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INTERNATIONAL STANDARD 6122

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Surface active agents — Technical alkane sulphonates — Determination of total alkane sulphonates content

Agents de surface — Alcanesulfonates techniques — Détermination de la teneur en alcanesulfonates totaux

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FOREWORD

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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 6122 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in September 1977.

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

Australia	India	Romania
Austria	Iran	South Africa, Rep. of
Belgium	Italy	Spain
Brazil	Japan	Switzerland
Bulgaria	Kenya	Turkey
Chile	Mexico	United Kingdom
France	Netherlands	U.S.A.
Germany	New Zealand	U.S.S.R.
Hungary	Poland	

No member body expressed disapproval of the document.

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Surface active agents — Technical alkane sulphonates — Determination of total alkane sulphonates content

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of total alkane sulphonates content (mono- + di-) of technical alkane sulphonates containing small quantities of paraffins.

It is applicable to all alkali metal salts of the products of sulphochlorination and sulphoxidation of paraffins.

2 REFERENCES

ISO 607, *Surface active agents and detergents — Methods of sample division.* [1)]

ISO 894, *Surface active agents — Technical sodium primary alkylsulphates — Methods of analysis.*

3 DEFINITION

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

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

4 PRINCIPLE

Dispersion of a test portion of the technical alkane sulphonate in a sodium sulphate solution, and addition of a mixture of butan-1-ol and acetone to precipitate sodium sulphate decahydrate and part of the sodium chloride.

Filtration to obtain a mixture of alkane sulphonates (mono- + di-), which may be contaminated by a little sodium chloride.

Evaporation of solvent so that the small quantities of paraffins are eliminated, gravimetric determination of the alkane sulphonates content, and potentiometric determination of any sodium chloride present.

5 REAGENTS

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

5.1 Acetone.

5.2 Butan-1-ol and acetone mixture (60 + 40) ($V_1 + V_2$).

5.3 Sodium sulphate, anhydrous, 200 g/l solution.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Boiling flask or crystallizing dish, glass, 250 ml.

6.2 Oven capable of being regulated at 120 ± 2 °C.

6.3 Magnetic stirrer.

7 SAMPLING

The laboratory sample shall be prepared and stored according to the instructions given in ISO 607.

8 PROCEDURE

8.1 Test portion

Weigh, to the nearest 0,001 g, into a 250 ml conical flask, a test portion which contains about 0,5 to 1 g of total alkane sulphonates.

8.2 Determination

Add to the test portion (8.1), 15 ml of the sodium sulphate solution (5.3) and heat the mixture to 50 °C, stirring with the magnetic stirrer (6.3).

1) At present at the stage of draft. (Revision of ISO/R 607.)

When the suspension is homogeneous, stop the heating and add, while stirring, 150 ml of the butan-1-ol and acetone mixture (5.2) in a thin trickle. Stopper the conical flask and cool to room temperature.

Decant the clear phase through a fast filter paper and collect the filtrate in the crystallizing dish or the boiling flask (6.1), previously weighed to the nearest 0,001 g.

Wash the large-grained precipitate and the sides of the conical flask four times, each time with 15 ml of the butan-1-ol and acetone mixture (5.3).

Evaporate the filtrate and the added washing solutions to dryness on a boiling water-bath, and under a slow stream of cold dry nitrogen or air.

To eliminate the last traces of water and unsulphonated matter, dissolve the residue in 10 ml of the acetone (5.1) in the boiling flask or the crystallizing dish (6.1) and evaporate to dryness on a water-bath under a dry unheated stream of air or nitrogen. Repeat this procedure twice more.

Dry the residue in the oven (6.2), regulated at 120 ± 2 °C, for 1 h, allow to cool in a desiccator to room temperature and weigh to the nearest 0,001 g.

To determine the sodium chloride which may be present, dissolve the residue obtained after weighing in 80 ml of water and 20 ml of the acetone (5.1) and determine the sodium chloride content by the method specified in 6.8 of ISO 894.

NOTE — In the case of alkane sulphonates containing 20 carbon atoms in the alkane chain, the procedure for removing unsulphonated matter is not fully effective.

9 EXPRESSION OF RESULTS

9.1 Calculation

The total alkane sulphonates content, expressed as a percentage by mass, is given by the formula :

$$\frac{m_1 - (V_4 \times T \times 0{,}058\,5)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (8.1);

m_1 is the mass, in grams, of the residue obtained;

V_4 is the value, in millilitres, of V_{EQ} corresponding to the determination in 6.8 of ISO 894;

T is the exact normality of the silver nitrate solution used for the determination in 6.8 of ISO 894.

9.2 Precision

Comparative analyses on a sample in the form of a homogeneous aqueous solution containing about 25 % (*m/m*) 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 18 laboratories, have given the following statistical results :

— mean (percentage by mass) : 24,10

— standard deviation of repeatability (σ_r) : 0,45

— standard deviation of reproducibility (σ_R) : 0,93

10 TEST REPORT

The test report shall contain the following information :

a) all information necessary for the complete identification of the sample;

b) the reference of the method used (reference to this International Standard);

c) the results and the method of expression used;

d) the test conditions;

e) any operational details not specified in this International Standard or the International Standards to which reference is made, or regarded as optional, as well as any incidents likely to have affected the results.