SLOVENSKI STANDARD
SIST EN 15109:2007

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Površinsko aktivne snovi - Določanje aktivnih snovi v alkilamidopropil betainih

Surface active agents - Determination of the active matter content of alkylamidopropylbetaines

Grenzflächenaktive Stoffe - Bestimmung des Aktivgehaltes von Alkylamidopropylbetainen

Agents de surface - Détermination de la teneur en matières actives des alkylamidopropylbétaines

Ta slovenski standard je istoveten z: **EN 15109:2006**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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English Version

Surface active agents - Determination of the active matter content of alkylamidopropylbetaines

Agents de surface - Détermination de la teneur en matières actives des alkylamidopropylbétaïnes

Grenzflächenaktive Stoffe - Bestimmung des Aktivgehaltes von Alkylamidopropylbetainen

This European Standard was approved by CEN on 6 October 2006.

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Contents

Foreword

This document (EN 15109:2006) 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of an identical text or by endorsement, at the latest by May 2007, and conflicting national standards shall be withdrawn at the latest by May 2007.

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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the active matter content of alkylamidobetaines in commercial surface active agents.

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.

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

The test sample to be analysed is alkalinized by addition of sodium hydroxide. In this way all substances present are converted into a defined form, namely:

— the betaine into its intermolecular salt form;

— the amidoamine into the free amidoamine;

— the acids (e.g. hydrochloric acid, fatty acids, chloroacetic acids and glycolic acid) into their sodium salt forms.

During the titration with perchloric acid in the non-aqueous medium:

— the betaine is changed into the protonated form;

— the amidoamine is changed into the amidoamine perchlorate;

— the excess sodium hydroxide and sodium salts of the different acids are transformed into weakly dissociated sodium perchlorate.

By using a solvent mixture which enables a good differentiation of varying pK(b) values, it is possible to differenciate the betaine from these accompanying substances.

Salts of chloroacetic acids, glycolic acid, fatty acid and amidoamine do not interfere. Short chain betaines are titrated together with long chain betaines.

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 1 as defined in EN ISO 3696.

4.2 1,4-dioxane, minimum purity 99 % (CAS number: 123-91-1).

WARNING — This substance can cause irreversible effects and eye irritation.

4.3 Methoxyethanol, minimum purity 99 % (CAS number : 32718-54-0).

WARNING — This substance is considered as mutagenic.

4.4 Methanol, minimum purity 99,5 % (CAS number : 67-56-1).

WARNING — This substance is considered toxic by ingestion or inhalation.

4.5 Sodium hydroxide, *c*(NaOH) = 1,0 mol/l (CAS number : 1310-73-2).

4.6 Sodium acetate, minimum purity 99 % (CAS number : 127-09-3).

4.7 Sodium hydroxide/sodium acetate solution

Dissolve 80 g of sodium acetate in aqueous sodium hydroxide (*c*(NaOH) = 1,0 mol/l) into a 1 l volumetric flask. Make up to the volume with the same sodium hydroxide solution. Stopper and mix.

4.8 Potassium hydrogen phthalate, purity (100 ± 0,1) %, (CAS number : 877-24-7).

4.9 Acetic acid, minimum purity 99,8 % (CAS number : 64-19-7).

4.10 Perchloric acid standard volumetric solution, $c(HClO_4)$ = 0,1 mol/l (CAS number: 7601-90-3), in 1,4-dioxane.

Fill approximately 500 ml of dioxane in a 1 000 ml volumetric flask. Add the capacity of an ampoule for the preparation of 0,1 mol/l perchloric acid standard volumetric solution. Make up to the volume with 1,4-dioxane.

For the determination of the concentration of the perchloric acid standard volumetric solution, weigh about 0,18 g of dried potassium hydrogen phthalate to the nearest 0,1 mg into the titration beaker and dissolve in about 100 ml of acetic acid.

Switch the stirrer on, immerse the electrodes and titrate with the perchloric acid standard volumetric solution beyond the potential jump.

Calculate the concentration, f_c, of the perchloric acid standard volumetric solution, in moles per litre, using the Equation (1):

$$fc = \frac{m_0 \times 1000}{M \times V} \tag{1}$$

where

m_0 is the mass of potassium hydrogen phthalate, in grams;

M is the molar mass of potassium hydrogen phthalate (M = 204,23 g/mol);

V is the volume, in millilitres, of perchloric acid standard volumetric solution consumed to the inflection point.

5 Apparatus

Ordinary apparatus and the following.

5.1 Automatic potentiometric titration apparatus, with drift-controlled data acquisition and dynamic titrimetric dosing equipped with a piston burette delivery system of 20 ml capacity.

5.2 pH glass electrode

5.3 Reference electrode Ag/AgCl

The instrument parameters shall be adjusted as to produce a curve similar to that shown in Figure 1.

6 Sampling and preparation of the test sample

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

7 Procedure

WARNING — Perform all titrations specified in a well ventilated hood.

