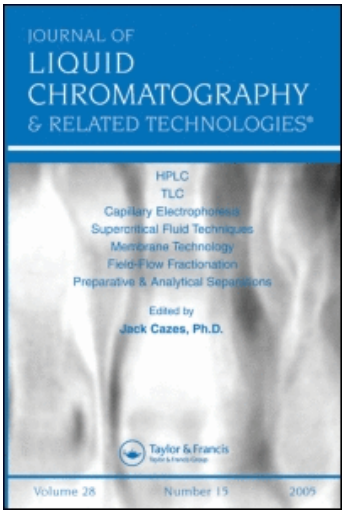

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Ion Chromatographic Determination of Sodium Isethionate

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ION CHROMATOGRAPHIC DETERMINATION OF SODIUM ISETHIONATE

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ABSTRACT

Single column anion chromatography with conductivity detection has been used for the determination of sodium isethionate. A mobile phase of aqueous phthalic acid - methanol (pH 2.5) allows for the separation of isethionate from chloride and alkyl isethionate ester surfactants. Commercially available samples of sodium isethionate and alkyl esters were analyzed by the described procedure.

INTRODUCTION

The alkyl isethionate esters (i.e. GAF's Igepon A™) constitute a class of anionic surfactants used in a variety of detergent and cosmetic applications where good foaming, lathering, and dispersing properties are needed. Typically, a fatty acid (e.g. tallow or coconut) is esterified with sodium

isethionate ($HO-CH_2CH_2-SO_3Na$) to produce the final product (1). The assay of sodium isethionate in the absence of surfactant can be accomplished by an acid-base titration after cation exchange and removal of interfering inorganic anions by precipitation with barium hydroxide. This method is extremely time-consuming and prone to interference by chloride ion. To our knowledge, there are no suitable procedures for the determination of sodium isethionate in the presence of surfactant based on a wet chemical approach.

We wish to report the development of a single column ion chromatographic procedure for the determination of sodium isethionate. Isethionate is selectively retained on a silica-based anion exchange column using a methanol - 5mM phthalic acid mobile phase. Quantitation is accomplished by conductivity detection and comparison to external standards. The method is selective for sodium isethionate in the presence of chloride and alkyl isethionate surfactant.

MATERIALS

Apparatus

A modular HPLC system comprised of a Rainin Rabbit HP pump, pulse dampener, Rheodyne 7125 injector, 100 microliter sample loop, and Bio-Rad CM-8 conductivity detector was used. Peak heights and retention times were determined using an Apple IIe microcomputer equipped with hardware/software obtained from the

Dynamic Solutions Division of Millipore Corporation. A Vydac 302 IC silica-based anion exchange column (4.6 mm x 250 mm) and 50 mm precolumn (packed with pellicular Vydac 302 IC anion exchange material) were used for analytical separations.

Reagents

HPLC grade water (J.T. Baker) was used for all reagent solutions. The HPLC mobile phase consisted of 150 mL methanol (EM Science) and 250 mL 20 mM aqueous phthalic acid (Aldrich Chemical Company) diluted to 1 L with HPLC grade water. The solution was filtered through 0.45 um Nylon-66 membranes and degassed by helium sparge for 15 minutes before use. Barium hydroxide octahydrate (98+ %, Aldrich), 0.1 N sodium hydroxide (VWR Scientific) and AG 50W-X8 cation exchange resin (hydrogen form, Bio-Rad Laboratories) were used for the assay of sodium isethionate standard (98%, Aldrich) as well as commercially available materials. Igepon AC-78 samples were donated by GAF Chemicals Corporation.

METHODS

Chromatographic Procedure

The following conditions were used for the analysis of sample and standard solutions:

(a) column: Vydac 302 IC, 4.6 mm x 250 mm
guard column filled with Vydac 302 IC pellicular packing, 3 mm x 50 mm

(b) mobile phase: 150 mL methanol + 250 mL 20 mM phthalic acid diluted to 1 L with water

(c) flow rate: 3 mL/min @ 1500 psi

(d) injection volume: 100 uL

(e) detection: conductivity, 10 uS full scale, 1 volt output

The system was allowed to equilibrate for 20 minutes at the specified conditions before conducting the analysis. Samples (isethionate and/or surfactant) were dissolved in water, filtered through 0.45 um Nylon-66 filters, and directly injected.

