounds that are responsible for, or representative of, certain flavor characteristics in foods (Heath and Reineccius, 1986). It is also important for the speed, sensitivity, and reproducibility of the analysis. Solid-phase extraction (SPE) techniques are relatively new in food flavor isolation but have been used in recent years for sample preparation of drug residues and environmental contaminants. A wide spectrum of sorbents has been used in biological and environmental areas for SPE, including normal and reversed phase, adsorption, and size exclusion materials (Zief and Kiser, 1990). The use of SPE techniques was extensively reviewed and summarized (Liska et al., 1989; Zief and Kiser, 1990). Szumilo (1989) demonstrated that trace quantities of drugs and their metabolites in complex sample matrices such as plasma, urine, and tissue could be effectively extracted with nonionic adsorbent materials. Other researchers have succesfully applied SPE techniques for the isolation of organochlorine and organophosphorus pesticides from sediment and seafood samples by utilizing Florisil, silica, and C_{18} reversed phase (Kohler and Su, 1986; Marble and Delfino, 1988). Newsome and Collins (1989) utilized a C_{18} reversed-phase material to extract fungicide residues from acetone extracts of fruits and vegetables. Hutta et al. (1989) extracted short-chain fatty acids in drinking water using a carbonaceous sorbent as a solid-phase material. Deeth et al. (1983) utilized neutral alumina for the isolation of free fatty acids from milk and dairy products. Takacs (1989) reported that stale flavor components in ultrahigh-temperature (UHT) processed milk could be extracted with C_{18} Sep-Pak materials and subsequently eluted with methylene chloride. The stale components isolated from milk were short-chain fatty acids and neutral volatile compounds. The objective of this study was to develop a method that provides rapid and simple isolation and quantification of relatively nonvolatile flavor compounds from milk using solid-phase materials as a medium in combination with appropriate organic solvents.

MATERIALS AND METHODS

Materials. All chemicals used in this research were of analytical grades. The methylene chloride was obtained from Mallinckrodt, Inc. (Paris, KY), and diethyl ether, *n*-hexane, carbon disulfide, methanol, *n*-pentane, *n*-heptane, acetonitrile, acetone, and cyclohexane were purchased from Fisher Scientific Co. (St. Louis, MO). The solid-phase materials used included C_{18} reversed-phase silica (37–105 μm) from Waters Associates (Milford, MA), activated carbons [60/80 mesh (177–250 μm) and 80/100 mesh (149–177 μm)] from Anspec (Ann Arbor, MI), silica gel (100/200 mesh, grade 923) from Fisher Scientific, and Florisil (60/80 mesh) from Varian Aerograph (Walnut Creek, CA). UHT processed milk (both indirect and direct) samples in 802-mL (indirect) and 236-mL (direct) cartons were obtained from two different commercial processors and stored under refrigeration until used. Fresh pasteurized milk samples were obtained from the Kansas State University dairy processing plant.

Extraction Procedure. Glass syringes of 5 mL (Eisele Co., Nashville, TN) were utilized as extraction columns in this study. First, a small amount of glass wool (0.3 g) was inserted at the bottom of each column, and then 0.5 g of the solid-phase material was loaded. When a combination of two solid-phase materials was used, 0.25 g of each was utilized and activated carbon used as the top layer. Once the solid-phase material was loaded onto the column, it was covered with a small amount of glass wool (0.3 g) material at the top. An 18-gauge needle was connected to the glass syringe to regulate the flow of the solvents and milk samples. The attached needle was inserted through a conical rubber stopper and set on the top of a 125-mL sidearm filtering flask. The columns were treated first with 5 mL of methanol (or other solvents in preliminary works) followed by 10 mL of double-distilled water. Next, the columns were eluted with 100 mL of UHT milk samples by natural gravity. After the samples were eluted, the columns were washed with another 10 mL of double-distilled water. Then the columns were vacuum dried (700 mmHg) for 3 min and washed with 3 mL of redistilled *n*-hexane to remove any residual milk fat. After the columns were vacuum dried for 8 min, the compounds bound to the solid phases were eluted with 2.5 mL of redistilled methylene chloride and collected into 2-mL vials (Supelco, Inc., Bellefonte, PA). In preliminary studies, carbon disulfide and diethyl ether also were utilized in this stage. A flow diagram of the extraction procedure for UHT milk is outlined in Figure 1. Each methylene chloride eluted was concentrated to 0.5 or 1.0 mL using a stream of nitrogen gas, and 1 or 3 μL was injected into a gas chromatograph (GC) equipped with a flame ionization detector (FID). For gas chromatograph–

† Contribution No. 92-59-J from the Kansas Agricultural Experiment Station.

