INTERNATIONAL STANDARD

ISO 2271

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

Agents de surface — Détergents — Détermination de la teneur en matière active anionique selon une méthode manuelle ou mécanique par titrage direct dans deux phases

Reference number
ISO 2271 : 1989 (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 2271 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 2271 : 1972), of which it constitutes a minor revision.

Annex A of this International Standard is for information only.

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

1 Scope

This International Standard specifies a manual or mechanical method for the determination of anionic-active matter present in detergents.

The method is applicable to solids or to aqueous solutions of the active material. The relative molecular mass of the anionic active matter has to be known.

The method is not applicable if cationic surface active agents are present.

The manual method is thus applicable to the determination of alkylbenzene sulfonates; alkane sulfonates, sulfates and hydroxysulfates; alkylphenol sulfates; fatty alcohol methoxy- and ethoxysulfates; dialkylsulfosuccinates and other active materials containing one hydrophilic group per molecule.

The mechanical method is applicable to all the above-mentioned products provided it gives results comparable with those obtained using the manual method.

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

Soap, urea and salts of (ethylenedinitrilo)tetraacetic acid do not interfere.

In the presence of non-ionic surface agents, their influence should be evaluated in each particular case.

Typical inorganic components of detergent formulations, such as sodium chloride, sulfates, borates, tripolyphosphates, perborates, silicates, etc., do not interfere, but bleaching agents other than perborate should be destroyed before the analysis, and the sample should be completely soluble in water.

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

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

ISO 648 : 1977, *Laboratory glassware — One-mark pipettes.*

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

3 Principle

Determination of the anionic-active matter in a medium consisting of an aqueous and a chloroform phase by titration with a standard volumetric cationic-active solution (benzethonium chloride) in the presence of an indicator which consists of a mixture of a cationic dye (dimidium bromide) and an anionic dye (acid blue 1).

NOTE — The chemical process is as follows : the anionic-active matter forms a salt with the cationic dye which dissolves in the chloroform to give this layer a reddish pink colour.

In the course of the titration, the benzethonium chloride displaces the dimidium bromide from this salt and the pink colour disappears from the chloroform layer as the dye passes into the aqueous phase. Excess benzethonium chloride forms a salt with the anionic dye, which dissolves in the chloroform layer and colours it blue.

4 Reagents

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

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

4.2 Sulfuric acid, approximately 245 g/l solution.

Carefully add 134 ml of sulfuric acid, ϱ_{20} 1,83 g/ml, to 300 ml of water and dilute to 1 litre.

4.3 Sulfuric acid, standard volumetric solution, $c(1/2\ H_2SO_4)$ = 1,0 mol/l.

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

4.5 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.

Check the purity of the sodium lauryl sulfate and simultaneously prepare the standard volumetric solution.

4.5.1 Determination of purity of sodium lauryl sulfate

Weigh, to the nearest 1 mg, 5 g ± 0,2 g of the product into a 250 ml round bottom flask with ground glass neck. Add exactly 25 ml of the standard volumetric sulfuric acid solution (4.3) 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 the phenolphthalein solution (4.7), and titrate the solution with the sodium hydroxide solution (4.4).

Carry out a blank test by titrating 25 ml of the sulfuric acid solution (4.3) with the sodium hydroxide solution (4.4).

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 sodium hydroxide solution (4.4) used for the blank test;

V_1 is the volume, in millilitres, of 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 the sodium hydroxide solution (4.4);

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

4.5.2 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 4.5.1.

4.6 Benzethonium chloride[1], standard volumetric solution, $c(C_{27}H_{42}ClNO_2) = 0{,}004$ mol/l.

Benzyl dimethyl-2-[2-4(1,1,3,3-tetramethylbutyl)phenoxyethoxy]ethyl ammonium chloride, monohydrate:

$$[(CH_3)_3\text{-}C\text{-}CH_2\text{-}C\text{-}(CH_3)_2\text{-}C_6H_4OCH_2\text{-}CH_2OCH_2\text{-}CH_2\text{-}N\text{-}(CH_3)_2\text{-}CH_2\text{-}C_6H_5]^+ Cl^-.\ H_2O$$

4.6.1 Preparation of the solution

Weigh, to the nearest 1 mg, between 1,75 g and 1,85 g of benzethonium chloride and dissolve in water.

Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask (5.3) fitted with a ground glass stopper, and make up to the mark with water.

NOTE – In order to prepare a 0,004 mol/l solution, dry the benzethonium chloride at 105 °C, allow to cool in a desiccator, weigh 1,792 g to the nearest 1 mg, dissolve in water and dilute to 1 000 ml.

Tests indicate that other cationic reagents, such as cetyl trimethyl ammonium bromide and benzalkonium chloride, give results identical to those obtained using benzethonium chloride. However, these tests have not been carried out in sufficient numbers to make it possible to state that the results will be identical no matter what the product analysed; for this reason, if benzethonium chloride is not available it is permitted to use another reagent provided that this is stated in the test report. However, in case of doubt, and always in case of dispute, only benzethonium chloride shall be used.

4.6.2 Standardization of the solution

By means of a pipette (5.4), transfer 25 ml of the standard volumetric sodium lauryl sulfate solution (4.5) to a bottle or measuring cylinder (5.1) or the titration vessel (5.5), and add 10 ml of water, 15 ml of chloroform (4.1) and 10 ml of the mixed indicator solution (4.8).

Titrate against the benzethonium chloride solution (4.6.1). If a bottle or measuring cylinder (5.1) is used, stopper the bottle or

1) Hyamine 1622 is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.