Titration Procedure

Sodium isethionate samples were dissolved in 100 mL of HPLC grade water and treated with 4 mL of 0.2 M barium hydroxide aqueous solution. After stirring and heating at 60°C for 1 h, the solutions were allowed to stand over a steam bath for 24 h. The precipitate was removed by filtration through medium porosity glass filters. The filtrates were then passed through 50 mL of wet cation exchange resin (hydrogen form) and titrated potentiometrically with 0.2 N sodium hydroxide.

RESULTS AND DISCUSSION

The approach to separation in this study was to use anion exchange to allow for retention of the alkyl sulfonate (isethionate) in the presence of counter ions and surfactant.

An organic acid at low pH was used to minimize ionization of isethionate, thus allowing for its resolution from other species (eg. chloride, surfactant). Methanol was added to minimize surfactant retention while simultaneously improving the isethionate peak shape. Chromatograms of the sodium isethionate standard and Igepon AC-78 are shown in Figures 1 and 2, respectively. Under the experimental conditions, isethionate elutes in 7.6 minutes while the surfactant elutes at the column void volume (1.2 minutes). The use of phthalic acid also allows for rapid elution of the "water dip" (2.5 minutes) which eliminated possible interference or carryover to subsequent injections. Chloride was found to elute at 9.3 minutes under the chromatographic conditions. As with typical phthalate - based anion exchange systems (2), the retention of anions can be reduced by using higher pH and/or ionic strength. However, it was found that higher pH resulted in co-elution of isethionate and chloride as well as tailing of the surfactant.

Response linearity was evaluated in the 5 ppm to 100 ppm range. A plot of peak height versus isethionate concentration (in ppm) (Figure 3) was found to obey the following equation:

$$H\ (uS) = (2.45 \pm 0.003\ uS\text{-}ppm^{-1})C - 0.50 \pm 0.16\ uS$$

with a correlation coefficient of 0.9997 and standard error of estimate of 0.29. The detection limit, defined as the isethionate concentration yielding a signal to noise ratio of 2, is 1 ppm (absolute). Depending on the sample size and injection

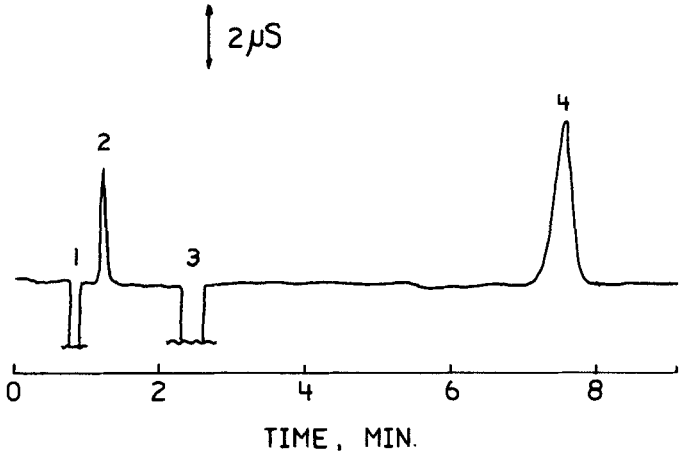

FIGURE 1 14 ppm sodium isethionate standard solution in distilled water. 1 = void peak; 2 = counter ion; 3 = water dip; 4 = isethionate.

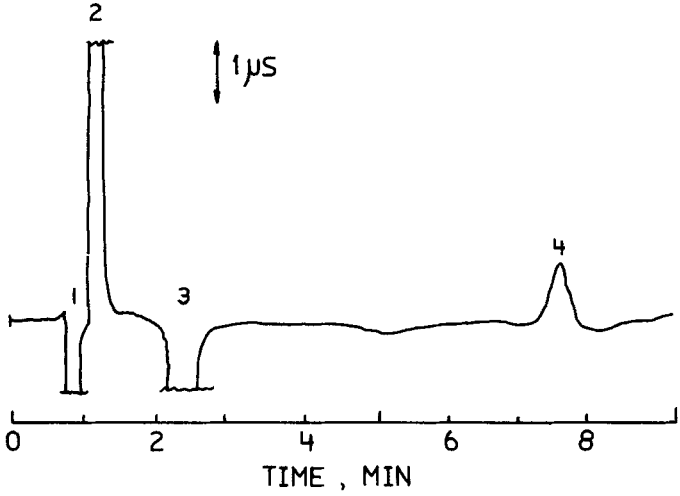

FIGURE 2 500 ppm Igepon AC-78 (coconut acid ester of sodium isethionate) in distilled water. 1 = void peak; 2 = surfactant; 3 = water dip; 4 = iesthionate.

