INTERNATIONAL STANDARD 895

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Surface active agents — Technical sodium secondary alkylsulphates — Methods of analysis

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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 895 was developed by Technical Committee ISO/TC 91, *Surface active agents*, and was circulated to the member bodies in October 1975.

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

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

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 895-1968, of which it constitutes a technical revision.

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Surface active agents — Technical sodium secondary alkylsulphates — Methods of analysis

0 INTRODUCTION

The word "secondary" preceding the generic name for the products in the title is intended to distinguish these products from those which, in accordance with current scientific usage, could be designated as technical sodium primary alkylsulphates. As shown in the general formula given below, the former may be considered as derived from secondary alcohols, whereas the latter are derived from primary alcohols.

It is therefore the former which are the subject of this International Standard. They are commonly known today as technical sulphates of secondary fatty alcohols.

In order to simplify the text of this International Standard and avoid unnecessary repetition, the word "secondary" has been omitted from the term "sodium alkylsulphates", but it should be understood that only "sodium secondary alkylsulphates" are covered.

The general formula of the products which are the subject of this International Standard is

$$\begin{matrix} R \\ R' \end{matrix}\!\!>\!\!CH - O - SO_3Na$$

where **R** and **R'** are aliphatic radicals.

1 SCOPE

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

- Measurement of pH.
- Determination of water content.
- Determination of free alkalinity or free acidity.
- Determination of total alkalinity.
- Determination of matter extractable by light petroleum.
- Determination of the sodium alkylsulphates content.
- Determination of sodium sulphate content.
- Determination of sodium chloride content.

It also sets out, in an annex, a general scheme of analysis.

2 FIELD OF APPLICATION

This International Standard is applicable only to technical sodium alkylsulphates in liquid form, free from other products extraneous to their manufacture. It is not applicable to powders or pastes.

3 REFERENCES

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

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

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

ISO 4315, *Surface active agents — Determination of alkalinity — Titrimetric method.*

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

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

ISO . . . , *Surface active agents — Determination of sulphate content — Titrimetric method.*[2)]

4 SAMPLING

As the material for analysis is a liquid and is thus homogenous at 20 °C, take, prepare and store a laboratory sample of approximately 300 g according to the instructions given in ISO 607.

1) In preparation. (Revision of ISO/R 607.)

2) In preparation.

5 GENERAL PRINCIPLE[1)]

Preparation of an aqueous alcoholic solution of a test portion, from which are isolated the products extractable by light petroleum.

Separation of the sodium alkylsulphates from an aliquot portion of the residual aqueous alcoholic liquid, after repeated evaporation to dryness in the presence of ethanol and final extraction of the anhydrous residue with ethanol.

On separate test portions :

- measurement of pH;
- determination of water content;
- determination of free alkalinity or free acidity;
- determination of total alkalinity;
- determination of sodium sulphate content;
- determination of sodium chloride content.

6 METHODS OF ANALYSIS

6.1 Measurement of pH

Carry out the measurement of pH by the method specified in ISO 4316, on a 10 % (*m/m*) solution of the laboratory sample.

NOTE – If the pH is below 7,0, the sample and the batch it represents will be unstable; therefore, the results for most of the tests will change with time. In these cases, the batch is usually rejected without further analysis.

6.2 Determination of water content

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 total alkalinity

It may happen that, on measuring the pH in accordance with 6.1, a pH that is significantly greater than 7 is observed and that, on determining the alkalinity in accordance with 6.3, an alkali value significantly greater than 0,3 is obtained. In such cases, it is advisable to carry out the determination of the total alkalinity by the method specified in ISO 4315.

6.5 Determination of matter extractable by light petroleum

6.5.1 *Introduction*

Matter extractable by light petroleum consists of sulphur-free products as well as products containing sulphur which, when present, do not ionize in aqueous solution.

6.5.2 *Principle*

Extraction with light petroleum, of the products specified in 6.5.1 from an aqueous alcoholic solution of the test portion, taking into consideration the volatility of the products in question.

6.5.3 *Reagents*

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

6.5.3.1 Sodium sulphate, anhydrous.

6.5.3.2 Ethanol, 96 % (*V/V*) solution.

6.5.3.3 Light petroleum, boiling between 40 and 60 °C.

The residue after evaporation shall not be greater than 0,002 % (*m/m*).

6.5.3.4 Sodium hydroxide, approximately 0,1 N solution.

6.5.3.5 Phenolphthalein, 1 g/l solution in ethanol.

6.5.4 Apparatus

Ordinary laboratory apparatus and

6.5.4.1 Round-bottomed flask, of capacity 250 ml, with ground glass neck.

6.5.4.2 Fractionating column, of 20 cm length and inside diameter approximately 10 mm, with a ground glass cone at its lower end to fit into the neck of the flask (6.5.4.1).