0021-8561/92/1440-0612$03.00/0

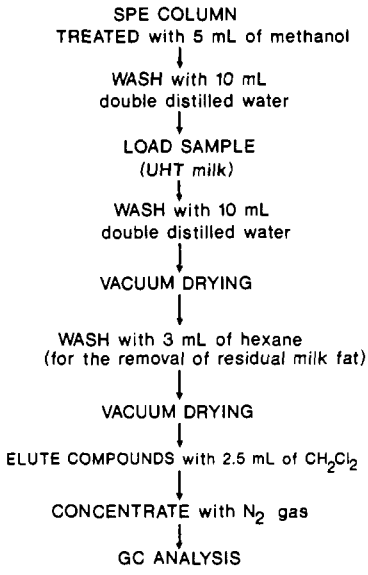

Figure 1. Flow diagram for the isolation of less volatile flavor compounds from UHT milk using the solid-phase extraction technique.

mass spectrometric analysis, 1 μL of 0.5-mL concentrates obtained from 500 mL of milk was injected.

To test the sensitivity of the combined solid-phase material, the seven lactone standards were prepared and added to 50 mL of pasteurized milk samples to obtain 30, 50, 70, and 90 ppb. The standards used were γ-valerolactone, δ-valerolactone, ε-caprolactone, γ-octalactone, γ-decalactone, δ-decalactone, and δ-dodecalactone. Then, milk samples were eluted through the extraction columns as indicated previously, and 3 μL of 0.5-mL concentrates was injected into the GC. The recovery of each added standard lactone was evaluated on the basis of peak areas.

Gas Chromatography. The gas chromatograph utilized in this study was a Hewlett-Packard Model 5880A in which the compounds were separated on a 5% methyl silicone cross-linked fused silica capillary column (30 m × 0.53 mm i.d. × 0.33 μm film thickness; Hewlett-Packard, Palo Alto, CA) with a one-level oven temperature program. The oven temperature was programmed from 40 to 205 °C at a rate of 7 °C/min and held for 15 min at the final temperature. The injection port and detector temperatures were both 250 °C. The carrier gas was helium with a flow rate of 8 mL/min.

Gas Chromatography–Mass Spectrometry. The gas chromatograph–mass spectrometer (GC–MS) used was a Hewlett-Packard Model 5890A gas chromatograph interfaced with a 5970 mass selective detector in which the compounds were separated on a 5% phenyl methyl silicone cross-linked fused silica capillary column (50 m × 0.2 mm i.d. × 0.33 μm film thickness; Hewlett-Packard). The oven temperature was programmed from 40 to 100 °C at a rate of 7 °C/min and from 100 to 280 °C at a rate of 10 °C/min and held for 15 min at the final temperature. The transfer line was maintained at 280 °C. Helium was used as the carrier gas with a flow rate of 1 mL/min. The spectra were obtained by electron ionization at 70 eV. Data were acquired with a HP 59970C ChemStation. Identification of the compounds was made by matching the mass spectra with the library spectral data as well as with the data of known standard compounds. The identification was considered positive when both spectra and GC retention times were matched.

Statistical Analysis. All experiments were replicated eight times for reproducibility, and the results were statistically treated by computing the peak areas for the mean and coefficients of variation.

