INTERNATIONAL STANDARD

ISO 2870

Third edition
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Surface active agents — Detergents — Determination of anionic-active matter hydrolysable and non-hydrolysable 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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2870 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 276, *Surface active agents*, in collaboration with Technical Committee ISO/TC 91, *Surface active agent*s, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 2870:1986) which has been technically revised.

The following major change was introduced: introduction of the potentiometric two-phase titration.

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

1 Scope

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 any oxidizing agent, this must be destroyed before the hydrolysis.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 607, *Surface active agents and detergents — Methods of sample division*

ISO 2271, *Surface active agents — Detergents — Determination of anionic-active matter by manual or mechanical direct two-phase titration procedure*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

EN 14480, *Surface active agents — Determination of anionic surface active agents — Potentiometric two-phase titration method*

3 Principle

Titration of an aliquot of a sample solution with benzethonium chloride solution according to the direct two-phase titration procedure specified in ISO 2271 or the potentiometric two-phase titration specified in EN 14480.

Hydrolysis, by refluxing under acidic conditions, of a second aliquot of the sample solution after destruction, if necessary, of any oxidizing agent in the sample by the addition of sodium sulfite.

Titration of non-hydrolyzed anionic-active matter as before.

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

4 Reagents

4.1 General.

WARNING — The procedures described in this International Standard involve the use of hazardous materials. The necessary precautions as described in regulations covering the handling of hazardous substances should be taken. Technical, organizational and personal protection measures should be observed.

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade that have been checked in advance as to not interfere with the analytical results and water complying with grade 1 as defined in ISO 3696.

4.2 Sulfuric acid, $c(H_2SO_4)$ = 490 g/l solution (CAS number: 7664-93-9).

4.3 Sodium sulfite, $c(Na_2SO_3)$ 20 g/l solution (CAS number: 7757-83-7).

4.4 Phenolphthalein, $c(C_{20}H_{14}O_4)$ = 10 g/l ethanolic solution (CAS number: 77-09-8).

4.5 Sodium hydroxide, c(NaOH) = 400 g/l solution (CAS number: 1310-73-2).

4.6 Sodium hydroxide, c(NaOH) = 40 g/l solution (CAS number: 1310-73-2).

4.7 Benzethonium chloride, (Hyamine 1622) standard volumetric solution, $c(C_{27}H_{42}ClNO_2.H_2O)$ = 0,004 mol/l (CAS number: 121-54-0).

In addition, the other reagents mentioned in ISO 2271 or, respectively, EN 14480 shall be used for the active matter titration.

5 Apparatus

Ordinary laboratory apparatus and in addition, the other apparatus mentioned in EN 14480 shall be used for the potentiometric active matter titration and the following:

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 test sample shall be prepared and stored in accordance with ISO 607.

7 Procedure

7.1 Test portion and test solution

Weigh, to the nearest 1 mg, a sample amount which contains 3 to 5 milli-equivalents of anionic-active matter, and dissolve in 30 ml of water. Transfer the solution quantitatively to a 100 ml volumetric flask and dilute to the mark (test solution A).

7.2 Determination of total anionic-active matter

Transfer a 10 ml aliquot of the solution A to a 100 ml volumetric flask and make up to the volume with water. Take a 10 ml aliquot of this solution and carry out the determination of total anionic-active matter present in the sample visually by the procedure described in ISO 2271 or potentiometrically by the procedure described in EN 14480.

7.3 Determination of hydrolyzable anionic-active matter

By means of a pipette, transfer a second aliquot of 10 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 (4.2) and a few anti-bumping granules. If the sample contains oxidizing agents, add also 10 ml of the sodium sulfite solution (4.3).

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 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 (4.4) and neutralize with the sodium hydroxide solution (4.5); add most of the sodium hydroxide solution at once and then complete the neutralization drop wise with the sodium hydroxide solution (4.6) until the solution turns pink.

Quantitatively transfer the content of the conical flask to a 100 ml volumetric flask and dilute to the mark with water.

Take a 10 ml aliquot of this solution and carry out the determination of non-hydrolyzed anionic active matter present in the sample visually by the procedure described in ISO 2271 or potentiometrically by the procedure described in EN 14480.

8 Calculation and expression of results

8.1 Calculations

8.1.1 Anionic-active matter

The content of total anionic active matter c_1, expressed as mmol/100 g, is given by Equation (1):

$$c_1 = \frac{V_1 \times c}{m} \times 100 \times 100 \qquad (1)$$

where

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

c is the actual concentration, expressed in moles per litre, of the benzethonium chloride solution (4.7);

V_1 is the volume, in millilitres, of the benzethonium chloride solution (4.7) used for the titration of total anionic-active matter.

8.1.2 Anionic-active matter non-hydrolyzable under acid conditions

The content of non-hydrolyzable anionic active matter c_2, expressed as mmol/100 g, is given by Equation (2):

$$c_2 = \frac{V_2 \times c}{m} \times 100 \times 100 \quad (2)$$

where

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

c is the actual concentration, expressed in moles per litre, of the benzethonium chloride solution (4.7);

V_2 is the volume, in millilitres, of the benzethonium chloride solution (4.7) used for the titration of anionic-active matter after acid hydrolysis.

8.1.3 Anionic-active matter hydrolyzable under acid conditions

The content of hydrolyzable anionic active matter c_3, expressed as mmol/100 g, is given by Equation (3):

$$c_3 = c_1 - c_2 \quad (3)$$

8.2 Precision

8.2.1 Repeatability limit

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 limit

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 information:

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

b) a reference to this International Standard (ISO 2870);

c) the test results;

d) details of any operation not specified in this International Standard or in the standards to which reference is made, and any operations regarded as optional, as well as any incidents like to have affected the results.

Bibliography

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

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