Quantitative Determination of Sodium Lauroyl Sarcosinate by Gas Chromatography

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A simple and rapid procedure is described for the isolation, silylation and wide-bore capillary gas chromatographic quantitation of sodium lauroyl sarcosinate in personal care products. The sample is dissolved in acidified dimethylformamide to simultaneously acidify/extract the lauroyl sarcosine; an aliquot is then derivatized with *bis* trimethylsilyltrifluoroacetamide and quantitated by wide-bore capillary gas chromatography with flame ionization detection.

KEY WORDS: Acidification, analysis, capillary GC, derivatization, extraction, lauroyl sarcosine, personal care products, quantitation, sodium lauroyl sarcosinate.

Sodium lauroyl sarcosinate is an anionic surfactant found in various personal care products. However, the analysis for this surfactant has not been straightforward. Although other analytical methods, including high-performance liquid chromatography (1,2) and thermogravimetric analysis (3–5), have been reported in the literature for raw materials and certain mixtures of surfactants, this gas chromatographic method combines specificity, quantitation and easy sample preparation for the analysis of this surfactant in commercial products.

Advances in capillary gas chromatography have greatly enhanced capabilities for resolving complex mixtures. Frequently, the resolving capacity of capillary columns can eliminate the need for extensive sample preparations or clean-ups. In our analytical laboratory we routinely analyze products for ingredients such as glycols (propylene, butylene, diethylene and hexylene), triclosan, sorbitol, fatty acids, fatty alcohols and their ethoxylates, fatty acid diethanolamides, triethanolamine, chloroxylenol, and methyl and propyl paraben by using adaptations of our previously published procedure for determining glycerin in soap bars (6). In this paper we report on a further adaptation in which one-step acidification/dissolution is accomplished by means of acidified dimethylformamide followed by silyl derivatization and wide-bore capillary gas chromatography (GC) analysis is used to quantitate sodium lauroyl sarcosinate in personal care products.

EXPERIMENTAL PROCEDURES

Instruments and conditions. Analyses were performed on a Hewlett-Packard Model 5890A gas chromatograph system (Palo Alto, CA), which included a flame ionization detector, model 7671A autosampler and model 3388A integrator. The column was a 5 m × 0.53 mm i.d. fused silica capillary column coated with methyl silicone fluid (crosslinked) with 2.65-μm film thickness, supplied by Hewlett-Packard (half of #19095Z-121). This column was installed in a packed-column style injection port with a Hewlett-Packard adapter (#19244-80540) and connected to a flame ionization detector without any make-up gas. The carrier gas was helium at about 7 mL/min. The column temperature was held at 150°C for 2 min, then programmed at a rate of 5.0°C/min to a final temperature of 280°C, where it was held constant for 5 min. The inlet temperature was 275°C, and the detector temperature was 290°C. A threshold value of 6 and an attenuation of 2^8 were employed. With these GC conditions, the retention time for silylated lauroyl sarcosine was about 13.5 min.

Gas chromatography-mass spectrometry (GC-MS) analyses were performed on a Hewlett-Packard Model 5890 Series II gas chromatograph interfaced with a Hewlett-Packard Model 5971A mass selective detector. The column was a 12 m × 0.22 mm i.d. fused silica capillary column coated with methyl silicone fluid (crosslinked) with 0.33-μm film thickness, supplied by Hewlett-Packard (#19091-60312). The carrier gas was helium at a column head pressure of 5 psi. The column temperature was held at 150°C for 2 min, then programmed at a rate of 10.0°C/min to a final temperature of 270°C, where it was held constant for 5 min. The inlet temperature was 275°C, and the transfer line temperature was 280°C.