RESULTS AND DISCUSSION

In preliminary studies, using 10 standard chemicals (ethyl acetate, 2-pentanone, 2-heptanone, 1-pentanol, 1-hexanal, phenol, 1-decanoic acid, and γ-hexa, -hepta-, and -octalactones) that were dissolved in diethyl ether,

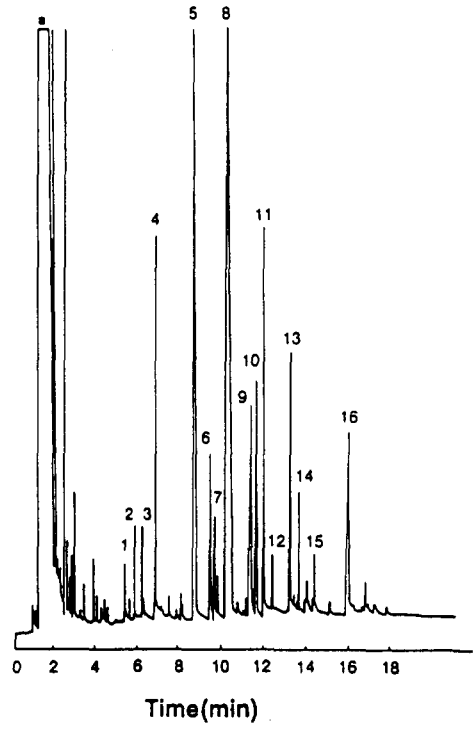

Figure 2. Typical gas chromatogram of volatile compounds isolated from 100 mL of UHT milk. Compounds were isolated using 0.5 g of activated carbon 60/80 mesh. Separations were achieved on a 5% methyl silicone fused silica capillary column (30 m × 0.53 mm i.d. × 0.33 μm film thickness). The oven temperature was programmed from 40 to 205 °C at a rate of 7 °C/min and held for 15 min at the final temperature (full run time not shown). "a" indicates solvent peak (methylene chloride).

four solid-phase materials were evaluated individually or in combination for their potential ability of binding chemicals. These included C_{18} reversed-phase silica, silica gel, activated carbon, and Florisil. Four organic solvents (diethyl ether, acetonitrile, acetone, and methanol) were also evaluated for their possible use as preconditioning agents for the solid-phase materials. The preliminary studies indicated that, among the solid-phase materials tested, 0.5 g of activated carbon (60/80 mesh) treated with methanol was the best for the retention of the 10 standard compounds. The standards retained on the activated carbon could not be eluted with *n*-hexane, *n*-pentane, *n*-heptane, or cyclohexane but could be eluted with methylene chloride except phenol and 1-decanoic acid. Because *n*-hexane did not remove the standards from the activated carbon, it was chosen as a solvent for removing residual fat and other contaminants from the solid phase. Further studies indicated that combining activated carbon (60/80 mesh) with C_{18} reversed phase was advantageous in retaining lactones as well as a number of other relatively nonvolatile compounds.

Figure 2 shows a typical gas chromatogram obtained from 100 mL of UHT milk using 0.5 g of activated carbon 60/80 mesh. It contains about 16 major peaks with 15-min retention time. However, as shown in Figure 3, a combination of activated carbon and C_{18} reversed phase yielded approximately 38 major compounds from the same volume of UHT milk. Moreover, it was observed that the peak areas were much larger with the combined solid phase than with the activated carbon alone. It appears that the combined solid phase was capable of isolating more compounds, as well as higher amounts, from UHT milk samples than the activated carbon alone. However, the elution time for 100 mL of milk through the activated carbon column was approximately 30 min, whereas it was

