L. Cohen, R. Vergara, A. Moreno and J. L. Berna, San Roque/Spain

Quantitative determination and separation of free oil components in Linear Alkylbenzene Sulfonic Acids

A liquid-solid extraction technique is described for the separation of LAB (linear alkylbenzene) and sulphones contained in a linear alkylbenzene sulfonic acid. This method is less time consuming and easier to carry out than the usual liquid-liquid extraction.

Es wird ein flüssig-fest Extraktionsverfahren zur Trennung von LAB (Linearem Alkylbenzol) und Sulfonen, die in einer Linearen Alkylbenzolsulfonsäure vorliegen, beschrieben. Diese Methode erfordert geringeren Zeitaufwand und ist leichter durchzuführen als die bisher übliche flüssig-flüssig Extraktion.

Introduction

The free oil components of a linear alkylbenzene sulfonic acid are mainly two: LAB, which is the unsulfonated matter, and sulphones resulting from oversulfonation. These two components have different polarities, so that a mixture of two selected solvents seems suitable for the extraction of these products. In the present method the solvent extraction mixture is petroleum ether/dichloromethane (85/15, v/v).

In this procedure the solid bed is a sepiolite having enough polarity to strongly adsorb the sulfonic acid while the other components are only weakly bend and can easily be extracted.

The conventional liquid-liquid extraction using hexane as solvent takes about 8 hours and needs a previous sample neutralization. The method described here requires less than half an hour and the sulfonic acid is used as such.

Table 1. Free Oil determination

Assay number	Liquid-liquid extraction	Liquid-solid extraction
1	2.7 %	2.4 %
2	2.4 %	2.3 %
3	2.5 %	2.6 %
4	2.6 %	2.3 %
5	2.4 %	2.4 %
6	2.4 %	2.3 %
7	2.7 %	2.6 %
8	2.5 %	2.7 %
Mean value (n = 8)	2.52 %	2.45 %
Standard Deviation	0.12	0.15

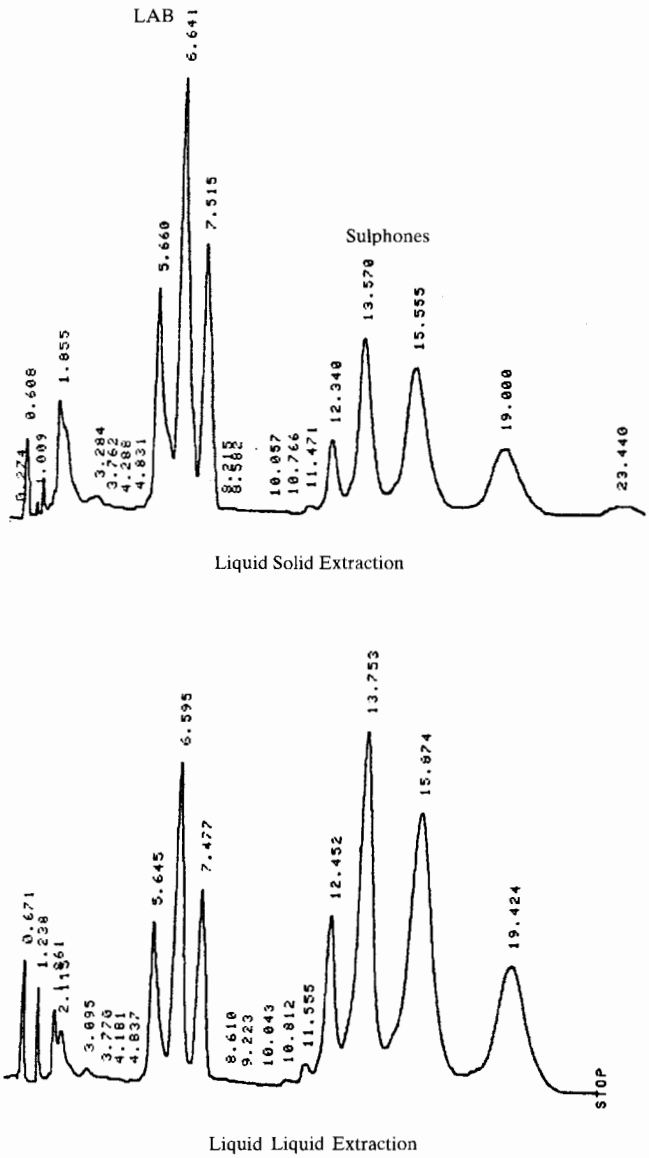


Fig. 1. Free oil HPLC analysis

Experimental

Reagents

Sepiolite, 40 mesh, from TOLSA – Apdo. 38017 – Madrid (Spain)

