SLOVENSKI STANDARD
SIST EN ISO 2871-2:1997

01-december-1997

Površinsko aktivne snovi - Detergenti - Določevanje kationsko aktivnih snovi - 2. del: Nizko molekularne kationsko aktivne snovi (med 200 in 500) (ISO 2871-2:1990)

Surface active agents - Detergents - Determination of cationic-active matter content - Part 2: Cationic-active matter of low molecular mass (between 200 and 500) (ISO 2871-2:1990)

Tenside - Waschmittel - Bestimmung der kationaktiven Substanz - Teil 2: Kationaktive Substanz niedriger Molmasse (zwischen 200 und 500) (ISO 2871-2:1990)

Agents de surface - Détergents - Détermination de la teneur en matiere active cationique - Partie 2: Matiere active cationique a faible masse moléculaire (entre 200 et 500) (ISO 2871-2:1990)

Ta slovenski standard je istoveten z: EN ISO 2871-2:1994

ICS:

71.100.40	Površinsko aktivna sredstva	Surface active agents

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English version

Surface active agents - Detergents - Determination of cationic-active matter content - Part 2: Cationic-active matter of low molecular mass (between 200 and 500) (ISO 2871-2:1990)

Agents de surface - Détergents - Détermination de la teneur en matière active cationique - Partie 2: Matière active cationique à faible masse moléculaire (entre 200 et 500) (ISO 2871-2:1990)

Tenside - Waschmittel - Bestimmung der kationaktiven Substanz - Teil 2: Kationaktive Substanz niedriger Molmasse (zwischen 200 und 500) (ISO 2871-2:1990)

This European Standard was approved by CEN on 1994-08-22. CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

This European Standard has been taken over by the Technical Committee CEN/TC 276 "Surface active agents " from the work of ISO/TC 91 "Surface active agents" of the International Organization for Standardization (ISO).

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 1995, and conflicting national standards shall be withdrawn at the latest by February 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Endorsement notice

The text of the International Standard ISO 2871-2:1990 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 2871-2

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1990-12-15

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Surface active agents — Detergents — Determination of cationic-active matter content —

Part 2:

Cationic-active matter of low molecular mass (between 200 and 500)

Agents de surface — Détergents — Détermination de la teneur en matière active cationique —

Partie 2: Matière active cationique à faible masse moléculaire (entre 200 et 500)

Reference number
ISO 2871-2:1990(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2871-2 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

ISO 2871 consists of the following parts, under the general title *Surface active agents — Detergents — Determination of cationic-active matter content*:

— *Part 1: High-molecular-mass cationic-active matter*

— *Part 2: Cationic-active matter of low molecular mass (between 200 and 500)*

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Surface active agents — Detergents — Determination of cationic-active matter content —

Part 2:
Cationic-active matter of low molecular mass (between 200 and 500)

1 Scope

This part of ISO 2871 specifies a method for the determination of low-molecular-mass cationic-active materials such as monoamines, amine oxides, quaternary ammonium compounds and alkylpyridinium salts which have a main chain of 10 to 22 carbon atoms and not more than 6 other carbon atoms in the cation.

The method is also suitable for other cationic-active materials.

The method is applicable to solids or to aqueous solutions of the active material. The relative molecular mass of the cationic-active matter shall be known or previously determined if its content is expressed as a percentage by mass. If more than one type of cationic-active material is present, an estimate of average relative molecular mass may be used.

The method is not applicable if anionic and/or amphoteric surface active agents are present.

NOTE 1 Low relative molecular mass sulfonates of toluene and xylene present as hydrotropes do not interfere when present in concentrations up to and including 15 % (m/m) with respect to the active material. At higher levels, their influence should be evaluated in each particular case.

Non-ionic surface active agents, soap, urea and the salts of (ethylenedinitrilo)tetraacetic acid do not interfere.

Typical inorganic components of detergent formulations, such as sodium chloride, sulfate, borate, tripolyphosphate, perborate, silicate, etc., do not interfere, but bleaching agents other than perborate shall be destroyed before the analysis, and the sample shall be completely soluble in water.

This part of ISO 2871 should be read in conjunction with ISO 2271.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 2871. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 2871 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

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

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 2271:1989, *Surface active agents — Detergents — Determination of anionic-active matter by manual or mechanical direct two-phase titration procedure.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

3 Principle

The cationic-active matter in a sample is titrated in a two-phase (aqueous chloroform) system against a standard anionic surface-active agent in the presence of an indicator consisting of mixed anionic and cationic dyes. The cationic surface-active agent present in the sample initially reacts with the anionic dye to form a salt which dissolves in the chloroform layer, imparting a blue colour to this layer. During the titration, the anionic surface-active agent displaces the anionic dye and, at the end point, forms a salt with the cationic dye, imparting a greyish-pink colour to the chloroform layer.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of at least equivalent purity complying with the specifications for grade 3 of ISO 3696.

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

4.2 Sodium lauryl sulfate (Sodium dodecyl sulfate) $[CH_3(CH_2)_{11}OSO_3Na]$, standard volumetric solution, $c(C_{12}H_{25}NaO_4S) = 0{,}004$ mol/l.

While preparing the standard volumetric solution as described in 4.2.2, check the purity of the solid sodium lauryl sulfate used as described in 4.2.1.

