



SLOVENSKI STANDARD

SIST EN 14480:2004

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Surface active agents - Determination of anionic surface active agents - Potentiometric two-phase titration method

Grenzflächenaktive Stoffe - Bestimmung von anionischen grenzflächenaktiven Stoffen - Potentiometrische Zweiphasen-Titration

Agents de surface - Détermination des agents de surface anioniques - Méthode potentiométrique de titrage dans deux phases

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ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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EUROPEAN STANDARD
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English version

Surface active agents - Determination of anionic surface active agents - Potentiometric two-phase titration method

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Grenzflächenaktive Stoffe - Bestimmung von anionischen grenzflächenaktiven Stoffen - Potentiometrische Zweiphasen-Titration

This European Standard was approved by CEN on 1 July 2004.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Foreword

This document (EN 14480:2004) has been prepared by Technical Committee CEN/TC 276 “Surface active agents”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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EN 14480:2004 (E)

1 Scope

This document specifies a method for the determination of the content of anionic surface active agents in raw materials, defined as being the amount of anionic surface active agents expressed in millimoles per 100 g of product.

NOTE 1 The applicability in products different than those tested should be checked in each particular case.

NOTE 2 In comparison to usual laboratory two-phase titration with visual endpoint determination (see ISO 2271) potentiometric titration offers the advantage of automation, operator-dependent differences in recognising the equivalence point can be neglected, and a non-critical solvent replaces the toxicologically critical chloroform.

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.

prEN 14670, *Surface active agents - Sodium dodecyl sulfate - Analysis method*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

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

3 Principle

Anionic surface active agents are combined with cationic surface active agents to form water-insoluble ion pairs, which are immediately extracted into a water immiscible organic solvent. This fundamental reaction is the basis for the titration of equivalents of ionic surface active agents with an oppositely charged surface active agent standard volumetric solution in the two-phase titration.

This process is supported by intensively stirring the two-phase mixture of aqueous solution and organic phase. The potential which is formed in the emulsion during the titration, is recorded with the help of a solvent-resistant surface active agent sensitive electrode in combination with a silver/silver chloride reference electrode against the amount of titrant added. The equivalence point of the added cationic surface active agent corresponds to that one of the test solution at the inflection point of the titration curve (Annex B).

4 Reagents

4.1 General

WARNING — Your attention is drawn to the regulations covering the handling of hazardous substances. Technical, organisational and personal protection measures should be observed.

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and have been checked in advance as to not interfere with the analytical results and water complying with grade 3 as defined in EN ISO 3696. If the water is purified via ion-exchange resins, ensure that no cationic or anionic species from the resins cause interference.

4.2 Sodium dodecyl sulfate, $C_{12}H_{25}OSO_3Na$, $w(C_{12}H_{25}OSO_3Na) \geq 99\%$ as determined following the method prEN 14670

4.3 Anionic surface active agent standard volumetric solution, $c_a(\text{C}_{12}\text{H}_{25}\text{OSO}_3\text{Na}) = 0,005 \text{ mol/l}$

Weigh 1,455 g of sodium dodecyl sulfate (sodium lauryl sulfate) (4.2) with a known active content to the nearest 1 mg in a conical flask and dissolve in about 500 ml water. Transfer quantitatively the solution into a 1000 ml volumetric flask, make up to the mark with water and mix well.

The concentration of the anionic surface active agent standard volumetric solution (c_a) expressed in millimoles per millilitre is calculated in accordance with the following equation (1):

$$c_a = m \times w / M \times 100 \quad (1)$$

where

m is the mass of sodium dodecyl sulfate (4.2) in grams;

w is the active matter content of sodium dodecyl sulfate (4.2) in grams per 100 g;

M is the molar mass of sodium dodecyl sulfate, in grams per mole (288,4 g/mol).

4.4 Benzethonium chloride (CAS number: 121-54-0), $\text{C}_{27}\text{H}_{42}\text{ClNO}_2$, $w(\text{C}_{27}\text{H}_{42}\text{ClNO}_2) \approx 99 \%$.

NOTE Benzethonium chloride is the short name for benzyldimethyl-2-[2-p(1,1,3,3 tetramethyl-butyl)-phenoxy-ethoxy] ethylammonium chloride.

4.5 Cationic surface active agent, standard volumetric solution, $c = 0,005 \text{ mol/l}$:

Weigh 2,2 g to 2,3 g of benzethonium chloride (4.4) to the nearest 1 mg in a conical flask and dissolve it in about 500 ml water. Transfer quantitatively the solution into a 1 000 ml volumetric flask, make up to the mark with water and mix well. Standardize the solution as specified in 7.1.

Standardize the cationic surface active agent solution (4.5) only when the solution has reached its equilibrium, i.e. after standing for at least one day.

NOTE Cationic surface active agents as benzethonium chloride adsorb on glass surfaces like such of the burette and reagent bottle of the titration unit.