Dichloromethane, reagent grade

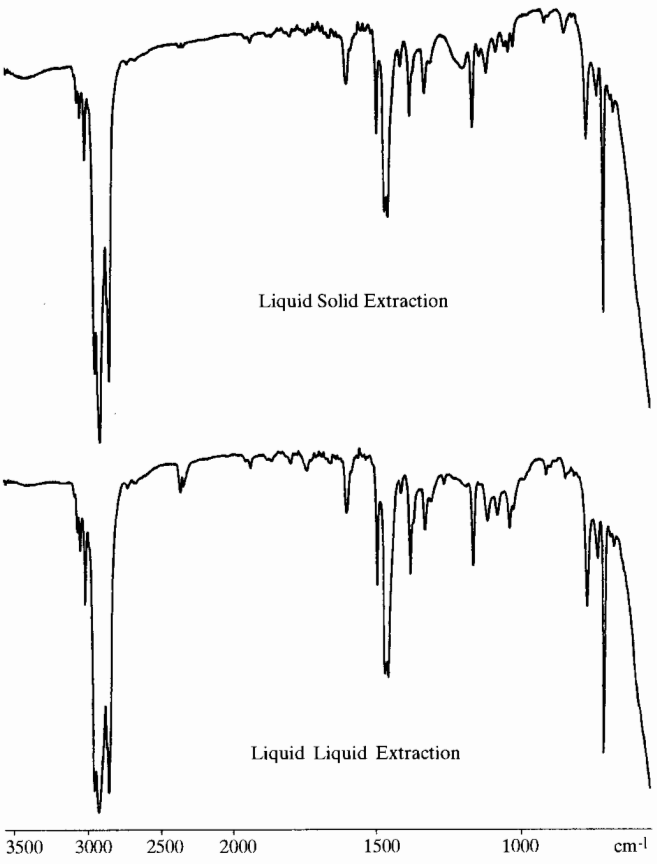


Fig. 2. Free oil IR spectra

Petroleum ether, reagent grade
Elution solvent, 15 % dichloromethane in petroleum ether, v/v

Procedure
1 g of sulfonic acid accurate to 0.01 g, is mixed with 6 g of sepiolite to obtain a semi-dry solid. The mixture is loaded into a glass column. The bed height obtained by carefully packing the mixture is approx. 5 cm.

60–100 ml of the elution solvent is added to the column at an extraction rate of approx. 2–3 ml per min.

After extraction the solvent is evaporated in a water bath at 80 °C and the residue is weighted to the nearest 0.001 g.

$$\text{Percent of free oil} = \frac{\text{Residue Weight}}{\text{Sample Weight}} \times 100$$

Results and discussion

Three methods were compared: the hexane extraction, the *Cross* method [1], and the present one.

The hexane extraction and the quantitative results of the present method for eight different free oil extractions of a single sulfonic acid are compared in Table 1, the results show good correspondence of the two methods.

In order to check the free oil composition on a qualitative basis, the eight extractions using each method were analyzed by HPLC according to [2]. As an example, Fig. 1 shows the results obtained in one extraction for each method.

Another way to confirm these results on a qualitative basis, were the IR spectra shown in Fig. 2.

As far as the *Cross* method is concerned, the free oil extraction level was not satisfactory because a small amount of LAS (linear alkylbenzene sodium sulfonate) was extracted as well.

References

1. *Cross, C. K.:* JAOCS, *67* (1990)
2. *Moreno, A.; Bravo, J.; Berna, J. L.:* JAOCS, *65* (1988) 1000–1006.

The authors of this paper

Dr. *Leon Cohen* got his Ph.D. in Chemistry at the University of Seville (Spain) in 1986. From 1994 he has obtained the European Chemist title. He has been in charge of the Customer Services Laboratory since 1984 at Petresa's plant. Besides this, he is a Professor of Chemistry at the University of Cadiz (Spain).

Mrs. *Rosa Vergara* was graduated in Chemistry at the University of Seville (Spain) in 1981. She joined Petresa in 1982 and she works at present at the Technical Service Laboratory.

Mr. *Alfonso Moreno* was graduated in Chemistry at the University of Granada (Spain) in 1965. Since 1969 he has worked in Petresa's petrochemical complex in San Roque (Spain) where he is currently Head of the Chemical Department.

Mr. *José Luis Berna* was graduated in Chemistry at the University of Zaragoza (Spain) in 1967 and in Petroleum Technology at Madrid University in 1969. In 1971 he joined Petresa as a research chemist and he is at present Director of Research and Development of this Company. (11561)

Neuer Elektrodenkatalog

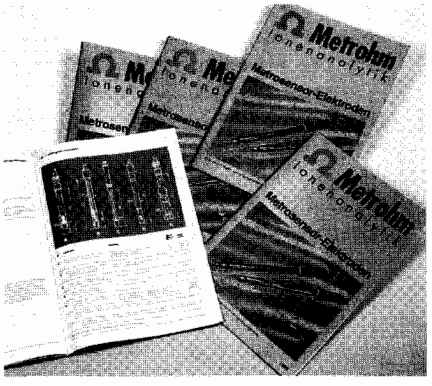
Übersichtlich gegliedert, informiert der Metrohm-Elektrodenkatalog über Elektrodentypen, Maße und über spezielle Anwendungsbereiche.

Der Leser erhält nützliche Hinweise über Aufbau, Behandlung, Lagerung und Prüfung von pH-, Metall- und Referenzelektroden.

Vorgestellt werden neben den herkömmlichen Elektroden

- die neuen Longlife pH-Elektroden mit Ag/AgCl-Kartusche,
- die Titroden, diaphragmalose kombinierte Elektroden für die Titration,
- die High Sense-Tensidelektrode für die umweltfreundliche Bestimmung von anionischen und kationischen Tensiden.

Über die Frage „Welche Elektrode für welche Titration?" informiert eine Tabelle und gibt dem Praktiker fundierte Einsatzlösungen für seine Titrationsprobleme.



Dem 56 seitigen Katalog ist ein Sonderpreisgutschein beigelegt, für den persönlichen Test einer pH-Longlife Elektrode. (66544)