INTERNATIONAL STANDARD

ISO 8799

First edition
1988-04-01

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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 : 1988 (E)

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.

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 8799 was prepared by Technical Committee ISO/TC 91, *Surface active agents.*

Printed in Switzerland

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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 607 : 1980, *Surface active agents and detergents — Methods of sample division.*

ISO 2271 : 1972, *Surface active agents — Detergents — Determination of anionic-active matter (Direct two-phase titration procedure).*

ISO 6842 : 1983, *Surface active agents — Polyethoxylated alcohol and alkylphenol sulfates — Determination of total active matter content.*

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

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Methanol.

4.2 Hydrochloric acid, 73 g/l solution.

4.3 Sodium hydroxide, 80 g/l solution.

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

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

5 Apparatus

Ordinary laboratory apparatus and:

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

5.2 Ion-exchange column: Tube of glass of internal diameter 25 mm and length 200 mm, with a constriction at the bottom and provided with a glass stopcock. The ion-exchange resin is supported at the bottom by a 10 to 20 mm layer of glass wool or by a sintered glass filter.

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

6 Sampling

The laboratory sample of surface active agent shall be prepared and stored according to the instructions given in 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 0,001 g, 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

If preferred, the resins may be prepared in amounts smaller than 1 kg with proportionately smaller volumes of reagents.

7.2.1 Anion-exchange resin

Take 1 kg of the anion-exchange resin (4.5) and allow to distend in water for 48 h. Transfer the resin to a suitable column and pass 5 litres of the sodium hydroxide solution (4.3) through the column, followed by 2 to 3 litres of water. Then pass 4 litres of the hydrochloric acid solution (4.2) through the column and again wash with 2 to 3 litres of water. The treated resin may be stored in water.

7.2.2 Cation-exchange resin

Take 1 kg of the cation-exchange resin (4.4) and allow to distend in water for 48 h. Transfer the resin to a suitable column, pass 5 litres of the hydrochloric acid solution (4.2) through the column and wash with water until the washings are neutral. 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 its volume of the sodium hydroxide solution (4.3) through the column, wash with water until neutral, then wash with 1 to 2 volumes of methanol (4.1).

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 (4.1).

7.4 Arrangement of the mixed-bed exchange column

Mix 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, compress the mixed resin with a glass rod to a volume between 50 and 60 ml and wash with 500 ml of methanol (4.1).

7.5 Separation of unsulfated matter

Dissolve the test portion (7.1) in 50 ml of methanol (4.1). Filter off the insoluble mineral salts through a fast-running filter paper above the prepared column (see 7.4). Pass the filtrate through the column 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), set at 25 to 40 °C, under vacuum, using a water-jet suction pump.

Rinse the beaker and the suction tubes of the evaporator with about 40 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 and then remove it and verify by smelling if the methanol is completely evaporated. If this is not the case, repeat the drying on the rotary evaporator for another 15 min. Place the flask in a vacuum desiccator for 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 $\pm$ 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.

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 3 ml/min and washing with 250 ml of methanol instead of 450 ml or using separate ion-exchange columns.

8 Expression of results

8.1 Method of calculation

The unsulfated matter content, expressed as a percentage by mass, is given by the equation

$$NS = \frac{m_1 \times 100}{m_0}$$

where

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

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

8.2 Precision

Comparative analyses of two samples with mean unsulfated matter contents of 0,6 % (m/m) and 2,3 % (m/m), 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

9 Test report

The test report shall include the following information:

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

b) the method used (i.e. reference to this International Standard);

c) the results and the way in which they have been expressed;

d) any operational details which are not specified in this International Standard or in the International Standards to which reference is made, or which are regarded as optional, as well as any incidents liable to have affected the results.

This page intentionally left blank

This page intentionally left blank