S & D SHORT COMMUNICATION

The Determination of Neutral Oil in Detergent Products by Solid Phase Extraction

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A column extraction technique is described for the determination of neutral oil content of sulfated or sulfonated detergent products. The advantages of this over the usual liquid/liquid extraction methods are the complete elimination of emulsion problems and the reduced time required to complete the analysis.

The measurement of neutral oil content is of considerable importance in the manufacture of sulfated or sulfonated products such as alkene sulfonates, lauryl ether sulfate, triethanolamine lauryl sulfate, ammonium lauryl sulfate, and alkyl benzene sulfonates.

The method generally employed is the ASTM procedure (1) or variations of it (2,3). About 1.5 to 2 hr are required for these methods. A more rapid method for production control was developed using a diatomaceous earth column technique. In this procedure, sulfated or sulfonated product, dissolved in 1:1 ethanol:water, is mixed with diatomaceous earth and packed into a glass column. The neutral oil is eluted preferentially with petroleum ether or dichloromethane/petroleum ether solution. The eluate is evaporated to dryness under vacuum and the neutral oil determined gravimetrically.

EXPERIMENTAL

The reagents used were the following: Celite 545—Johns Manville. Use as received; dichloromethane—reagent grade; petroleum ether—reagent grade b.p. 30-60°C; elution solvent—7% dichloromethane in petroleum ether, v/v; 1:1 ethanol:water—commercial alcohol denatured with methanol is satisfactory. Mix equal volumes of ethanol and distilled water.

The sample (approximately 1 g, accurate to 0.01 g) was weighed into a 100 ml beaker. Ethanol:water (2 ml) was added and the surfactant dispersed to give a clear solution. Celite 545 (4 g) was added and mixed well with a spatula to obtain a homogeneous, semi-dry solid. The celite mixture was loaded into a 19 × 400 mm glass column fitted with a coarse sintered disc and Teflon stopcock. A bed height of 42—44 mm was obtained by carefully packing the mixture with a flattened glass rod; overpacking of the column resulted in excessively slow elution rates. A tared 50 ml round-bottomed-flask (RBF) was placed to receive column effluent. Eluting solvent (about 5 ml), which had been used to rinse the sample beaker, was added to the column. When the solvent had entered the bed, a further 15 ml was added and collected in the RBF at a rate of about 2 ml per min. After all the solvent had entered the bed, the RBF was removed and the solvent evaporated carefully under water aspirator vacuum on a 60°C water bath until the solvent evaporated (about 5 min). The minimum pressure achieved was 12 mm of mercury. The flask was removed from the water bath, disconnected from the evaporator, and allowed to cool to room temperature for weighing to the nearest 0.1 mg.

TABLE 1

Neutral Oil in Sulfated and Sulfonated Product

		Percent neutral oil		
Sample		ASTM	Wolf (3)	Celite column
α-Olefin sulfonate	#1		0.86	0.86
α-Olefin sulfonate	#2		1.68	1.65
α-Olefin sulfonate	#3		0.85	0.81
α-Olefin sulfonate	#4		0.96	0.91
α-Olefin sulfonate	#5	0.86	0.91	0.87
Triethanolamine lauryl sulfate	#6	1.17	1.30	1.28
Ammonium lauryl sulfate	#7	1.19	1.30	1.25
Alkyl benzene sulfonate	#8		2.75	2.57[a]
Sodium lauryl ether sulfate (2 ethylene oxides)	#9		1.33	1.51
Sodium lauryl ether sulfate (3 ethylene oxides)	#10	NA[c]	NA[c]	1.77[b]

[a] The eluting solvent, 7% dichloromethane in 30-60°C petroleum ether, is not applicable to this product because it caused the breakdown of the water/ethanol/celite bond. In this case 30-60°C petroleum ether alone gives satisfactory results.

[b] When the product contains 3 moles of ethylene oxide, it is necessary to increase the volume of eluent to 35 ml in order to obtain 90% recovery of added neutral oil.

[c] NA—the method is not applicable.

$$\text{percent neutral oil} = \frac{\text{residue weight}}{\text{sample weight}} \times 100$$

The time required to complete a single analysis was 35–40 min.

RESULTS AND DISCUSSION

Preliminary experiments, in which water was used to dissolve the product for dispersion on the celite, showed that the neutral oils could not be eluted with petroleum ether or dichloromethane in petroleum ether. The use of 1:1 ethanol:water as described allows complete elution of the neutral oil without contamination with more polar material.

The method described here has been applied to α-olefin sulfonates, sodium lauryl sulfate, triethanolamine lauryl sulfate, ammonium lauryl sulfate and an alkyl benzene sulfonate. The results have been compared with those obtained using the method of Wolf and McPherson (3) and the standard ASTM method where applicable. The sample which contained two ethylene oxide units (#9) was difficult to analyze by the liquid/liquid extraction methods due to strong emulsion formation. An even stronger emulsion formed when three ethylene oxide units were present (#10) with the result that the sample could not be analyzed. The data in Table 1 show good agreement was obtained among the different methods. Six analyses of a single α-olefin sulfonate sample (#5) were performed to establish the precision of the method. The mean value was 0.869% (s=0.0102, n=6).

REFERENCES

1. *ASTM Book of Standards,* Part 30, Method D 1568.
2. *Official and Tentative Methods,* American Oil Chemists' Society, Dd 4-60, Champaign, Illinois.
3. Wolf, T., and B.P. McPherson, *J. Am. Oil Chem. Soc.* *54*:347 (1976).

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