INTERNATIONAL STANDARD 2870

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Surface active agents — Detergents — Anionic-active matter hydrolyzable under acid conditions — Determination of hydrolyzable and non-hydrolyzable anionic-active matter

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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 2870 was drawn up by Technical Committee ISO/TC 91, *Surface active agents,* and circulate to the Member Bodies in August 1972.

It has been approved by the Member Bodies of the following countries :

Austria	Ireland	Spain
Belgium	Japan	Switzerland
Egypt, Arab Rep. of	Mexico	Thailand
France	New Zealand	Turkey
Germany	Poland	United Kingdom
Hungary	Romania	U.S.A.
India	South Africa, Rep. of	U.S.S.R.

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

No Member Body expressed disapproval of the document.

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Surface active agents — Detergents — Anionic-active matter hydrolyzable under acid conditions — Determination of hydrolyzable and non-hydrolyzable anionic-active matter

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination, in detergents, of anionic-active matter hydrolyzable under acid conditions.

This active matter includes alkyl sulphates and hydroxysulphates alkylphenol and fatty alcohol ethoxysulphates. The method is applicable to the analysis of mixtures containing hydrolyzable and non-hydrolyzable anionic-active matter.

The molar mass of the two types of active matter must be known or previously determined, if their content is expressed as a percentage by mass. If the detergent contains perborate, this must be destroyed before the hydrolysis.

2 REFERENCE

ISO 2771, *Surface active agents — Detergents — Determination of anionic-active matter (direct two-phase titration procedure).*

3 PRINCIPLE

Titration of an aliquot portion of a sample solution with benzethonium chloride solution according to the direct two-phase titration procedure described in ISO 2271.

Hydrolysis, by refluxing under acid conditions, of a second aliquot portion of the sample solution after destruction, if necessary, of any perborate in the sample by the addition of sodium sulphite.

Titration of unhydrolyzed anionic-active matter with benzethonium chloride solution as before.

Calculation of the contents of hydrolyzable and non-hydrolyzable anionic-active matter from the results obtained.

4 REAGENTS

The water used shall be distilled water or water of at least equivalent purity.

In addition to the reagents mentioned in ISO 2271 and given below as a reminder :

4.1 Chloroform, ρ_{20} 1,48 g/ml, distilling between 59,5 and 61,5 °C.

4.2 Sulphuric acid, 5 N solution.

4.3 Sulphuric acid, 1,0 N solution.

4.4 Sodium hydroxide, 1,0 N standard volumetric solution.

4.5 Sodium lauryl sulphate, 0,004 M standard volumetric solution.

4.6 Benzethonium chloride, 0,004 M standard volumetric solution.

4.7 Phenolphthalein solution.

4.8 Mixed indicator solution.

the following reagents are necessary :

4.9 Sulphuric acid, 10 N solution.

4.10 Sodium hydroxide, 10 N solution.

4.11 Sodium hydroxide, 1 N solution.

4.12 Sodium sulphite, 2 % solution.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Conical flask, 250 ml, with a conical ground glass joint.

5.2 Reflux condenser, water-cooled, with a conical ground glass joint at the bottom.

6 PROCEDURE

6.1 Determination of total anionic-active matter

Carry out the determination of total anionic-active matter present in the sample by the procedure described in ISO 2271.

6.2 Determination of hydrolyzable anionic-active matter

By means of a pipette, transfer a second aliquot portion of 15 ml of the anionic-active matter solution to the conical flask (5.1). Add, by means of a pipette, 25 ml of the sulphuric acid solution (4.9) and a few anti-bumping granules.

NOTE – If the sample contains perborate, also add to the conical flask 10 ml of the sodium sulphite solution (4.12).

Attach the water-cooled reflux condenser (5.2), well washed with water, to the conical flask and reflux for 3 h. Apply heat cautiously at the start to avoid excessive foaming.

At the end of the reflux period of 3 h, allow to cool, wash down the water-cooled reflux condenser well with at least 5 ml of water, detach the conical flask and wash the ground glass joints with a little water, collecting the washings in the conical flask.

Add a few drops of the phenolphthalein solution (4.7) and neutralize with the sodium hydroxide solution (4.10); add most of the sodium hydroxide solution at once and then complete the neutralization drop by drop with the sodium hydroxide solution (4.11).

Transfer 15 ml of the chloroform (4.1) and 10 ml of the mixed indicator solution (4.8) to the conical flask, stopper and shake well.

Titrate with the benzethonium chloride solution (4.6) as described in ISO 2271.

NOTE – The absence of non-hydrolyzable anionic-active matter after the hydrolysis may be checked by the addition of 1 ml of the benzethonium chloride solution (4.6). The pink coloured chloroform layer should not appear.

7 EXPRESSION OF RESULTS

7.1 Calculations

7.1.1 *Anionic-active matter hydrolyzable under acid conditions*

The content, as a percentage by mass, is equal to

$$\frac{(V_0 - V_1) \times T \times 1\,000 \times M_1 \times 100}{1\,000 \times 25 \times m} = \frac{(V_0 - V_1) \times T \times M_1 \times 4}{m}$$

The amount, expressed in milli-equivalents per gram, is equal to

$$\frac{(V_0 - V_1) \times T \times 1\,000}{25 \times m} = \frac{(V_0 - V_1) \times T \times 40}{m}$$

where the symbols have the meanings given in 7.1.2.

7.1.2 *Anionic-active matter non-hydrolyzable under acid conditions*

The content, as a percentage by mass, is equal to

$$\frac{V_1 \times T \times 1\,000 \times M_2 \times 100}{1\,000 \times 25 \times m} = \frac{V_1 \times T \times M_2 \times 4}{m}$$

The amount, expressed in milli-equivalents per gram, is equal to

$$\frac{V_1 \times T \times 1\,000}{25 \times m} = \frac{V_1 \times T \times 40}{m}$$

where

M_1 is the molar mass of the anionic-active matter hydrolyzable under acid conditions;

M_2 is the molar mass of the anionic-active matter non-hydrolyzable under acid conditions;

m is the mass, in grams, of the test portion;

T is the normality or the molarity of the benzethonium chloride solution (4.6);

V_0 is the volume, in millilitres, of the benzethonium chloride solution (4.6) used for the titration of total anionic-active matter;

V_1 is the volume, in millilitres, of the benzethonium chloride solution (4.6) used for the titration of anionic-active matter after acid hydrolysis.

7.2 Repeatability

The difference found between the results of two determinations carried out on the same sample simultaneously or in rapid succession by the same analyst using the same apparatus should not exceed 2 % of the mean value.

7.3 Reproducibility

The difference between the results obtained on the same sample in two different laboratories should not exceed 4 % of the average value.

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.