6.5.4.3 Glass condenser, of nominal (jacket) length 160 mm, complying with the requirements of ISO 4799.

6.5.4.4 Three separating funnels, of capacity 500 ml, with ground glass stoppers, complying with the requirements of ISO 4800.

6.5.4.5 One-mark volumetric flask, of capacity 500 ml, complying with the requirements of ISO 1042.

6.5.4.6 Conical flask, of capacity 250 ml, complying with the requirements of ISO 1773.

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

6.5.5 *Procedure*

6.5.5.1 TEST PORTION

Weigh, to the nearest 0,01 g, a mass of the laboratory sample containing approximately 4 g of sodium alkylsulphates, into a 100 ml beaker.

6.5.5.2 DETERMINATION

Place the test portion (6.5.5.1) in one of the 500 ml separating funnels (6.5.4.4) (A) and wash the beaker with water so as to obtain a final volume of approximately 125 ml. Add 50 ml of the ethanol solution (6.5.3.2).

Check that the solution is slightly alkaline to the phenolphthalein solution (6.5.3.5) and, if necessary, make it so with the sodium hydroxide solution (6.5.3.4) until a pale pink colour is obtained with the phenolphthalein indicator.

Shake to render the mixture homogeneous. Allow to cool.

Add 50 ml of the light petroleum (6.5.3.3).

Shake vigorously for approximately 30 s and allow to separate. Add the minimum quantity of the ethanol solution (6.5.3.2) necessary to break any emulsion that may be formed.

Transfer the lower layer into the second separating funnel (6.5.4.4) (B).

Extract with another 50 ml portion of the light petroleum.

Collect the lower layer in the third separating funnel (6.5.4.4) (C) and transfer the upper layer to the first separating funnel (A).

Extract the aqueous alcoholic phase three times more, each time using 50 ml of the light petroleum.

Combine the hydrocarbon phases in the separating funnel (A) and transfer the aqueous alcoholic phase into a 400 ml beaker after the last extraction. Wash the separating funnels (B) and (C) three times, each time using 20 ml of water. Alternatively, a 5 to 10 % (*V*/*V*) solution of ethanol may be used.

Add the washings to the aqueous alcoholic phase in the beaker.

Wash the hydrocarbon extract with successive portions of 15 ml of water until the washings are no longer alkaline; add the washings to the aqueous alcoholic phase.

Heat the aqueous alcoholic phase on a boiling water bath for 10 to 15 min to evaporate the light petroleum and allow to cool.

Ensure that the solution is still alkaline to the phenolphthalein solution, and if necessary render alkaline with the sodium hydroxide solution (6.5.3.4) until a pale pink colour is obtained with the phenolphthalein indicator.

Transfer the solution to the one-mark volumetric flask (6.5.4.5), rinsing the beaker with water and adding the washings to the volumetric flask. Dilute to the mark. This solution, L_1, is used for the determination of the sodium alkylsulphates content.

Transfer the hydrocarbon layer quantitatively into the conical flask (6.5.4.6) containing about 10 g of the sodium sulphate (6.5.3.1). Shake the liquid, allow it to stand for 30 min, and filter through filter paper into the previously tared flask (6.5.4.1) containing a few glass beads. Wash the conical flask, the sodium sulphate and the filter five times, each time using 10 ml of the light petroleum. Pay particular attention to the edges of the filter paper, which should not show any greasy marks.

Fit the fractionating column (6.5.4.2) and condenser (6.5.4.3) to the flask, place the assembly on a hot-plate or in a water bath and distil until almost all the solvent has passed over. Remove the fractioning column, cool to about 30 °C, and eliminate the last traces of solvent by a gentle current of dry, cold air. To do this, maintain the current of air and rotate the flask by hand, in an inclined position, away from the hot-plate or water bath. In this way, the liquid in the flask will spread over the interior in a thin film, facilitating the removal of the last traces of solvent.

To avoid losses, care is necessary in evaporating the solvent, especially while passing the current of air. For this purpose, first weigh the flask, cooled to room temperature and carefully dried, while there is still a detectable odour of solvent. Note the mass, then reheat the flask to approximately 30 °C so as to liquefy the contents and pass a current of air over them for a further 1 min. After cooling and drying the flask, weigh it again and note the mass.

By repeating these operations and plotting successive weighings on a graph it will be noted that, after a rapid fall, the curve reaches a practically horizontal minimum. The second weighing on the horizontal part is regarded as the end of the operation and the mass noted is recorded as that of the final dry residue. Any difference between the last two weighings should be apparent only in the third significant figure.

