International Standard 2870

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

Agents de surface — Détergents — Détermination de la matière active anionique hydrolysable et non hydrolysable en milieu acide

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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 2870 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 2870-1973), of which it constitutes a minor revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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

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1 Scope and field of application

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

This active matter includes alkyl sulfates and hydroxysulfates and alkylphenol and fatty alcohol ethoxysulfates.

The mean relative molecular 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 References

ISO 607, *Surface active agents — Detergents — Methods of sample division.*

ISO 2271, *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 specified 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 sulfite.

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

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of 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 Sulfuric acid, 245 g/l solution.

4.3 Sulfuric acid, 49 g/l solution.

4.4 Sodium hydroxide, standard volumetric solution, c(NaOH) = 1,0 mol/l.

4.5 Sodium lauryl sulfate, standard volumetric solution, $c[CH_3(CH_2)_{11}OSO_3Na]$ = 0,004 mol/l.

4.6 Benzethonium chloride, standard volumetric solution, $c(C_{27}H_{42}ClNO_2 \cdot H_2O)$ = 0,004 mol/l.

4.7 Phenolphthalein, 10 g/l ethanolic solution.

4.8 Mixed indicator solution.

the following reagents are necessary:

4.9 Sulfuric acid, 490 g/l solution.

4.10 Sodium hydroxide, 400 g/l solution.

4.11 Sodium hydroxide, 40 g/l solution.

4.12 Sodium sulfite, 20 g/l solution.

5 Apparatus

Ordinary laboratory apparatus and

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

5.2 Reflux condenser, water-cooled, with a conical ground glass joint at the bottom, fitting on to the conical flask (5.1).

6 Sampling

The laboratory sample of detergents shall be prepared and stored in accordance with the instructions given in ISO 607.

7 Procedure

7.1 Test portion and test solution

Weigh, to the nearest 0,001 g, a sample which contains 3 to 5 milli-equivalents of anionic-active matter, and dissolve in 100 ml of water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask and dilute to the mark (test solution A).

7.2 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 on an aliquot portion of 25 ml of test solution A (7.1).

7.3 Determination of hydrolyzable anionic-active matter

By means of a pipette, transfer a second aliquot portion of 25 ml of the solution A (7.1) to the conical flask (5.1). Add, by means of a pipette, 5 ml of the sulfuric acid solution (4.9) and a few anti-bumping granules. [If the sample contains perborate, also add to the conical flask 10 ml of the sodium sulfite 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 joint 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.

8 Expression of results

8.1 Calculations

8.1.1 Anionic-active matter hydrolyzable under acid conditions

The content, expressed as a percentage by mass, is given by the formula

$$\frac{(V_0 - V_1) \times c \times 1\,000 \times M_{r1} \times 100}{1\,000 \times 25 \times m}$$

$$= \frac{(V_0 - V_1) \times c \times M_{r1} \times 4}{m}$$

The molality, expressed in milli-equivalents per gram, is given by the formula

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

$$= \frac{(V_0 - V_1) \times c \times 40}{m}$$

where the symbols have the meanings given in 8.1.2.

8.1.2 Anionic-active matter non-hydrolyzable under acid conditions

The content, as a percentage by mass, is given by the formula

$$\frac{V_1 \times c \times 1\,000 \times M_{r2} \times 100}{1\,000 \times 25 \times m}$$

$$= \frac{V_1 \times c \times M_{r2} \times 4}{m}$$

The molality, expressed in milli-equivalents per gram, is given by the formula

$$\frac{V_1 \times c \times 1\,000}{25 \times m}$$

$$= \frac{V_1 \times c \times 40}{m}$$

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where

M_{r1} is the mean relative molecular mass of the anionic-active matter hydrolyzable under acid conditions;

M_{r2} is the mean relative molecular mass of the anionic-active matter non-hydrolyzable under acid conditions;

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

c is the actual concentration, expressed in moles per litre, of the benzethonium chloride solution (4.6);

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

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

8.2 Precision

8.2.1 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.

8.2.2 Reproducibility

The difference between the results obtained on the same sample in two different laboratories should not exceed 4 % of the average 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 non included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

10 Bibliography

REID, V. W. *et al.*, *Tenside* **5**, 1968, pp. 90-96.

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