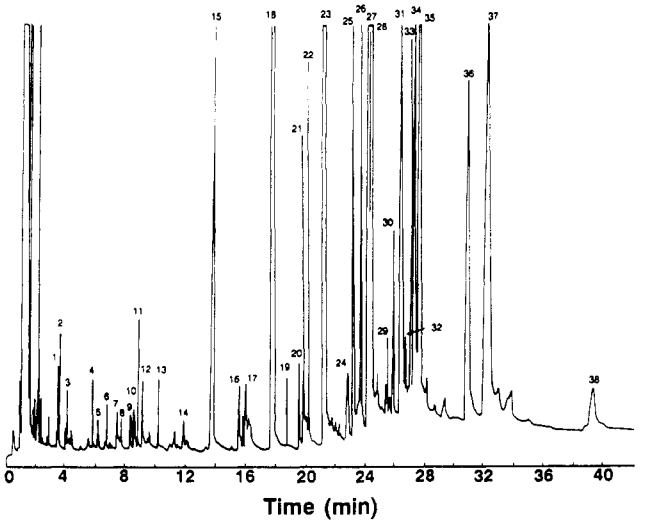

Figure 3. Typical gas chromatogram of volatile compounds isolated from 100 mL of UHT milk. Compounds were isolated using 0.25 g of activated carbon 60/80 mesh combined with 0.25 g of C_{18} reversed-phase silica. Separations were achieved on a 5% methyl silicone fused silica capillary column (30 m × 0.53 mm i.d. × 0.33 μm film thickness). The oven temperature was programmed from 40 to 205 °C at a rate of 7 °C/min and held for 15 min at the final temperature.

Table I. Major Compounds Isolated and Identified from UHT Milk Using Activated Carbon 60/80 Mesh (0.25 g) Combined with C_{18} Reversed-Phase Silica (0.25 g)

peak	compound	identification
11	1-hexanoic acid	positive
15	2-heptanone	positive
16	1-ethyl-2,5-pyrrolidinedione	tentative
18	1-octanoic acid	positive
21	γ-octalactone	positive
22	δ-octalactone	positive
23	1-decanoic acid	positive
25	γ-decalactone	positive
26	1-dodecanoic acid	positive
27	δ-nonalactone	tentative
28	δ-decalactone	positive
30	δ-undecalactone	tentative
31	γ-dodecalactone	positive
33	δ-dodecalactone	positive
34	1-tetradecanoic acid	tentative
35	δ-tetradecalactone	tentative
36	decanoic acid, 2-hydroxy-1-(hydroxymethyl) ester	tentative
37	2-methyl-4-methylthiazole	tentative
38	dodecanoic acid, 2,3-dihydroxypropyl ester	tentative

1 h with the combined solid phase. Among the 38 major compounds isolated from UHT milk samples (Figure 3), we attempted to identify 19 major compounds by GC and GC–MS. The compounds identified are listed in Table I. These include one ketone, five free fatty acids, nine lactones, two esters, and two heterocyclics. Identities of the compounds were first attempted for unknown spectra by probability-based matching (PBM) using the computer library search. On the basis of the best fit for the spectra, standard compounds were injected into the GC under the same temperature programming, and their retention times were compared. The compounds matched by GC retention times were 1-hexanoic acid, 2-heptanone, 1-octanoic acid, γ-octalactone, δ-octalactone, 1-decanoic acid, γ-decalactone, δ-decalactone, 1-dodecanoic acid, γ-dodecalactone, and δ-dodecalactone. The spectra of these standard compounds were compared and found to match well with those of the compounds isolated from UHT milk samples. The remaining eight compounds in Table I [1-ethyl-2,5-pyrrolidinedione; decanoic acid, 2-hydroxy-1-(hydroxymethyl) ester; δ-undecalactone; 1-tetradecanoic acid; 2-methyl-4-methylthiazole; δ-nonalactone; δ-tetradecalactone; and dodecanoic acid, 2,3-dihydroxypropyl ester] were tentatively identified by GC–MS spectra based on PBM. The literature indicated that 2-heptanone is a major volatile compound in fresh UHT milk, and its concentration increases as a function of storage time and temperature (Jeon et al., 1978). Takacs (1989) observed that he could remove 2-heptanone from stale UHT milk using a Sep-Pak C_{18} cartridge. Free fatty acids are known to be natural components of milk (Jenness, 1988), but their concentrations in UHT milk increase as a function of storage time (Takacs, 1989; Schmidt and Renner, 1978). Although we observed in preliminary studies that 1-decanoic acid could not be eluted with methylene chloride when the standard compound was dissolved in diethyl ether and directly loaded onto the activated carbon columns, it is interesting to note that the same compound from UHT milk was apparently eluted with methylene chloride from the combined solid phase. Lactones were reported to be present in UHT milk and believed to be heat-induced compounds (Scanlan et al., 1968). Badings and Neeter (1980) reported a number of lactones present in milk, six of which were associated with UHT milk samples. They are difficult to isolate by conventional methods because of their low volatility. Lactones in UHT milk were not observed with steam distillation and headspace analysis (Rerkrai et al., 1987) or with a large-scale steam-vacuum distillation (Jeon, 1976).

