
International Standard



6845

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● Surface active agents — Technical alkane sulphonates — Determination of mean relative molecular mass of alkane monosulphonates

*Agents de surface — Alcanesulfonates techniques — Détermination de la masse moléculaire relative moyenne des
alcanemonosulfonates*

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Descriptors : surfactants, tests, determination, molecular mass.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6845 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in February 1982.

It has been approved by the member bodies of the following countries:

Austria	Hungary	Romania
Belgium	Italy	South Africa, Rep. of
China	Japan	Spain
Czechoslovakia	Korea, Rep. of	Switzerland
Egypt, Arab Rep. of	Mexico	United Kingdom
France	Netherlands	USA
Germany, F.R.	Poland	USSR

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Surface active agents — Technical alkane sulphonates — Determination of mean relative molecular mass of alkane monosulphonates

0 Introduction

Knowledge of the mean relative molecular mass of alkane monosulphonate is necessary for the determination of alkane monosulphonates content by direct two-phase titration.

1 Scope and field of application

This International Standard specifies a method for the determination of the mean relative molecular mass of the alkane monosulphonate present in technical alkane sulphonates which do not contain significant quantities of paraffins.

The method is applicable to all alkali metal salts of products of sulphochlorination and sulphoxidation of paraffins.

2 References

ISO 607, *Surface active agents and detergents — Methods of sample division.*

ISO 893, *Surface active agents — Technical sodium alkane sulphonates — Methods of analysis.*

3 Definition

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

alkane monosulphonate: Alkali metal salts of the monosulphonic acids present in the technical products of sulphochlorination and sulphoxidation of straight-chain paraffins, free from branched-chain compounds, between 12 and 20 carbon atoms.

4 Principle

Evaporation of a test portion to dryness, dissolution of the residue in ethanol and acidification of the ethanolic solution with hydrochloric acid.

Extraction with light petroleum in a liquid-liquid extractor, giving quantitative separation of the alkane monosulphonic

acids into the light petroleum phase and of the alkane disulphonates and sulphate ions into the aqueous/ethanolic phase.

Evaporation of the light petroleum phase and addition of ethanol.

Removal of traces of alkali by means of a cation-exchange column.

Neutralization of the eluate with carbonate-free sodium hydroxide solution. Drying and weighing of the sodium alkane monosulphonate so formed. Calculation of the mean relative molecular mass.

5 Reagents and products

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, free from carbon dioxide.

5.1 Acetone.

5.2 Ethanol, 95 % (V/V).

5.3 Ethanol, 50 % (V/V).

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

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

5.6 Sodium hydroxide, standard volumetric aqueous/ethanolic solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$, free from carbonates.

5.6.1 Preparation of the solution.

Weigh, to the nearest 1 mg, into a 250 ml conical flask fitted with a soda-lime tube, 8,1 g of sodium hydroxide pellets and dissolve them in 100 ml of the ethanol (5.2).

Allow to cool. The main part of the sodium carbonate present, which is insoluble, will settle. Then, using a pipette, transfer 10,0 ml of the clear solution to a 200 ml one-mark volumetric flask and dilute to the mark with the carbon dioxide-free water.

5.6.2 Standardization of the solution.

Determine the exact concentration of the sodium hydroxide solution (5.6.1) using potassium hydrogen phthalate as the primary standard and phenolphthalein as the indicator.

5.6.3 Check for absence of carbonates.

Check that the solution is free from carbonates by transferring a small amount of the solution to a test tube and adding the same volume of a 130 g/l barium nitrate solution. The solution shall remain clear for at least 5 min for the sodium hydroxide solution to be considered suitable.

5.7 Bromophenol blue, 1 g/l ethanolic solution.

5.8 Cation-exchange resin, strongly acidic (sulphonic group), with a degree of cross-linking corresponding to 2 % of divinylbenzene.

6 Apparatus

Usual laboratory equipment, and

6.1 Liquid-liquid extractor of capacity about 300 ml, with two ground glass joints (see the figure).

6.2 Round-bottomed flask, capacity of 500 ml, with a ground glass neck to fit the lower glass joint of the extractor (6.1).

6.3 Flat-bottomed flask, of capacity 250 ml, with a ground glass neck and a ground glass stopper.

6.4 Reflux condenser, with a ground glass joint at its lower end, to fit the upper ground glass joint of the extractor (6.1).

6.5 Beakers, of capacities 150 and 250 ml.

6.6 Burette, of capacity 50 ml, complying with the requirements of ISO 385/1, class A.

6.7 Column for ion exchange resin, of internal diameter 12 mm and height 150 mm.

6.8 Boiling water bath.

6.9 Water bath, capable of being controlled at 70 °C.

6.10 Vacuum oven, capable of being controlled at 120 ± 2 °C.

6.11 Magnetic stirrer.

7 Sampling

The laboratory sample of surface active agent shall be prepared and stored in accordance with the instructions given in ISO 607.

8 Procedure

8.1 Test portion

After obtaining a dilute sample containing 20 to 30 % of alkane sulphonates (see ISO 893, clause 5), weigh, to the nearest 0,001 g into one of the 150 ml beakers (6.5), a test portion corresponding to between 0,6 and 0,8 g of alkane monosulphonate.

8.2 Preparation of the cation-exchange column

Place, in one of the 150 ml beakers (6.5), 10 ml of the cation-exchange resin (5.8) and 30 ml of hydrochloric acid solution [prepared by mixing 10 ml of the hydrochloric acid (5.5), with 20 ml of water]. By means of the magnetic stirrer (6.11), stir slowly for 2 h.