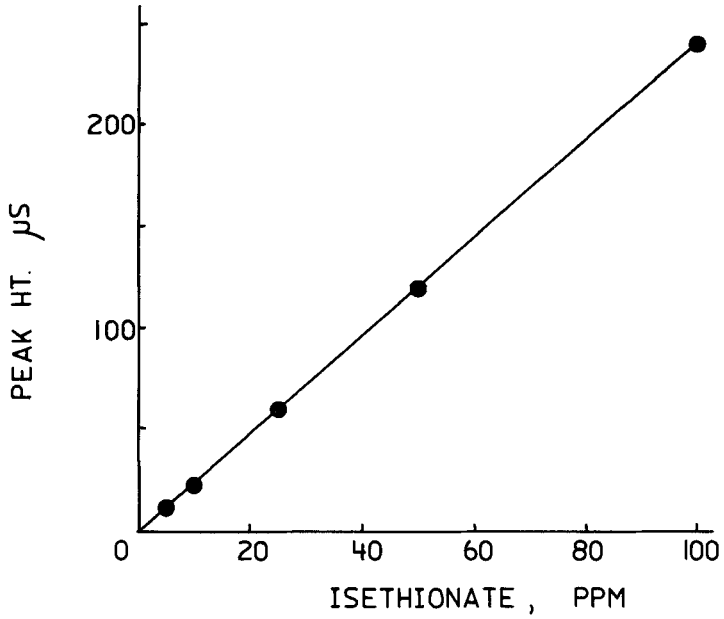

FIGURE 3 Calibration curve of peak height versus isethionate concentration.

volume, one can detect a minimum of 20 ppm isethionate in the sample.

Various samples of sodium isethionate from two different manufacturers were analyzed by the proposed chromatographic procedure as well as a titration method (see procedure section). The results of these analyses are shown in Table 1. Excellent agreement was observed between the two methods. It should be noted that improved precision is obtained with the proposed procedure compared to the titration procedure. For multiple

TABLE 1

Assay of Sodium Isethionate by Ion Chromatography and Titration Procedures

Manufacturer	%, proposed method[a]	%, titration[b]
A	92.35 ± 0.52	92.00 ± 1.20
A	95.50 ± 0.58	94.85 ± 1.63
A	50.05 ± 0.40	49.89 ± 1.01
A	46.53 ± 0.38	47.02 ± 1.31
B	52.50 ± 0.21	53.05 ± 1.81

[a]average of six determinations ± s
[b]average of four determinations ± s

(n = 10) injections of a single sample solution (25 ppm), the coefficient of variation was found to be 0.9%.

The described method is easily adapted for the assay of sodium isethionate in the presence of alkyl isethionate surfactant. The surfactant is simply dissolved in distilled water, filtered, and directly injected. Various Igepon A™ - type samples were analyzed by this approach (50 mg/100 mL) and yielded the results shown in Table 2. Again, good reproducibility is obtained. Recovery was evaluated by fortifying surfactant samples with isethionate standard and analyzing as previously described. As shown in Table 3, quantitative recovery was obtained at the two recovery levels.

As esterification is a reversible process in the presence of acid, there were concerns that the alkyl isethionate ester might hydrolyze and generate isethionate during the chromatographic

TABLE 2

Assay of Sodium Isethionate in Igepon AC-78™

Sample	% isethionate ± s
1	3.56 ± 0.06
2	5.40 ± 0.10
3	2.38 ± 0.02
4	1.93 ± 0.05
5	2.80 ± 0.13
6	1.83 ± 0.15

TABLE 3

Recovery of Sodium Isethionate from Igepon AC-78

Sample	ug added	ug found	recovery, %
1	43.4	43.8	101
	43.4	43.5	100
2	21.2	20.9	99
	21.2	21.0	99

separation. In order to evaluate the hydrolytic stability of the surfactant, Igepon AC-78 was dissolved in the mobile phase and injected every 15 minutes for up to 3 hours. During this time, the measured isethionate level did not change. Samples injected after contact with the mobile phase at 22°C for 24 hours did show a doubling of the apparent isethionate level. This experiment confirmed the stability of the surfactant during the typical analysis time frame. Under normal conditions, hydrolysis is negligible, especially if distilled water is used for sample preparation.

CONCLUSIONS

The use of anion chromatography with conductivity detection allows for the rapid and precise assay of sodium isethionate. In addition, the analyte can be measured in the presence of alkyl isethionate esters which is not possible by any other known approach.

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