Table II. Comparison of Recoveries for the Direct Loading of 250 μg of Seven Lactone Mixtures to Activated Carbon and to a Combination of C_{18} Reversed-Phase Silica and Activated Carbon[a]

	act. carbon[b]		C_{18} + act. carbon[c]	
compound	mean	CV[d]	mean	CV[d]
γ-valerolactone	80.7	7.6	126.5	4.1
δ-valerolactone	76.0	8.8	40.0	3.2
ε-caprolactone	77.4	8.5	125.8	4.0
γ-octalactone	75.6	6.8	126.3	3.8
γ-decalactone	73.7	6.5	123.7	3.8
δ-decalactone	74.2	10.9	85.6	3.6
δ-dodecalactone	68.4	6.3	81.1	3.2

[a] Based on eight replicates and 1-μL injection from 1.0-mL concentrates of samples. [b] 0.5 g (60/80 mesh). [c] 0.25 g of each solid phase. [d] Coefficient of variation.

Table II summarizes a comparison of recoveries for seven lactone standards that were loaded directly onto columns of activated carbon alone or combined with C_{18} reversed-phase silica. Recoveries of the lactones with activated carbon ranged between 68.4 and 80.7% with coefficients of variation (CV) of 6.3–10.9%. However, the combined solid phase provided much higher recoveries than the activated carbon alone, ranging from 81.1 to 126.5%, except δ-valerolactone (40.0%). The CV were all low, ranging between 3 and 5%. Therefore, we assumed that the combined solid-phase material provided better recoveries than the activated carbon alone. We also assumed that the reproducibility for the combined solid phase was more consistent and acceptable. However, the explanation for a recovery as high as 126.5% with the combined solid phase is unclear. Examination of the spectra for the compound before and after passing through the solid-phase column indicated no apparent interference from other compounds. The reason for low recovery (40%) observed with δ-valerolactone is also unclear and requires further investigations. As shown in Table III, the recoveries of the seven lactones added to pasteurized milk appeared to be good with the combined solid-phase material. We observed that,

Table III. Effect of the Amounts of Standard Lactones Added to 50 mL of Pasteurized Milk on Their Recoveries through a Combination of Activated Carbon 60/80 Mesh and C_{18} Reversed-Phase Silica

compound	lactone standards,[a] ppb			
	30	50	70	90
γ-valerolactone	–[b]	34.6	29.1	40.8
δ-valerolactone	–	–	–	–
ε-caprolactone	–	12.9	18.6	31.8
γ-octalactone	35.9	64.3	71.7	105.4
γ-decalactone	44.3	60.8	76.6	147.2
δ-decalactone	40.6	54.7	59.5	91.0
δ-dodecalactone	34.9	59.8	86.5	100.6

[a] Recoveries based on eight replicates with 3-μL injection from 0.5-mL concentrates. [b] No compounds detected.

Table IV. Reproducibility of Some Major Flavor Compounds That Were Isolated from 100 mL of Two UHT Milk Samples by a Combination of Activated Carbon (60/80 Mesh) and C_{18} Reversed-Phase Silica

compound	UHT milk 1 (indirect UHT)		UHT milk 2 (direct UHT)	
	peak area[a]	CV[b]	peak area[a]	CV[b]
2-heptanone	636.0	4.7	192.6	2.2
1-octanoic acid	2374.5	1.6	807.4	3.6
γ-octalactone	225.5	2.1	82.1	0.8
1-decanoic acid	2021.9	4.9	1297.9	2.4
γ-decalactone	330.0	2.3	180.6	1.6
1-dodecanoic acid	1751.4	4.7	103.3	5.1
δ-decalactone	129.5	4.9	102.2	6.7
δ-dodecalactone	355.7	7.5	729.7	7.6
1-ethyl-2,5-pyrrolidinedione[c]	20.9	12.9	18.7	9.6
decanoic acid, 2-hydroxy-1-(hydroxymethyl) ester[c]	153.7	2.2	898.0	0.7
dodecanoic acid, 2,3-dihydroxypropyl ester[c]	364.3	1.3	449.8	0.2