Allow the resin to settle, replace the aqueous phase by 30 ml of the ethanol (5.2) and stir slowly for 1 h.

Transfer the resin to the column (6.7) and wash it with the ethanol (5.2) until chloride ions can no longer be detected in the eluate.

NOTE — The column should not be allowed to run dry and the resin should always be impregnated with liquid.

The rate of elution should be adjusted to about 6 ml/min for filling, washing, or regeneration.

8.3 Regeneration of the column

After about 100 determinations, pass 30 ml of hydrochloric acid solution [prepared by mixing 10 ml of the hydrochloric acid (5.5), with 20 ml of water] through the column and wash with 50 ml of water followed by 50 ml of the ethanol (5.2). Continue washing with ethanol until chloride ions can no longer be detected in the eluate.

8.4 Determination

WARNING — Evaporate the petroleum and ethanolic solutions in a well-ventilated fume cupboard.

Evaporate the test portion (8.1) to dryness on the boiling water bath (6.8) and dissolve the residue in 50 ml of the ethanol (5.3) and 20 ml of the hydrochloric acid (5.5). Transfer the solution from the beaker to the extractor (6.1), rinsing with 30 ml of ethanol (5.3).

Fit the extractor (6.1) to the flask (6.2) containing about 400 ml of the light petroleum (5.4) and fit the reflux condenser (6.4) to the extractor.

Heat the contents of the flask to boiling on the water bath (6.9), controlled at about 70 °C, and carry out the extraction for 5 h at a distillation rate of about 1,5 l/h.

Remove the flask (6.2) from the extractor (6.1) and evaporate to dryness on the water bath (6.9), while passing a gentle current of nitrogen over the surface of the liquid in order to accelerate the evaporation.

Dissolve the residue in 50 ml of the ethanol (5.2) and pass this solution through the previously prepared cation-exchange column (see 8.2).

Collect the eluate in the flat-bottomed flask (6.3), previously tared with its stopper. Wash the column with 100 ml of the ethanol (5.2), collecting the washings in the flask.

Add a few drops of the bromophenol blue solution (5.7) to the eluate. Stir this solution using the magnetic stirrer (6.11) and add the sodium hydroxide solution (5.6) by means of the burette (6.6) until a blue colour is obtained.

Evaporate the neutralized solution to dryness on the boiling water bath (6.8) while passing a gentle current of nitrogen over the surface of the solution.

To eliminate the last traces of water, carry out the following procedure three times. Dissolve the residue in 10 ml of the acetone (5.1) and evaporate to dryness on the boiling water bath (6.8) while passing a gentle current of nitrogen over the surface of the solution.

Dry the residue and the flask for 1 h in the vacuum oven (6.10), controlled at 120 ± 2 °C, allow to cool in a desiccator containing phosphorus(V) oxide (diphosphorus pentaoxide) and weigh the stoppered flask (6.3) 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.

9 Expression of results

9.1 Method of calculation

The mean relative molecular mass, \bar{M}_r , of the sodium alkane monosulphonate present in the technical alkane sulphonate is given by the formula

$$\frac{m}{V \times c} \times 1\,000 = \frac{1\,000\,m}{V \times c}$$

where

m is the mass, in grams, of the sodium monosulphonate formed in 8.4;

V is the volume, in millilitres, of the sodium hydroxide solution (5.6), used for neutralization of the extracted monosulphonic acid;

c is the exact concentration, in moles of NaOH per litre, of the sodium hydroxide solution (5.6).

9.2 Precision

Comparative analyses on a homogeneous aqueous solution containing about 25 % (m/m) of total soluble matter (compris-

ing alkane mono-, di- and polysulphonates in the form of their sodium salts, sodium sulphate and small quantities of paraffins) carried out in 17 laboratories, have given the following statistical results:

– mean (mean relative molecular mass)	: 320
– standard deviation of repeatability	: 2,03
– standard deviation of reproducibility	: 6,49

10 Test report

The test report shall include the following information:

- all the information necessary for the complete identification of the sample;
- the reference of the method used (reference to this International Standard);
- the results and the method of expression used;
- details of any operations not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have affected the results.

Dimensions in millimetres

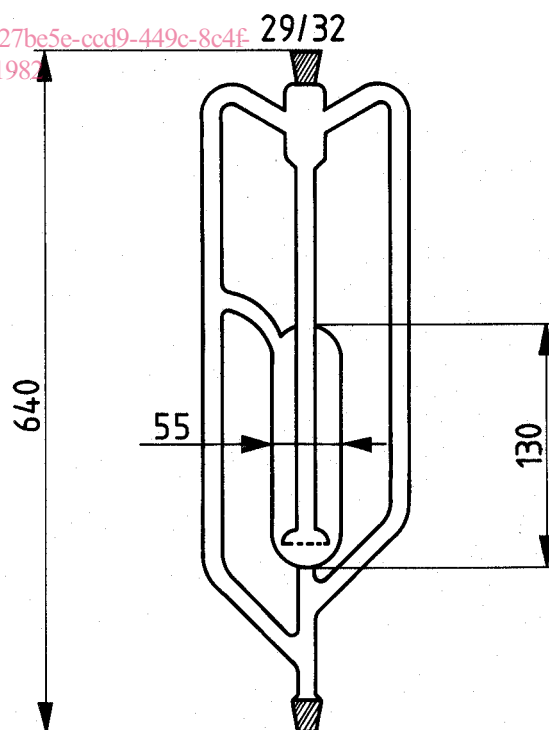


Figure — 300 ml liquid-liquid extractor (6.1)