6.5.6 *Expression of results*

6.5.6.1 METHOD OF CALCULATION

The content of matter extractable by light petroleum is given, as a percentage by mass, by the formula

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

where

m_0 is the mass, in grams, of the test portion (6.5.5.1);

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

6.5.6.2 REPRODUCIBILITY

The difference between results obtained on the same sample, in two different laboratories, should not exceed 1 %.

6.6 Determination of the sodium alkylsulphate content

6.6.1 *Principle*

Evaporation of an aliquot portion of the aqueous alcoholic liquid L_1 from the previous determination (see 6.5.5.2) to one-tenth of its volume, addition of ethanol and evaporation to dryness. Further addition of ethanol and evaporation to dryness (these successive evaporations are made to remove completely the water in the aqueous alcoholic solution). Extraction with hot ethanol of the sodium alkylsulphates from the dry residue thus obtained.

Isolation of the sodium alkylsulphates by evaporation of the solvent. The residue may include some of the sodium chloride and sodium carbonate present, the masses of which are determined and subtracted from the total mass of the residue of the extract.

NOTE – It is very important that the solution remains alkaline throughout this determination.

6.6.2 *Reagents*

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

6.6.2.1 Acetone. The residue on evaporation shall not be greater than 0,005 g per 100 ml.

6.6.2.2 Ethanol, 96 % (*V*/*V*) solution, rendered slightly alkaline with a 0,1 N sodium hydroxide solution in the presence of the phenolphthalein solution (6.6.2.4) as indicator.

6.6.2.3 Sulphuric acid, 0,1 N standard volumetric solution.

6.6.2.4 Phenolphthalein, 1 g/l solution in ethanol.

6.6.3 Apparatus

Ordinary laboratory apparatus and

6.6.3.1 Pipette, of capacity 100 ml, complying with the requirements of ISO 643.

6.6.3.2 Oven, capable of being controlled at 85 to 90 °C.

6.6.3.3 Vacuum desiccator.

6.6.4 *Procedure*

6.6.4.1 Test portion

Using the aqueous alcoholic solution L_1 remaining from the determination of matter extractable by light petroleum (see 6.5), and corresponding to a test portion of mass m_0, introduce by means of the pipette (6.6.3.1) an aliquot portion of 100 ml into a 250 ml beaker.

6.6.4.2 Determination

Reduce the volume of the test portion (6.6.4.1) to about 10 ml by evaporation on a water-bath using a current of air. Add 20 ml of the ethanol solution (6.6.2.2) and evaporate to dryness. Add a further 20 ml of the ethanol solution and evaporate again to dryness. Then add 50 ml of the ethanol solution, break up the residue thoroughly with a glass stirrer and bring to the boil on the water bath. Allow to boil for 3 min and stir from time to time.

Allow the matter insoluble in the ethanol to settle and decant the hot supernatant liquor, filtering it through a filter paper, into a previously tared 250 ml flask containing a few glass beads.

Place the flask on a boiling water bath and evaporate the filtrate, drawing the solvent vapour off through a tube inserted into the neck of the flask.

Add 25 ml of the ethanol solution to the beaker, bring to the boil and allow to boil gently for 2 min. Then allow to settle and filter the supernatant liquid through the same filter paper into the flask.

Repeat this extraction twice more, each time with 25 ml of the hot ethanol solution, transferring most of the insoluble matter to the filter paper with the last addition of ethanol solution. Wash the beaker, the filter and its contents with hot ethanol solution and pour through the filter, paying particular attention to the edges of the filter paper, which should not show any greasy marks.

Continue to evaporate the contents of the flask to dryness, with aspiration. Add 10 ml of the acetone (6.6.2.1).

Evaporate the solvent. To do this, rotate the flask by hand, in an inclined position, while blowing air through it. In this way, the liquid in the flask is spread over the interior in a thin film, facilitating the removal of the last traces of solvent.

Then place the flask in the oven (6.6.3.2), controlled at 85 to 90 °C, for 5 min. Allow it to cool in the vacuum desiccator (6.6.3.3). Repeat the operations of drying, cooling and weighing until constant mass is attained, i.e. until the results of two consecutive weighings carried out at an interval of 15 min do not differ by more than 0,005 g.

Dissolve the residue in water, with gentle heating if necessary, until dissolution is complete. Ensure that this solution is alkaline to phenolphthalein and determine the content of any sodium carbonate[1] carried over during the extraction, by titration with the sulphuric acid solution (6.6.2.3), using the phenolphthalein solution (6.6.2.4) as indicator. Then determine the content of chloride (Cl^-) ions in the remaining aqueous liquor, from any sodium chloride which may have been carried over, using the potentiometric method (see 6.8) and all the remaining aqueous liquor as the test portion.

1) If the results obtained for total alkalinity (see 6.4) do not differ from those for free alkalinity (see 6.3), it is not necessary to determine the sodium carbonate content.