[a] Based on eight replicates. [b] Coefficient of variation. [c] Tentatively identified.

as the concentrations of the added standards increased from 30 to 90 ppb, the percent recoveries for octa- and dodecalactones approached full recovery. In addition, the increases were approximately linear within the limit of the concentrations tested for each lactone standard. However, the recoveries for lactones were less than 50% when the concentration of added lactones was below 50 ppb.

Table IV shows reproducibility of some major compounds that were isolated and identified from 100 mL of two different UHT milk samples. Results indicated that the combined solid-phase method yielded relatively low CV. They were less than 5% for UHT milk sample 1 except for δ-dodecalactone and 1-ethyl-2,5-pyrrolidinedione (7.5 and 12.9%, respectively). The CV for UHT milk sample 2 were less than 5% for seven compounds, whereas four others had CV between 5.1 and 9.6%. The overall results indicated that the CV for the majority of the compounds were less than 5% with eight replicates from two different UHT milk samples. Therefore, we considered that the reproducibility was within the acceptable range. Another important observation was that the peak areas of the compounds isolated from UHT milk sample 1 were generally much larger than those of the corresponding compounds isolated from UHT milk sample 2. UHT milk sample 1 was indirectly processed, whereas UHT milk sample 2 was directly steam injected. It is interesting that the indirect UHT milk (sample 1) contained much higher concentrations of volatile compounds than the direct UHT milk (sample 2), although the difference may not necessarily be related to the processing methods.

CONCLUSION

The results obtained from this study indicated that preconditioning activated carbon for SPE columns with methanol improved the retention of several compounds including lactones. Treating the solid-phase materials in the extraction column with *n*-hexane was efficient in removing residual milk fat and other contaminants, and methylene chloride appeared to be the best eluting solvent. We determined that washing the SPE column with organic-free water is essential for removing water-soluble materials from the column. The combination of C_{18} reversed-phase silica and activated carbon 60/80 mesh was found to be the most appropriate solid-phase material for isolating neutral volatiles, particularly less volatile compounds such as lactones, from UHT milk. It is well-known that sulfurous flavor in UHT milk dissipates rapidly within a few weeks of processing, but cooked flavor remains in the milk. Therefore, we suspect that relatively nonvolatile compounds such as lactones may be involved in UHT milk flavor. This SPE method may be helpful in the isolation of these heat-induced compounds in UHT milk. Furthermore, the combined SPE method can be successfully used as a quantitative tool according to the CV we observed. In addition, the method is relatively rapid (30–60 min), requires a small volume of samples (50–100 mL), and possesses the ability to isolate compounds present at the parts per billion level.

ACKNOWLEDGMENT

This study was supported in part by the Government of the Republic of Ivory Coast in collaboration with the Institute of International Education (IIE). K.C. is a recipient of a Fulbright Scholarship through IIE.

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Zief, M.; Kiser, R. An overview of solid phase extraction. *Am. Lab.* **1990**, *22* (1), 70–83.

Received for review August 8, 1991. Accepted January 29, 1992.

Registry No. 1-Hexanoic acid, 142-62-1; 2-heptanone, 110-43-0; 1-ethyl-2,5-pyrrolidinedione, 2314-78-5; 1-octanoic acid, 124-07-2; γ-octalactone, 104-50-7; δ-octalactone, 698-76-0; 1-decanoic acid, 334-48-5; γ-decalactone, 706-14-9; 1-dodecanoic acid, 143-07-7; δ-decalactone, 705-86-2; δ-nonalactone, 3301-94-8; δ-undecalactone, 710-04-3; γ-dodecalactone, 2305-05-7; δ-dodecalactone, 713-95-1; 1-tetradecanoic acid, 544-63-8; δ-tetradecalactone, 2721-22-4; 2-methyl-4-methylthiazole, 541-58-2; dodecanoic acid, 2,3-dihydroxypropyl ester, 142-18-7.