Reagents and solutions. ACS reagent-grade DMF (*N,N*-dimethylformamide) and HCl (37% hydrochloric acid) were obtained from Mallinckrodt Specialty Chemicals Co. (St. Louis, MO). Acid-DMF reagent was prepared by adding 2.5 mL HCl to 500 mL DMF and mixing. BSTFA (*bis*-trimethylsilytrifluoroacetamide + 1% trichloromethylsilane) was obtained from Regis Chemical Co. (Chicago, IL). Sodium lauroyl sarcosinate standard (95% purity) was supplied by W. R. Grace (Hamposyl L-95) (South Pittsburgh, TN). To prepare the sodium lauroyl sarcosinate standard solution, about 0.3 g sodium lauroyl sarcosinate standard was accurately weighed (±0.0001 g) into a 200-mL volumetric flask, then dissolved and diluted to volume with acid-DMF. A 250-μL portion was transferred to an autosampler vial where it was mixed with 500 μL BSTFA.

Assay procedure and calculation. Five grams of sample (liquid sample or chopped bar soap) was weighed (±0.01 g) into a 200-mL volumetric flask. Acid-DMF was added to dissolve the sample and to dilute to volume (a magnetic stir bar and stirrer should be used for bar soap samples). A portion of the solution was filtered (if necessary) by gravity through Whatman No. 41 paper into a beaker. Filtrate (250 μL) was transferred to an autosampler vial and mixed with 500 μL BSTFA. A 1-μL sample was then injected into the GC and compared to 1-μL injections of the standard sodium lauroyl sarcosinate solution. Each was injected three times. Routine external-standard calculations are used to determine percent sodium lauroyl sarcosinate:

$$\%\ \text{sodium lauroyl sarcosinate} = \frac{\text{Area}_{\text{sample}}}{\text{Area}_{\text{standard}}} \times \frac{\text{Concentration}_{\text{standard}}}{\text{Concentration}_{\text{sample}}} \times 100 \qquad [1]$$

where $\text{Area}_{\text{sample}}$ and $\text{Area}_{\text{standard}}$ are the average peak areas of derivatized lauroyl sarcosine in the sample and standard, and the concentration terms are the final concentrations.

RESULTS AND DISCUSSION

Method development. In our laboratory, there was a need for a simple, straightforward procedure to determine sodium lauroyl sarcosinate in personal care products. After reviewing published methods, we saw the opportunity to develop a new method that would require less operator time, be amenable to automated analysis and provide reliable quantitation. Since we had considerable experience in making trimethylsilyl derivatives of various carboxylic acids and analyzing such by GC, the initial plan was to acidify an aqueous solution containing sodium lauroyl sarcosinate to produce lauroyl sarcosine, extract, then derivatize with BSTFA. However, this was not tried because a simple one-step acidification/extraction with acid-DMF was developed instead; a sample could be dissolved in acid-DMF, mixed with BSTFA, then injected into the GC. Because only one major peak was observed from the standard, this procedure looked promising for quantitation. GC-MS analysis confirmed the major peak from the standard to be derivatized lauroyl sarcosine by showing the expected MW-15 ion (characteristic loss of a methyl group from trimethylsilyl derivatives) at 328 (Fig. 1) and absence of other fatty acid sarcosine peaks.

GC-MS analyses of commercially available bar soap and liquid soap samples showed that the peak of interest was composed exclusively of derivatized lauroyl sarcosine.

Wide-bore capillary GC with flame ionization detection was used for this method because it provided reliable quantitation and resolution from other materials that might be present in most personal care products. Typical wide-bore capillary GC curves for derivatized lauroyl sarcosine standard and a commercially available personal care product are shown in Figure 2.

Slight modifications in this method have also been used in our laboratory to analyze for triethanolamine lauroyl sarcosinate and salts of citric acid and benzoic acid in a similar manner, but these applications have not been studied thoroughly.