4.6 Potassium chloride solution, KCl , $c(\text{KCl}) = 3 \text{ mol/l}$.

4.7 Sulfuric acid, H_2SO_4 , $c(1/2 \text{ H}_2\text{SO}_4) = 0,5 \text{ mol/l}$ or Hydrochloric acid, HCl , $c(\text{HCl}) = 0,5 \text{ mol/l}$.

4.8 Emulsifier (TEGO Add¹⁾).

NOTE The emulsifier has the task of supporting the formation of a stable emulsion and at the same time of preventing the deposition of the ion associate formed during the titration on the electrode surface.

4.9 Ethanol denatured 96 % (v/v).

4.10 Methyl isobutyl ketone (MIBK) (CAS number: 108-11.2), 4-methyl-2-pentanol, $w(\text{C}_6\text{H}_{12}\text{O}) \geq 99 \%$.

4.11 MIBK/ethanol-mixture

Measure 500 ml MIBK (4.10) and 500 ml ethanol using a volumetric cylinder, transfer into a 1l flask and mix well.

1) TEGO Add is the trade name of a product supplied by Metrohm Ltd. (CH-9101 Herisau, Switzerland). This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent product may be used if it can be shown to lead to the same results.

5 Apparatus

5.1 General

Ordinary laboratory apparatus and the following.

5.2 Automatic potentiometric titration apparatus, with drift controlled data acquisition and dynamic titrimetric dosing equipped with 20 ml piston burette.

5.3 Propeller stirrer equipment

In a potentiometric two-phase titration a thorough blending is required. Hence, a stirring propeller is compulsory. The stirrer should be constructed so that an optimal emulsification of the vessel contents is achieved with a simultaneous low degree of air entrapment. Propeller stirrers shaped like ship screws have proven effective, while magnetic stirrers are not suitable. It is advisable to pay special attention to the geometric arrangement of the immersing parts (electrodes, burette tip, and stirrer). If arranged optimally, no foam is produced, not even with heavy stirring.

5.4 Combined glass pH-electrode

5.5 Solvent-resistant surface active agent -sensitive electrode (Surfactrode Resistant or Surfactrode Refill²⁾).

5.6 Ag/AgCl- double-junction ground joint diaphragm reference electrode, inner and outer chambers filled with potassium chloride solution (4.6).

6 Sampling and preparation of the test solution

6.1 Sampling

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

6.2 Preparation of the test solutions

The sample amount and the concentration of test solution shall be calculated in a way that the consumption of the titrant solution (4.5) used for the titration of 10 ml test solution is approximately 10 ml.

Weigh to the nearest 1mg, the calculated mass of the homogenized sample in a glass beaker, and dissolve it in about 100 ml water. Transfer quantitatively the sample solution into a 250 ml volumetric flask, to minimise foaming add a small quantity of ethanol (4.9). Make up to the mark with water and mix well.

7 Procedure

7.1 Standardization of the cationic surface active agent standard volumetric solution

Transfer 10 ml of the sodium dodecyl sulfate standard volumetric solution (4.3) into the titration vessel and add about 70 ml of water.

Adjust with the sulfuric acid solution (4.7) until the pH value $\text{pH} = 3 \pm 0,2$.

Add 20 ml of MIBK/ethanol-mixture (4.11) and 200 μl of emulsifier (4.8) and stir the mixture intensively for 60 s in order to form a stable emulsion.

2) Surfactrode Resistant and Surfactrode Refill are trade names of apparatus supplied by Metrohm Ltd. (CH-9101 Herisau, Switzerland). This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of these apparatus. Equivalent apparatus may be used if they can be shown to lead to the same results.

Carry out the titration with the cationic surface active agent standard volumetric solution (4.5) under intensive stirring (5.3).

Record the reagent consumption, V , at the inflection point of the titration curve (see Figure B.1).

NOTE Examples for instrument settings are given in Annex A.

Calculate the factor, f , of the cationic surface active agent standard volumetric solution (4.5) by the equation (2):

$$f_i = \frac{c_a \times V_1}{V \times c_b} \quad (2)$$

where

f_i is the determined factor of cationic surface active agent standard volumetric solution (4.5);

c_a is the concentration of the anionic surface active agent standard volumetric solution expressed in millimoles per millilitre (4.3);

c_b is the concentration of the cationic surface active agent standard volumetric solution expressed in millimoles per millilitre (here 0,005 mmol/ml);

V_1 is the volume of anionic surface active agent standard volumetric solution used, in millilitres (here : 10 ml);

V is the consumption of cationic surface active agent standard volumetric solution (4.5), in millilitres.

Repeat the determination at least three times and calculate from the factors f_i the average factor f .

7.2 Determination of the anionic surface active agents

Transfer 10 ml of the test solution (see 6.2) into the titration vessel and add 70 ml water.

Carry out the determination as specified in 7.1.

The anionic surface active agent concentration is calculated according to 8.

7.3 Cleaning, rinsing and conditioning of the measuring apparatus

Rinse the measuring apparatus with ethanol (4.9) after each titration. Condition it by stirring for 20 s in the titration beaker filled with ethanol (4.9).

NOTE For Surfactrode Resistant, it is recommended to carry out three or four titrations before to take into account the results.