INTERNATIONAL STANDARD ISO 3206

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Surface active agents — Analysis of technical alkane sulphonates — Determination of alkane monosulphonates content

Agents de surface — Analyse des alcanesulfonates techniques — Détermination de la teneur en alcanemonosulfonates

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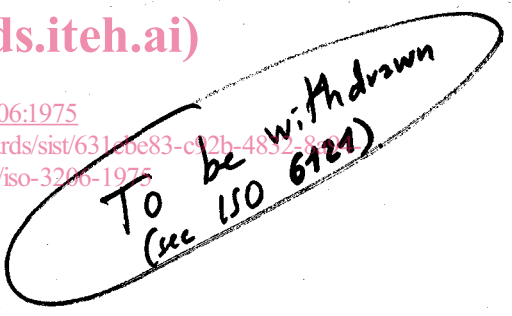

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Surface active agents — Analysis of technical alkane sulphonates — Determination of alkane monosulphonates content

0 INTRODUCTION

The analysis based on selective solvent extraction which has been applied successfully for the analysis of technical sodium alkylsulphonates in ISO/R 893 leads to only approximate results during the analytical examination of the products of sulphochlorination and sulphoxidation of paraffins, owing to the effect of the alkane disulphonates formed during the synthesis. However, because of the variety of technological and application properties arising from variations in the relative proportions of mono- and disulphonates, the knowledge of their contents is an all-important matter.

Therefore, it has seemed advisable to establish this method in the first place, to allow the determination of the content of alkane monosulphonates. The method is applicable to all synthetic alkanesulphonates.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the alkane monosulphonates content of technical alkane sulphonate with small quantities of paraffins. It is applicable to all alkali metal salts of the products of sulphochlorination and sulphoxidation of paraffins.

2 REFERENCE

ISO/R 893, *Surface active agents — Technical sodium alkylsulphonates — Methods of analysis.*

3 DEFINITION

For the purpose of this International Standard, the following definition applies :

alkane monosulphonate : Alkali metal salt of the monosulphonic acids present in the technical products of sulphochlorination and sulphoxidation of pure straight-chain paraffins of which the chain consists of between 12 and 20 carbon atoms.

4 PRINCIPLE

Acidification with hydrochloric acid of an aqueous ethanolic solution of technical alkane sulphonates.

Extraction with light petroleum in a liquid-liquid extractor.

Separation of the alkane monosulphonic acids and of the unsulphonated matter in the light petroleum phase and of the alkane disulphonates and the sulphate ions in the aqueous ethanolic phase.

Evaporation of the light petroleum phase, addition of water and neutralization, followed by evaporation so that the small quantities of paraffins are eliminated. Determination of the alkane monosulphonates by content weighing.

5 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade, and only distilled water or water of equivalent purity.

5.1 Acetone, ρ_{20} 0,790 5 to 0,791 1 g/ml.

5.2 Light petroleum, distilling between 40 and 60 °C.

5.3 Hydrochloric acid, ρ_{20} 1,18 g/ml.

5.4 Ethanol, 50 % (*V*/*V*) neutralized with 0,1 N sodium hydroxide solution (5.6) in the presence of phenolphthalein (5.7) to a pale pink colour.

5.5 Sodium hydroxide, 0,5 N solution.

5.6 Sodium hydroxide, 0,1 N solution.

5.7 Phenolphthalein, 1 % solution in ethanol.

6 APPARATUS

Ordinary laboratory apparatus, and

6.1 Liquid-liquid extractor,[1] of about 300 ml capacity, fitted with two 29/32 ground glass joints (see figure).

6.2 Round-bottomed flasks, 500 ml, fitted with 29/32 ground glass sockets.

6.3 Reflux condenser, with 29/32 ground glass joints.

6.4 Porcelain dishes, 500 ml.

1) It is permitted to use another type of extractor for example : "Jalade Perforator" provided that this is stated in the test report.

6.5 Crystallizing dish, glass, 100 ml.

6.6 Oven, capable of being maintained at 103 ± 2 °C.

6.7 Burette, 50 ml, class B complying with ISO/R 385.

6.8 Burette, 10 ml, class A complying with ISO/R 385.

6.9 Water bath.

6.10 Desiccator, containing phosphorus pentoxide (P_2O_5).

6.11 Flask heater, for 500 ml flasks.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,001 g, 10 g of a dilute sample containing 20 to 30 % of alkane sulphonates, obtained by the procedure described in Part I, clause 3 of ISO/R 893, into a 150 ml beaker.

7.2 Determination

Dissolve the test portion in 50 ml of the ethanol (5.4), add 15 ml of the hydrochloric acid (5.3) and pour into the extractor (6.1), rinsing with 30 ml of the ethanol (5.4).

Fit the extractor (6.1) on the flask (A) (6.2) containing about 400 ml of the light petroleum (5.2) and fit the condenser (6.3) on the extractor (6.1).

