Analysis of Sodium Isethionate in Soap and Lye Process Streams by Suppressed Ion Chromatography

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Suppressed ion chromatography has been used to determine sodium isethionate in various soap production process streams at concentrations of 0.1–1.0%. The method was validated by spiking placebo soap and lye process stream samples. Overall recoveries in soap were 98.5 ± 2.3% (n = 24). Recoveries in lye were 98.4 ± 2.8% (n = 24).

KEY WORDS: **Ion chromatography, sodium isethionate.**

A nonsuppressed ion chromatography (IC) method, reported by Ianniello (1), has been used in our laboratory to analyze for sodium isethionate in cocoyl isethionate raw materials and in finished soap products. However, in our experience, the method is not suitable for the analysis of isethionate in lye process streams due to high chloride concentrations.

Comparisons of suppressed and nonsuppressed IC have been published elsewhere (2,3). A potential advantage of suppressed IC is better sensitivity. Because it will reduce the sample size required, and therefore the degree of chloride overloading, the latter approach was investigated as a means of determining isethionate in process stream samples.

EXPERIMENTAL PROCEDURES

Sample preparation. Spiked placebos containing 0.1, 0.2, 0.5 and 1.0% sodium isethionate were prepared by weighing 1.0-g samples of soap or lye placebo (obtained in-house) into 100-mL volumetric flasks and spiking with 1, 2, 5 or 10 mL of 1 mg/mL sodium isethionate (Aldrich Chemical Co., Milwaukee, WI) in MilliQ water (Millipore, Milford, MA). The samples were then diluted to the mark with 50:50 (vol/vol) MilliQ water/3A alcohol (Quantum Chemical Corporation, Cincinnati, OH) and stirred to dissolve. Working samples were obtained by diluting 5 mL of the resultant to 25 mL with MilliQ water. Standards were prepared by successive dilution of sodium isethionate with water to yield a concentration of 0.01 mg/mL.

Chromatography. Chromatographic analyses were performed with a Dionex IonPac AS10 analytical column (250 × 4 mm) fitted with an AG10 guard column (Dionex Corporation, Sunnyvale, CA). The eluent was 100 mM sodium hydroxide (J.T. Baker, Phillipsburg, NJ) at 1 mL/min. Suppression was achieved with a Dionex Anion Micromembrane Suppressor and 50 mM sulfuric acid at

