INTERNATIONAL STANDARD

ISO 8799

Second edition
2009-03-15

Surface active agents — Sulfated ethoxylated alcohols and alkylphenols — Determination of content of unsulfated matter

Agents de surface — Sulfates d'alcools et d'alkylphénols éthoxylés — Détermination de la teneur en matière insulfatée

Reference number
ISO 8799:2009(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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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 8799 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 276, *Surface active agents*, in collaboration with ISO Technical Committee ISO/TC 91, *Surface active agents*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 8799:1988), which has been technically revised.

Surface active agents — Sulfated ethoxylated alcohols and alkylphenols — Determination of content of unsulfated matter

1 Scope

This International Standard specifies a method for the determination of the content of unsulfated matter present in ordinary commercial neutralized products of sulfation of ethoxylated alcohols or alkylphenols [alkyl oxyethylene sulfates (ethoxylated alcohol sulfates) or alkylphenol oxyethylene sulfates (ethoxylated alkylphenol sulfates)] containing an average of not more than 20 oxyethylene groups per molecule.

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

From a methanolic solution of the test portion, the unsulfated matter is separated on an ion-exchange column (filled with a mixture of cation-exchange resins and anion-exchange resins).

The unsulfated matter is recovered from the eluate by evaporation and weighing of the residue.

4 Reagents and materials

4.1 General.

WARNING — The procedures described in this International Standard involve the use of hazardous substances. 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 so as to not interfere with the analytical results, and water of grade 1 as defined in ISO 3696.

4.2 Methanol, CH_3OH, (CAS number: 67-56-1).

4.3 Hydrochloric acid solution, c(HCl), about 1 mol/l, (CAS number: 7647-01-0).

4.4 Sodium hydroxide solution, c(NaOH), about 2 mol/l, (CAS number 1310-73-2).

4.5 Cation-exchange resin, polystyrene sulfonic acid type, 2 % to 3 % crosslinked, 150 µm to 330 µm, hydrogen form.

4.6 Anion-exchange resin, polystyrene quaternary ammonium type, 2 % to 3 % crosslinked, 150 µm to 330 µm, chloride form.

5 Apparatus

Ordinary apparatus and the following:

5.1 Rotary evaporator, with round-bottom flasks of capacity 250 ml.

5.2 Ion-exchange column: open glass column of internal diameter 25 mm and length 200 mm, provided with a 10 mm to 20 mm layer of glass wool or sintered glass filter and with a stopcock.

5.3 Water bath, the temperature of which can be adjusted between 25 °C to 40 °C.

6 Sampling

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

7 Procedure

7.1 Test portion

From the laboratory sample, if necessary homogenized by introducing an appropriate and known quantity of water, weigh, to the nearest 1 mg, into a 100 ml beaker, a quantity of the homogeneous laboratory sample corresponding to 5 mmol of anionic-active matter.

7.2 Preparation of the ion-exchange resins

7.2.1 Anion-exchange resin clean-up

Take 100 g of the anion-exchange resin (4.6) and allow to swell in water for 24 h. Transfer the resin to a suitable column and pass 500 ml of the sodium hydroxide solution (4.4) through the column, followed by about 1 000 ml of water. Then pass 400 ml of the hydrochloric acid solution (4.3) through the column and again wash with enough water until the washings have a pH between 5 and 7. The treated resin may be stored in water.

7.2.2 Cation-exchange resin clean-up

Take 100 g of the cation-exchange resin (4.5) and allow it to swell in water for 24 h. Transfer the resin to a suitable column, pass 500 ml of the hydrochloric acid solution (4.3) through the column and wash with water until the washings have a pH between 5 and 7. The treated resin may be stored in water.

7.3 Final preparation of resins

Take the required amount of anion-exchange resin, prepared as specified in 7.2.1, namely 25 ml per determination, and transfer to a suitable column. Pass five times the volume of the sodium hydroxide solution (4.4) through the column, wash with water until neutral, then wash with 1 to 2 volumes of methanol (4.2).

Take the required amount (25 ml) of cation-exchange resin, prepared as specified in 7.2.2, place it in a suitable column and wash it with twice its volume of methanol (4.2).

7.4 Arrangement of the mixed-bed exchange column

Mix with the help of a glass rod 25 ml of the cation-exchange resin and 25 ml of the anion-exchange resin, prepared as specified in 7.3, in a beaker. Fill the column (5.2) with the mixed resin in small portions, let the mixed resin settle to a volume between 50 ml and 60 ml and wash with 500 ml of methanol (4.2).

7.5 Separation of unsulfated matter

Dissolve the test portion (7.1) in 50 ml of methanol (4.2). Filter off the insoluble matter through a fast-running filter paper above the prepared column (see 7.4). Pass the filtrate through the column at a flow rate of 2 ml/min and collect the eluate in a 500 ml beaker.

Adjust the flow to 3 ml/min and wash with about 450 ml of methanol. Transfer the eluate and the washings in portions to a tared 250 ml round-bottom flask (see 5.1) and evaporate by means of the rotary evaporator (5.1) on the water bath (5.3) at not more than 40 °C, under vacuum.

Rinse the beaker and evaporator with about 40 ml to 50 ml of methanol and allow the methanol to evaporate.

When the flask appears to be free from methanol, leave it on the evaporator for about 15 min. Weigh the flask plus residue, then dry again in the vacuum desiccator for 15 min.

Weigh again and repeat the procedure of drying and weighing until a mass constant to ±3 mg is obtained.

7.6 Check of the ion-exchange resin

In the case of ethoxylated alcohols, it may happen that the exchange of anions is not complete. Dissolve the residue obtained in 7.5 in 20 ml of water and determine the content of total active matter by direct two-phase titration in accordance with ISO 2271 or EN 14480.

If the anion-active matter content exceeds 0,005 mmol, reject the result and repeat the determination with a fresh sample, using a flow rate of less than 2 ml/min and washing with 250 ml of methanol instead of 450 ml or using separate ion-exchange columns.

8 Calculation and expression of results

The unsulfated matter content, w, expressed as a percentage by mass, is given by Equation (1):

$$w = \frac{m_1 \times 100}{m_0} \tag{1}$$

where

m_0 is the mass, in grams, of the test portion (see 7.1);

m_1 is the mass, in grams, of the residue obtained in 7.5.

9 Precision

9.1 Repeatability limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit, r, in more than 5 % of cases.

9.2 Reproducibility limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit, R, in more than 5 % of cases.

10 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 8799);

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 likely to have affected the results.

Annex A
(informative)

Statistical and other data derived from the results of interlaboratory tests

Comparative analyses of two samples with unsulfated mass fractions of 0,6 % and 2,3 %, respectively, carried out in 15 laboratories, have given the following statistical results:

— standard deviation of repeatability, σ_r: 0,18;

— standard deviation of reproducibility, σ_R: 0,39.