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Surface active agents — Technical alkane sulfonates — Methods of analysis

Agents de surface — Alcanesulfonates techniques — Méthode d'analyse



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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 893 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 893 : 1978), of which it constitutes a minor revision.

Annexes A and B of this International Standard are for information only.

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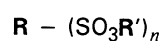
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Introduction

Alkane sulfonates have the general formula



where

R is a saturated aliphatic radical having a chain length of about 12 to 20 carbon atoms;

R' is an alkali metal;

n may be 1 or 2.

They are obtained by sulfochlorination and sulfoxidation of straight-chain paraffins free from branched-chain compounds.

They are alkali metal salts of mono- and disulfonic acids.

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Surface active agents — Technical alkane sulfonates — Methods of analysis

1 Scope

This International Standard specifies methods of analysis of technical alkane sulfonates. It covers the following determinations:

- measurement of pH;
- determination of water content;
- determination of free alkali or free acid;
- determination of matter extractable by light petroleum;
- determination of total alkane sulfonate content;
- determination of alkane monosulfonate content;
- determination of sulfite content;
- determination of sulfate content;
- determination of chloride content.

It also sets out, in annexes:

- A: a general scheme of analysis;
- B: a method for the determination of total salts content.

This International Standard is applicable to technical alkane sulfonates in powder, paste or liquid form, free from any products extraneous to their manufacture.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 607 : 1980, *Surface active agents and detergents — Methods of sample division*.

ISO 894 : 1977, *Surface active agents — Technical sodium primary alkylsulphates — Methods of analysis*.

ISO 1104 : 1977, *Surface active agents — Technical sodium alkylarylsulphonates (excluding benzene derivatives) — Methods of analysis*.

ISO 4314 : 1977, *Surface active agents — Determination of free alkalinity or free acidity — Titrimetric method*.

ISO 4316 : 1977, *Surface active agents — Determination of pH of aqueous solutions — Potentiometric method*.

ISO 4317 : 1977, *Surface active agents — Determination of water content — Karl Fischer method*.

ISO 4318 : 1989, *Surface active agents and soaps — Determination of water content — Azeotropic distillation method*.

ISO 6121 : 1988, *Surface active agents — Technical alkane sulfonates — Determination of alkane monosulfonates content by direct two-phase titration*.

ISO 6122 : 1978, *Surface active agents — Technical alkane sulphonates — Determination of total alkane sulphonates content*.

ISO 6844 : 1983, *Surface active agents — Determination of mineral sulfate content — Titrimetric method*.

ISO 6845 : 1989, *Surface active agents — Technical alkane sulfonates — Determination of the mean relative molecular mass of the alkane monosulfonates and the alkane monosulfonate content*.

3 General principle¹⁾

Dissolution of the laboratory sample in an appropriate volume of water so that the technical alkane sulfonate content is approximately 20 % (m/m) to 30 % (m/m).

From an aliquot portion of this solution, known as the diluted sample, preparation of an aqueous alcoholic solution from which the products extractable by light petroleum are isolated.

1) See the general scheme of analysis in annex A.

On other portions of the diluted sample

- determination of total alkane sulfonate content;
- determination of alkane monosulfonate content;
- determination of sulfate content;
- determination of chloride content.

On separate test portions of the laboratory sample

- measurement of pH;
- determination of water content;
- determination of free alkali or free acid;
- determination of sulfite content.

4 Sampling

4.1 Laboratory sample

Prepare and store a laboratory sample of approximately 300 g of raw product in accordance with the instructions given in ISO 607.

4.2 Preparation of diluted sample

To a part of the laboratory sample so obtained (m_0), add a quantity of water (m) so that the technical alkane sulfonate content is approximately 20 % (m/m) to 30 % (m/m). The dilution factor, f , is given by the formula

$$f = \frac{m_0}{m_0 + m}$$

NOTE — To convert the results of the analysis into percentages by mass based on the raw material, multiply the results by the reciprocal of the dilution factor, i.e.

$$\frac{1}{f} = \frac{m_0 + m}{m_0}$$

5 Methods of analysis

5.1 Measurement of pH

Carry out the measurement of pH by the method specified in ISO 4316, on a 5 % (m/m) solution of the laboratory sample.

5.2 Determination of water content

Depending on the amount of water in the product, carry out the determination by one of the following two methods:

- a) the Karl Fischer method, applicable to products having less than 10 % (m/m) of water;
- b) the azeotropic distillation method, applicable only for products containing more than 5 % (m/m) of water.