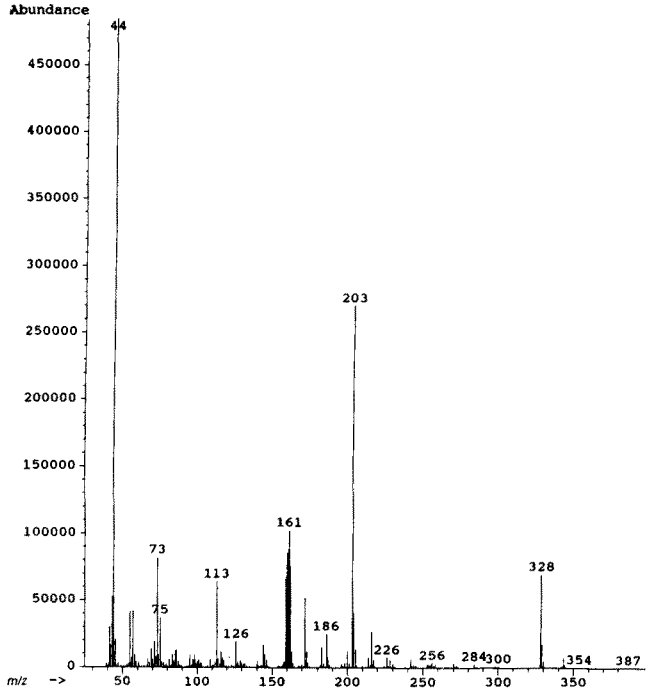

FIG. 1. Mass spectrum of trimethylsilyl derivative of lauroyl sarcosine.

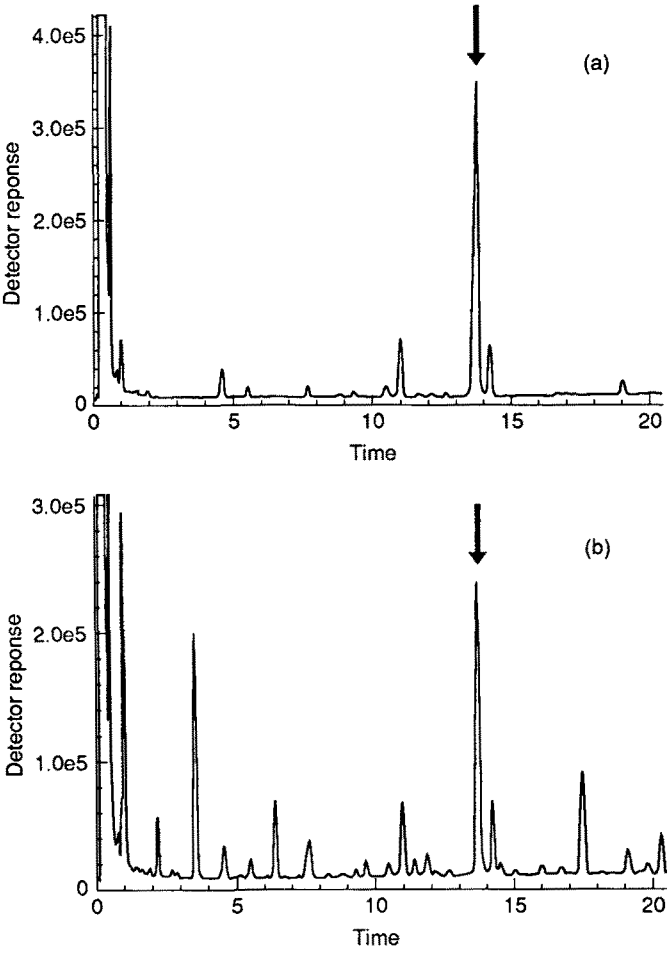

FIG. 2. Capillary gas chromatograms of: (a) silylated standard lauroyl sarcosine; (b) silylated extract of commercially available liquid soap product.

Accuracy and precision. Methodology validation studies were conducted in which personal care products of various compositions were analyzed by this method, and results were compared to target values. Also, a commercial liquid soap product without sodium lauroyl sarcosinate was spiked at a realistic use level of 5.19% sodium lauroyl sarcosinate, then analyzed; levels of 5.40 ± 0.13% sodium lauroyl sarcosinate (corresponding to recoveries of 104.1 ± 2.5%) were obtained. Results obtained from analyzing

TABLE 1

Assay of Commercially Available Products

Type of product	% Sodium lauroyl sarcosinate
Liquid soap 1	6.70
	6.86
	6.79
Liquid soap 2	2.80
	2.78
Bar soap	12.9
	12.5

commercially available personal care products are detailed in Table 1.

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