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Surface active agents — Detergents — Anionic-active matter stable to acid hydrolysis — Determination of trace amounts

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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 2868 was drawn up by Technical Committee ISO/TC 91, *Surface active agents,* and circulated to the Member Bodies in August 1972.

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

Austria
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Mexico
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Poland
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Spain
Switzerland
Thailand
Turkey
United Kingdom
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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 stable to acid hydrolysis — Determination of trace amounts

1 SCOPE

This International Standard specifies a method for the determination of small quantities of stable anionic-active matter present in detergents, in the presence of a large excess of hydrolyzable anionic-active matter which will hydrolyze under conditions of acid reflux.

2 FIELD OF APPLICATION

This method is applicable to the determination of cyclic sulphonates in alkylphenol ether sulphates and of small amounts of sulphonates in sulphated fatty alcohols.

The molar mass of the anionic-active matter must be known or previously determined, if its content is expressed as a percentage by mass.

3 REFERENCE

ISO 2271, *Surface active agents — Detergents — Determination of anionic-active matter (direct two-phase titration procedure).*

4 PRINCIPLE

Hydrolysis, by refluxing under acid conditions, of anionic-active matter.

Determination of non-hydrolyzable anionic-active matter by the direct two-phase titration procedure described in ISO 2271.

5 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 :

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

5.2 **Sulphuric acid,** 5 N solution.

5.3 **Sulphuric acid,** 1,0 N solution.

5.4 **Sodium hydroxide,** 1,0 N standard volumetric solution.

5.5 **Sodium lauryl sulphate,** 0,004 M standard volumetric solution.

5.6 **Benzethonium chloride,** 0,004 M standard volumetric solution.

5.7 **Phenolphthalein solution.**

5.8 **Mixed indicator solution.**

The following reagents are necessary :

5.9 **Sulphuric acid,** 2 N solution.

5.10 **Sodium hydroxide,** 10 N solution.

5.11 **Sodium hydroxide,** 1 N solution.

6 APPARATUS

Ordinary laboratory apparatus, and

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

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

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 1 mg, a quantity of sample containing between 0,5 and 2 g of anionic-active matter to be subjected to hydrolysis, into the conical flask (6.1).

7.2 Determination

Add to the test portion, by means of a pipette, 25 ml of the sulphuric acid solution (5.9) and a few anti-bumping granules.

Attach the water-cooled reflux condenser (6.2), 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 (5.7) and neutralize with the sodium hydroxide solution (5.10); add most of the sodium hydroxide solution at once, and then complete the neutralization drop by drop with the sodium hydroxide solution (5.11).

Remove the major part of the inorganic salts formed during neutralization by the addition of ethanol. Filter the precipitated salts and evaporate the filtrate to dryness to remove the ethanol. Add water (approximately 25 ml) to the residue in the conical flask (6.1) and then add 15 ml of the chloroform (5.1) and 10 ml of the mixed indicator solution (5.8), stopper the conical flask and shake well.

Titrate with the benzethonium chloride solution (5.6) as described in ISO 2271.

8 EXPRESSION OF RESULTS

8.1 Calculation

The content, as a percentage by mass, of non-hydrolyzable anionic-active matter is equal to

$$\frac{V \times T \times M \times 100}{1\,000\,m} = \frac{V \times T \times M}{10\,m}$$

The amount of anionic-active matter, expressed in milli-equivalents per gram, is equal to

$$\frac{V \times T}{m}$$

where

M is the molar mass of the non-hydrolyzable anionic-active matter;

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

T is the normality or the molarity of the benzethonium chloride solution (5.6);

V is the volume, in millilitres, of the benzethonium chloride solution used for the titration.

8.2 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 1,5 % of the mean value.

8.3 Reproducibility

The difference between the results obtained on the same sample in two different laboratories should not exceed 3 % of the mean value.

9 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.