5.2.1 Karl Fischer method

Carry out the determination of water content by the method specified in ISO 4317.

5.2.2 Azeotropic distillation method

Carry out the determination of water content by the method specified in ISO 4318.

5.3 Determination of free alkali or free acid

Carry out the determination of free alkali or free acid by the method specified in ISO 4314.

5.4 Determination of matter extractable by light petroleum

Carry out the determination of matter extractable by light petroleum by the method specified in 6.4 of ISO 1104 : 1977, on a test portion of 80 g, weighed to the nearest 0,05 g, of the diluted sample (see 4.2).

The combined aqueous alcoholic residue, L_1 (see annex A), can be used for the determination of the total salts content (see annex B); this determination may be used to check the result obtained for the matter extractable from the raw product by light petroleum.

5.5 Determination of total alkane sulfonate content

Carry out the determination of total alkane sulfonate content by the method specified in ISO 6122. For a check on the result obtained, see annex B.

5.6 Determination of alkane monosulfonate content

Carry out the determination of alkane monosulfonate content either by the method specified in ISO 6845 or by the method specified in ISO 6121.

5.7 Determination of sulfite content

Carry out the determination of sulfite content by the method specified in 6.6 of ISO 1104 : 1977, on a test portion of 10 g, weighed to the nearest 1 mg, of the laboratory sample (4.1).

5.8 Determination of sulfate content

Carry out the determination of sulfate content by the method specified in ISO 6844.

5.9 Determination of chloride content

Carry out the determination of chloride content by the method specified in 6.8 of ISO 894 : 1977, on a test portion of 4 g to 5 g, weighed to the nearest 1 mg, of the diluted sample (see 4.2).

6 Test report

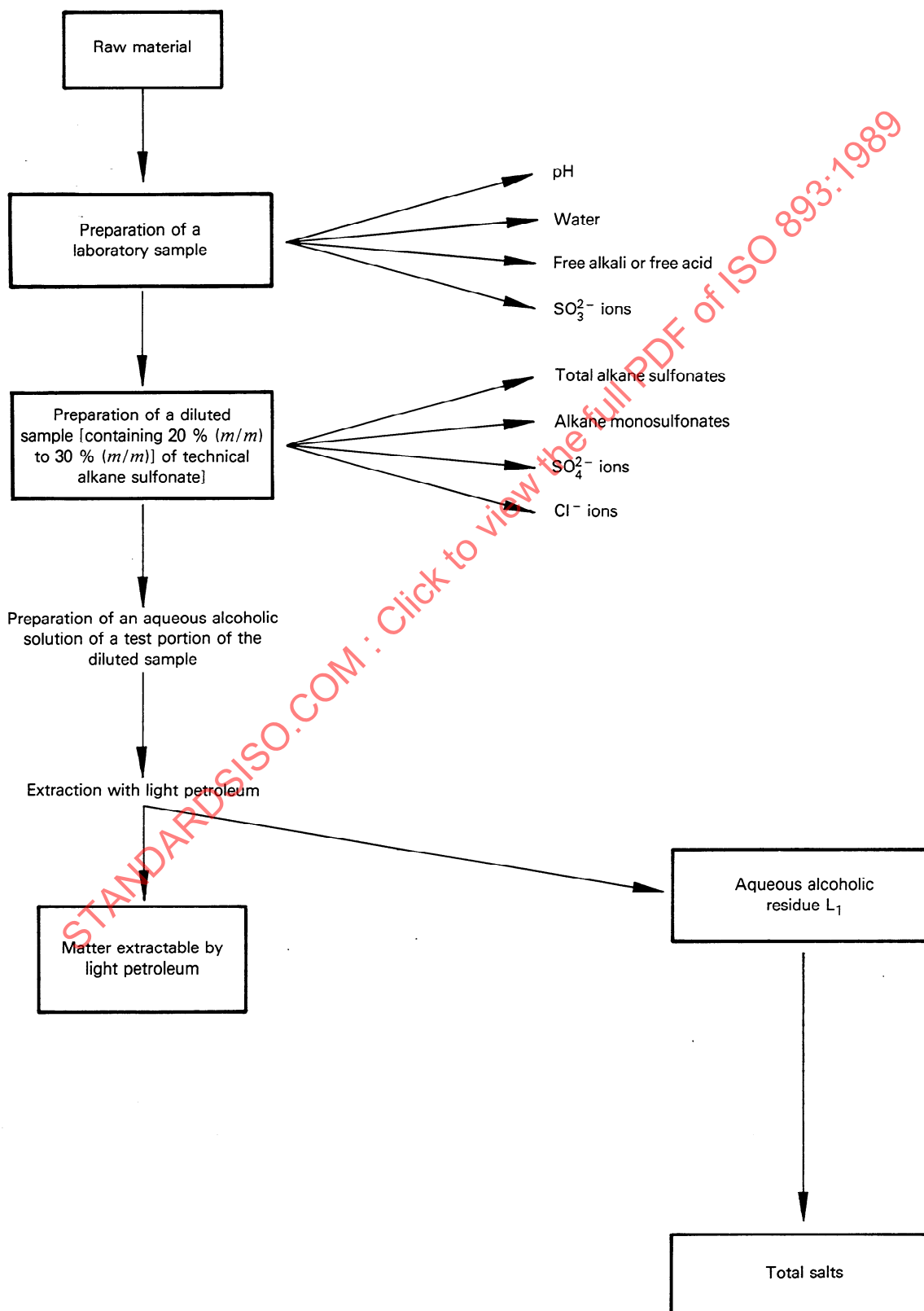
The test report shall include the following particulars:

- a) all information necessary for the complete identification of the sample;
- b) a reference to this International Standard and to the methods used;
- c) the results obtained and the units in which they have been expressed;
- d) any operational details not specified in this International Standard or in the International Standards to which reference is made, and any operation regarded as optional, as well as any incidents likely to have affected the results.

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Annex A (informative)

General scheme of analysis



Annex B (informative)

Determination of total salts

B.1 General

When necessary, check the results obtained from the methods specified in 5.4 (matter extractable by light petroleum) and 5.5 (total alkane sulfonate content) by means of the following equations:

$$\text{total alkane sulfonates} = \text{total salts} - (\text{alkali metal chloride} + \text{alkali metal sulfate} + \text{alkali metal sulfite}) \quad \dots \text{ (B.1)}$$

$$\text{matter extractable by light petroleum} = 100 - (\text{total salts} + \text{water content}) \quad \dots \text{ (B.2)}$$

For this purpose, it is necessary to know the total salts content.

By "total salts content" is meant the sum of the contents of all the alkali metal salts present in the raw product, i.e. the sum of the following:

$$\text{alkali metal chloride} + \text{alkali metal sulfate} + \text{alkali metal sulfite} + \text{total alkane sulfonates}$$

In view of the fact that the matter extractable by light petroleum may contain volatile products which could give rise to losses during the direct determination by the method specified in 5.4, it may be preferable, if in any doubt, to calculate the matter extractable by light petroleum using equation (B.2).

B.2 Principle

Evaporation to dryness of an aliquot portion of the aqueous alcoholic liquid, L₁, obtained by the method specified in 5.4. Drying the residue at 130 °C and weighing it.

B.3 Apparatus

Ordinary laboratory apparatus and:

B.3.1 Two crystallizing dishes, 50 mm diameter, 100 ml capacity.

B.3.2 Pipette, capacity 25 ml, complying with the requirements of ISO 648*).

B.3.3 Oven, capable of being maintained at 130 °C to 135 °C.

B.4 Procedure

B.4.1 Test portion

Into each of the two crystallizing dishes (B.3.1), previously dried in the oven (B.3.3) maintained at 130 °C to 135 °C, allowed to cool and weighed to the nearest 1 mg, place, by means of the pipette (B.3.2), 25,0 ml of the aqueous alcoholic liquor, L₁, produced during the determination of the matter extractable by light petroleum (see 5.4).

B.4.2 Determination

Evaporate the test portion (B.4.1) in the dishes to dryness on a water bath. Transfer the dishes to the oven (B.3.3), maintained at 130 °C to 135 °C, and dry to constant mass (i.e. until the results of two consecutive weighings carried out at an interval of 30 min do not differ by more than 5 mg).

B.5 Expression of results

B.5.1 Method of calculation

The total salts content in the raw material, expressed as a percentage by mass, is given by the formula

$$\begin{aligned} & \frac{m_2 + m_3}{2} \times \frac{100}{m_1} \times \frac{500}{25} \times \frac{1}{f} \\ &= \frac{m_2 + m_3}{m_1} \times 1\,000 \times \frac{1}{f} \end{aligned}$$

where

m_1 is the mass, in grams, of the test portion specified in 5.4;

m_2 is the mass, in grams, of the residue in the first dish;

m_3 is the mass, in grams, of the residue in the second dish;

f is the dilution factor of the diluted sample as calculated in 4.2.

*) ISO 648 : 1977, *Laboratory glassware — One-mark pipettes*.