TC 91

INTERNATIONAL STANDARD 893

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

Surface active agents — Technical sodium ~~alkane~~ alkyl sulphonates — Methods of analysis

Agents de surface — Alcanesulfonates de sodium techniques — Méthode d'analyse

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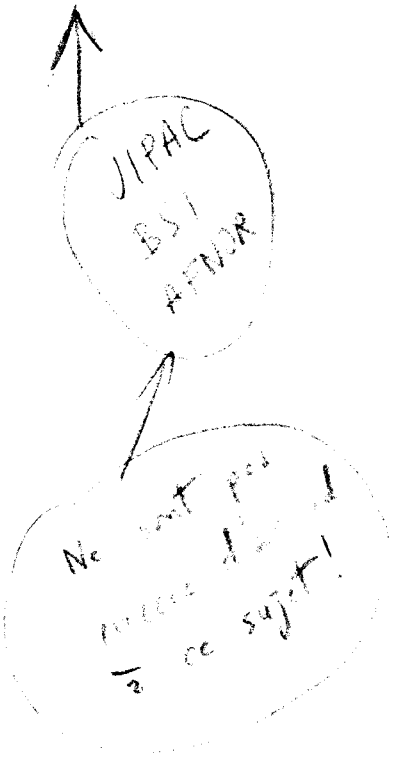

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Descriptors : surfactants, alkysulphonate, chemical analysis.

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

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

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

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 893-1968, of which it constitutes a technical revision.

Printed in Switzerland

Surface active agents — Technical sodium alkane sulphonates — Methods of analysis

0 INTRODUCTION

Sodium alkane sulphonates have the general formula

$$R - (SO_3Na)_n$$

where **R** is a saturated aliphatic radical having a mean chain length of about 12 to 20 carbon atoms, and n may be 1 or 2. They are sodium salts of mono- and disulphonic acids.

They are obtained by sulphochlorination and sulphoxidation of straight-chain paraffins free from branched-chain compounds.

1 SCOPE

This International Standard specifies methods of analysis of technical sodium alkanesulphonates. It covers the following :

- Measurement of pH.
- Determination of water content.
- Determination of free alkalinity or free acidity.
- Determination of matter extractable by light petroleum.
- Determination of sodium alkane sulphonates content.
- Determination of alkane monosulphonates content.
- Determination of sodium sulphite content.
- Determination of sodium sulphate content.
- Determination of sodium chloride content.

It also sets out, in annexes :

A : a general scheme of analysis;

B : a method for the determination of total salts content.

2 FIELD OF APPLICATION

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

3 REFERENCES

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

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

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

ISO 3206, *Surface active agents — Analysis of technical alkane sulphonates — Determination of alkane monosulphonates content.*

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

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

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

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

ISO 6121, *Surface active agents — Technical alkane sulphonates — Determination of alkane monosulphonates content — Direct two-phase titration method.*

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

4 GENERAL PRINCIPLE[2)]

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

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

1) At present at the stage of draft. (Revision of ISO/R 607-1967.)

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

On other portions of the dilute sample :

- determination of alkane sulphonates content;
- determination of alkane monosulphonates content;
- determination of sodium sulphate content;
- determination of sodium chloride content.

On separate test portions of the laboratory sample :

- measurement of pH;
- determination of water content;
- determination of free alkalinity or free acidity;
- determination of sodium sulphite content.

5 SAMPLING

5.1 Laboratory sample

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

5.2 Preparation of dilute sample

To a part of the laboratory sample so obtained (m_0) add a quantity of water (m) so that the technical sodium alkane sulphonate content is approximately 20 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.

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

6 METHODS OF ANALYSIS

6.1 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.

6.2 Determination of water content

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

a) the Karl Fischer method, applicable to products having less than 10 % (m/m) of water;

b) the azeotropic distillation method, which should be used only for products containing more than 5 % (m/m) of water.

6.2.1 *Karl Fischer method*

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

6.2.2 *Azeotropic distillation method*

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

6.3 Determination of free alkalinity or free acidity

Carry out the determination of free alkalinity or free acidity by the method specified in ISO 4314.

6.4 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, on a test portion of 80 g, weighed to the nearest 0,05 g, of the dilute sample (5.2).

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

6.5 Determination of alkane sulphonates content

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

6.6 Determination of alkane monosulphonates content

Carry out the determination of alkane monosulphonates either by the method specified in ISO 3206 or by the method specified in ISO 6121.

6.7 Determination of sodium sulphite content

Carry out the determination of sodium sulphite content by the method specified in 6.6 of ISO 1104 on a test portion of 10 g, weighed to the nearest 0,001 g, of the laboratory sample (5.1).

6.8 Determination of sodium sulphate content

An International Standard dealing with this determination is in preparation. In the meantime, carry out this determination on a test portion of the dilute sample (5.2) using any suitable method.

6.9 Determination of sodium chloride content

Carry out the determination of sodium chloride content by the method specified in 6.8 of ISO 894 on a test portion of 4 to 5 g, weighed to the nearest 0,001 g, of the dilute sample (5.2).