Heat to boiling with flask heater (6.11) and carry out the extraction for 4 h or more at a distillation rate of about 1,5 l/h.

Replace the flask (A), in which the alkane monosulphonate has collected, by a second flask (B), containing 300 ml of fresh light petroleum, bring to the boil and continue the extraction for 2 h, at least.

NOTE – After extraction for about 4 h, the intermediate phase which often forms at the beginning of the extraction between the aqueous alcoholic and the light petroleum layers should disappear. If this is not the case, the proportions of water, ethanol and hydrochloric acid are unsatisfactory. It is then preferable to extract a new test portion.

Transfer separately the two fractions soluble in light petroleum into the porcelain dishes (6.4) and concentrate as far as possible on the water bath (6.9).

Dissolve each residue in 50 ml of the ethanol (5.4), add several drops of the phenolphthalein (5.7) and neutralize.

Neutralize the first extract with the sodium hydroxide solution (5.5) using the burette (6.7) and the second extract with the sodium hydroxide solution (5.6) using the burette (6.8).

The extraction is complete if the neutralization of the second extract requires less than 0,5 ml of the 0,1 N sodium hydroxide solution (5.6); otherwise, continue the extraction for periods of 2 h until this condition is met.

Combine the neutralized light petroleum extracts, transfer to the crystallizing dish (6.5), previously weighed, and evaporate to dryness on a water bath (6.9).

To eliminate the last traces of water and of unsulphonated matter, redissolve three times, each time with 5 to 10 ml of the acetone (5.1) and evaporate to dryness.

Dry the residue in the oven (6.6) for 2 h, allow to cool in the desiccator (6.10) and weigh to the nearest 0,001 g.

Repeat the operations of drying, cooling and weighing until the difference between two successive weighings does not exceed 0,001 g.

8 EXPRESSION OF RESULTS

8.1 Method of calculation

The alkane monosulphonates content of the diluted sample, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 \times 100}{m_0}$$

The alkane monosulphonates content of the raw material, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 \times 100}{m_0} \times \frac{1}{f}$$

where

m_0 is the mass, in grams, of the test portion (7.1);

m_1 is the mass, in grams, of the residue (sodium salt of the alkane monosulphonate);

f is the dilution factor of the test sample (see Part I, clause 3 of ISO/R 893).

8.2 Precision

Comparative analyses on a sample in the form of a homogeneous aqueous solution containing about 20 % of total soluble matter comprising : alkane mono-, di- and poly-sulphonates in the form of their sodium salts, sodium sulphate and small quantities of paraffins, carried out in 24 laboratories, have given the following statistical results :

– mean (percentage by mass)	16,92
– standard deviation of repeatability (σ_r)	0,15
– standard deviation of reproducibility (σ_R)	0,29

8.2.1 *Repeatability*

The maximum deviation found between the result of two determinations carried out on the same product simultaneously or in rapid succession by the same analyst using the same apparatus shall not exceed, in 5 cases out of 100, 2,77 σ_r i.e. 0,415.

8.2.2 *Reproducibility*

The maximum deviation between two results obtained on the same sample, using the same apparatus in two different laboratories, shall not exceed, in 5 cases out of 100, 2,77 σ_R, i.e. 0,803.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the type of extractor used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional.

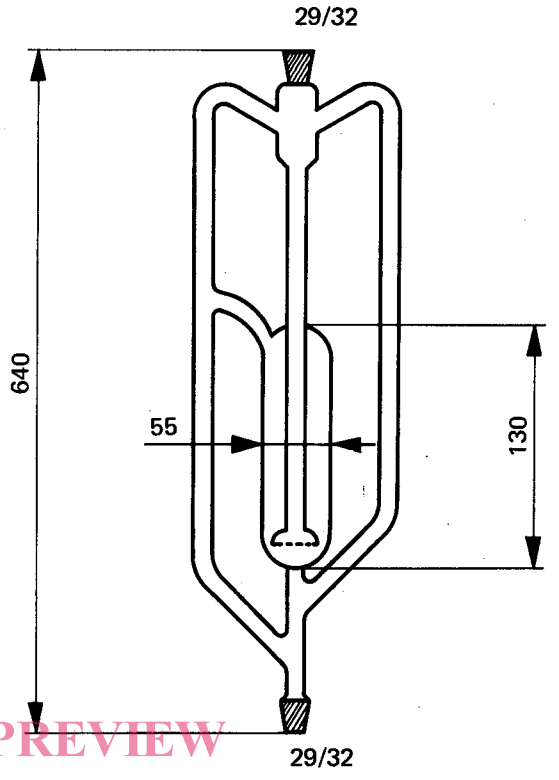

FIGURE – Liquid-liquid extractor of 300 ml (6.1)

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AMENDMENT

Foreword (Inside front cover)

The ISO Member Body for the U.S.A has now withdrawn its disapproval of this International Standard. The U.S.A. should therefore be included in the list of countries whose Member Bodies have approved the document.