4.2.1 Determination of purity of sodium lauryl sulfate.

Weigh, to the nearest 1 mg, 5 g ± 0,2 g of the solid product into a 250 ml round bottom flask with ground-glass neck. Add exactly 25 ml of a standard volumetric sulfuric acid solution, $c(0{,}5H_2SO_4) = 1$ mol/l, and reflux using a water condenser. During the first 5 min to 10 min, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask.

In order to avoid excessive foaming, instead of refluxing, the solution may be left on a boiling water bath for 60 min.

After a further 10 min, the solution will become clear and foaming will cease. Reflux for a further 90 min.

Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water.

Add a few drops of ethanolic phenolphthalein solution (concentration 10 g/l), and titrate the solution with sodium hydroxide solution $c(NaOH) = 1$ mol/l.

Carry out a blank test by titrating 25 ml of the 1 mol/l sulfuric acid solution with the 1 mol/l sodium hydroxide solution.

Calculate the purity, τ, expressed as a percentage by mass, of the sodium lauryl sulfate using the formula

$$\frac{28{,}84(V_1 - V_0)c_0}{m_1}$$

where

V_0 is the volume, in millilitres, of the 1 mol/l sodium hydroxide solution used for the blank test;

V_1 is the volume, in millilitres, of the 1 mol/l sodium hydroxide solution used for the test portion of sodium lauryl sulfate taken;

c_0 is the exact concentration, expressed in moles of NaOH per litre, of sodium hydroxide solution used;

m_1 is the mass, in grams, of the test portion of sodium lauryl sulfate taken.

4.2.2 Preparation of standard volumetric sodium lauryl sulfate solution.

Weigh, to the nearest 1 mg, between 1,14 g and 1,16 g of sodium lauryl sulfate and dissolve in 200 ml of water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask (5.3) fitted with a ground-glass stopper, and dilute to the mark with water.

Calculate the exact concentration c_2, expressed in moles of $C_{12}H_{25}NaO_4S$ per litre, of the solution thus obtained, using the formula

$$\frac{m_2\tau}{288{,}4 \times 100}$$

where

m_2 is the mass, in grams, of sodium lauryl sulfate used to prepare the solution;

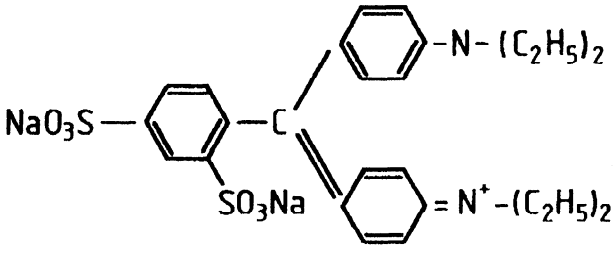
τ has the same meaning as in 4.2.1.

4.3 Mixed indicator solution[1].

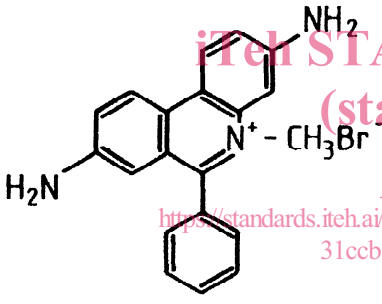
4.3.1 Stock solution.

This solution shall be prepared from acid blue 1 and dimidium bromide.

4.3.1.1 Acid blue 1[2] (Colour Index 42045) (disodium-4′,4″-dinitrilodiethyltriphenylmethane-2,4-disulfonate).



4.3.1.2 Dimidium bromide (3,8-diamino-5-methyl-6-phenylphenanthridinium bromide).



4.3.1.3 Preparation of the stock solution.

Weigh, to the nearest 1 mg, 0,5 g ± 0,005 g of dimidium bromide (4.3.1.2) into a 50 ml beaker, and 0,25 g ± 0,005 g of acid blue 1 (4.3.1.1) into a second 50 ml beaker.

Add between 20 ml and 30 ml of hot 10 % (*V*/*V*) ethanol to each beaker.

Stir until dissolved and transfer the solutions to a 250 ml one-mark volumetric flask. Rinse the beakers into the volumetric flask with the ethanol and dilute to the mark with the ethanol.

4.3.2 Acid solution.

Add 200 ml of water to 20 ml of the stock solution (4.3.1) in a 500 ml one-mark volumetric flask. Add 20 ml of approximately 245 g/l sulfuric acid solution, mix and dilute to the mark with water. Store in the dark.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Flask or **measuring cylinder**, 250 ml capacity, with ground-glass stopper.

5.2 Burette, 25 ml capacity, complying with the specifications for class A of ISO 385-1.

5.3 One-mark volumetric flask, 1 000 ml capacity, with ground-glass stopper, complying with ISO 1042.

6 Sampling

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

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,5 mg, sufficient of the laboratory sample to contain between 0,002 mol and 0,003 mol of cationic-active matter.

NOTE 2 Table 1, which has been calculated on the basis of a relative molecular mass of 360, may be used as a rough guide.

Table 1 — Guide to mass of test portion

Expected cationic-active % (*m*/*m*)	Mass of test portion g
10	10
20	5
100	1

7.2 Determination

Dissolve the test portion (7.1) in water and transfer to the 1 000 ml one-mark volumetric flask (5.3). Dilute to the mark with water and mix well.

By means of a pipette, transfer 25 ml of this solution to the 250 ml flask or measuring cylinder (5.1).

1) This mixed indicator is available commercially in the form of a basic solution, which should be acidified and diluted before use.

2) Acid blue 1, VS blue and disulfine blue VN 150 are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.