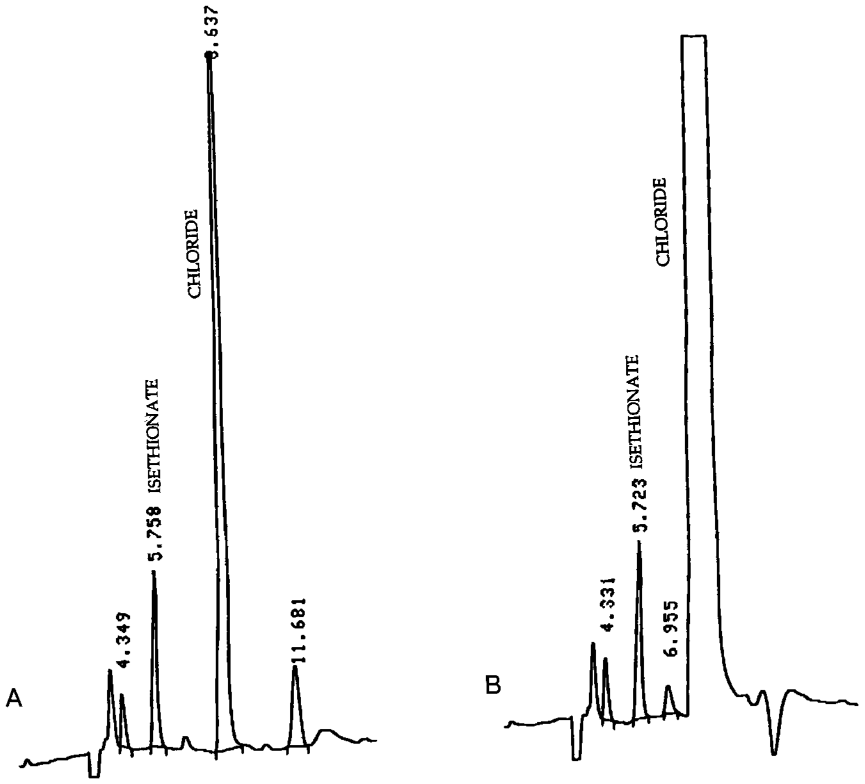

FIG. 1. Chromatograms of 0.2% sodium isethionate in (A) soap and (B) lye process streams. For conditions see text.

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TABLE 1

Sodium Isethionate Recoveries (%)

Sample	Day 1	Day 2	Day 3	Average (±)
in Soap (%)				
0.1	100.7, 98.7	99.6, 99.0	94.2, 91.6	97.3 ± 3.6
0.2	96.9, 98.2	99.0, 99.3	95.2, 97.8	97.7 ± 1.5
0.5	98.8, 96.0	101.4, 102.1	99.0, 99.3	99.3 ± 2.1
1.0	98.7, 99.4	100.2, 100.3	99.3, 99.7	99.6 ± 0.6
in Lye (%)				
0.1	95.1, 99.1	95.9, 97.7	91.2, 92.3	95.2 ± 3.0
0.2	102.0, 99.5	99.9, 98.8	94.6, 95.9	98.5 ± 2.7
0.5	99.8, 99.5	101.6, 99.7	99.0, 98.1	99.6 ± 1.2
1.0	100.4, 98.8	101.4, 101.3	98.9, 99.1	100.2 ± 1.1

6–8 mL/min. The chromatographic hardware consisted of a Waters ActION pump/controller (Waters Chromatography, Milford, MA) and a Waters Model 431 conductivity detector. A Waters WISP 710B autoinjector was used to inject 50-μL samples. Quantitation was performed with a Shimadzu CR501 Chromatopac integrator (Kyoto, Japan).

RESULTS AND DISCUSSION

Initial efforts directed toward developing a method for the analysis of sodium isethionate in both soap and lye process streams were attempted with the procedure reported previously (1). Good results were obtained for soap samples containing from 0.1 to 1.0% sodium isethionate when 100-μL samples of 2 g soap/100 mL were injected. This loading corresponds to 20 ppm (absolute) sodium isethionate at the lowest level of interest, which is approximately 20 times the limit of detection. Lye samples, however, contain a substantial amount of chloride (>5% wt/vol), which overloads the column to the point where isethionate and chloride coelute. Due the limit of detection, this problem cannot be overcome by reducing the sample size.

As an alternative approach, the use of suppressed IC was investigated. An IonPac AS10 column was chosen, based on its high capacity (manufacturer specifies 170 μeq for a 250 × 4 mm column). To further minimize chloride overload, absolute sample loadings were reduced by a factor of 20. The chromatograms obtained for soap and lye samples containing 0.2% sodium isethionate are shown in Figure 1. Resolution between isethionate and chloride, as well as sensitivity, is adequate, and analysis times are less than 15 min.

To determine whether the method also provided adequate analytical reproducibility, spiked placebos were analyzed in duplicate on each of three days, at levels ranging from 0.1 to 1.0% in each matrix. The results are shown in Table 1. Recoveries ranged from 91.6 to 101.4% in soap, to yield an overall average recovery of 98.5 ± 2.3% (n = 24). In lye, recoveries ranged from 91.2 to 102.0% for an overall average recovery of 98.4 ± 2.8% (n = 24). Recoveries by level ranged from 95.2 to 100.2% (n = 6 for each level), indicating that the method is linear over the concentration range studied.

The method has been used to analyze for sodium isethionate in various production streams to determine required lye purge frequencies and to quantitate sodium isethionate levels in finished soap products.

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