7 TEST REPORT

The test report shall include the following particulars :

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

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

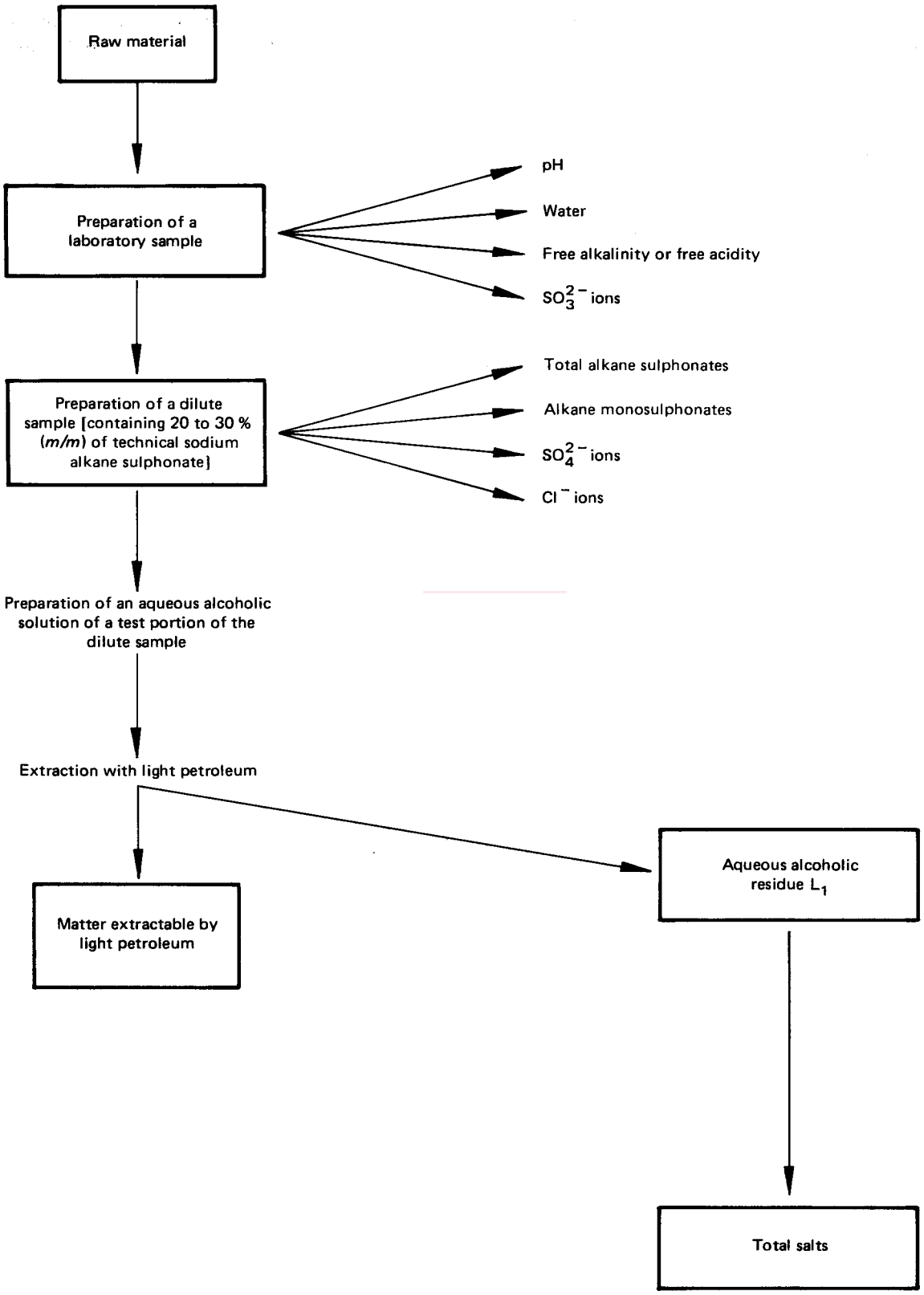
c) the results obtained and the method of expression used;

d) the test conditions;

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

ANNEX A

GENERAL SCHEME OF ANALYSIS



ANNEX B

DETERMINATION OF TOTAL SALTS

B.0 INTRODUCTION

When necessary, check the results obtained from the methods specified in 6.4 (matter extractable by light petroleum) and 6.5 (sodium alkane sulphonates content), by means of the following equations :

sodium alkane sulphonates = total salts − (sodium chloride + sodium sulphate + sodium sulphite) ... (1)

matter extractable by light petroleum = 100 − (total salts + water content) ... (2)

For this purpose, a knowledge of the total salts content is necessary.

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

sodium chloride + sodium sulphate + sodium sulphite + sodium alkane sulphonates

In case of doubt, knowing 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 6.4, it may be preferable to calculate the matter extractable by light petroleum using equation (2).

B.1 PRINCIPLE

Evaporation to dryness of an aliquot portion of the aqueous alcoholic liquid, L_1, obtained by the method specified in 6.4. Drying the residue at 130 °C and weighing it.

B.2 APPARATUS

Ordinary laboratory apparatus and :

B.2.1 Two crystallizing dishes, 50 mm diameter, 100 ml capacity.

B.2.2 Pipette, capacity 25 ml, complying with the requirements of ISO 648.

B.2.3 Oven, capable of being controlled at 130 to 135 °C.

B.3 PROCEDURE

B.3.1 Test portion

Into each of the two crystallizing dishes (B.2.1), previously dried in the oven (B.2.3), controlled at 130 to 135 °C, allowed to cool and weighed to the nearest 0,001 g, place, by means of the pipette (B.2.2), 25,0 ml of the aqueous alcoholic liquor, L_1, produced during the determination of matter extractable by light petroleum (see 6.4).

B.3.2 Determination

Evaporate the test portion (B.3.1), in the dishes, to dryness on a water bath. Transfer the dishes to the oven (B.2.3), controlled at 130 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 0,005 g).