Weigh 1,3 g of the test sample to the nearest 0,1 mg into the beaker. Add 20 ml of methanol and dissolve.

Add 0,5 ml of sodium hydroxide/sodium acetate solution measured with a graduated 1 ml pipette and allow to stand for 5 min at room temperature to react.

Add further 20 ml of methanol and 80 ml of methoxyethanol, both measured with a 100 ml graduated cylinder.

Switch the stirrer on, immerse the electrodes and titrate with the perchloric acid standard volumetric solution beyond the third potential jump. A typical titration curve is shown in Figure 1.

NOTE 1 Bad shaped curves can occasionally be obtained because of the following reasons:

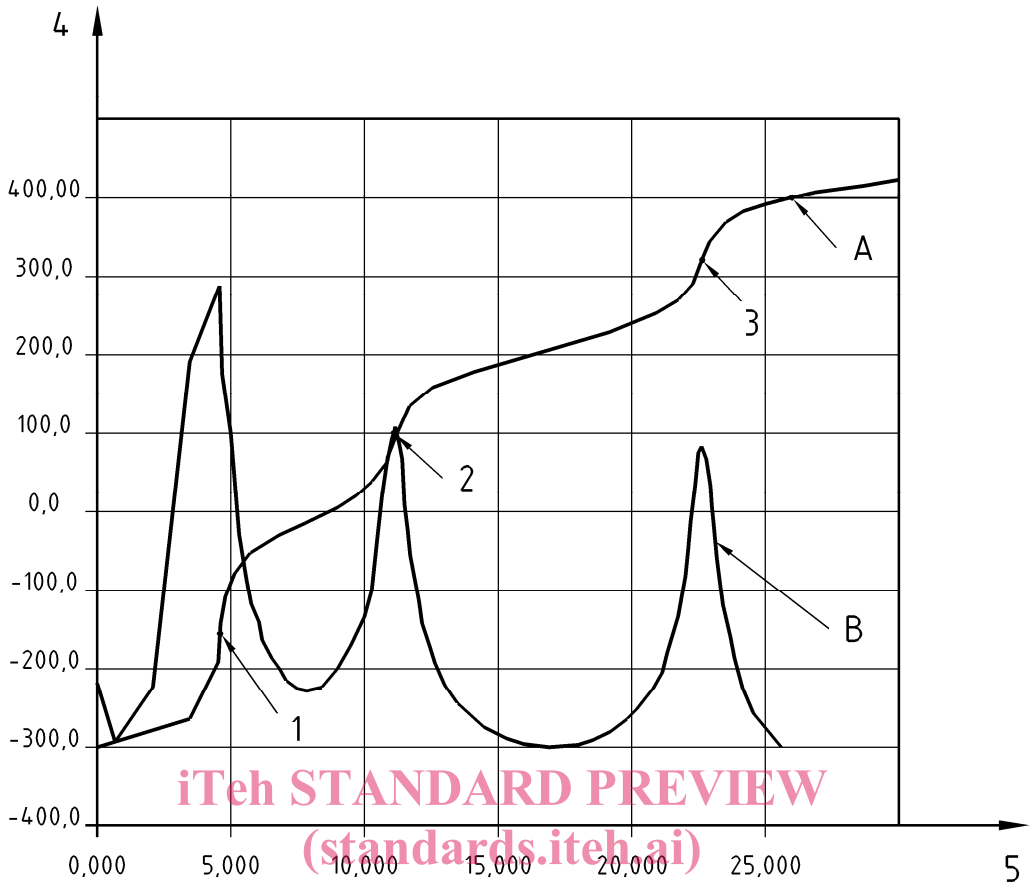
a) static electricity problems (to prevent these problems, special electronic arrangements (e.g. differential potentiometry) should be used);

b) clogging of the electrode diaphragm (remedy by scratching with a needle);

c) overaged perchloric acid standard volumetric solution (remedy by preparing a fresh solution).

NOTE 2 Typical curves start in the negative potential zone (between -100 mV and -400 mV) and show three distinct potential jumps (see Figure 1).

The first jump corresponds to the protonation of excess alkali. The second jump corresponds to the protonation of 1-hydroxy acetate, chloroacetates, fatty acid salts and free amine (i.e. amidoamine). It occurs at positive potential zone. The third jump corresponds to the protonation of betaines. Short chain betaines are also titrated at this point.



Key

1 first inflection point
2 second inflection point
3 third inflection point
4 voltage, in millivolts
5 volume consumed of perchloric acid standard volumetric solution, in millilitres

Figure 1 — Typical titration curve (A) and its first derivative (B)

8 Calculation and expression of results

The content of betaine, w_B, expressed as grams per 100 g, is calculated by the Equation (2):

$$w_B = \frac{(V_3 - V_2) \times M \times f_c}{m \times 10} \quad (2)$$

where

V_3 is the volume consumed of the perchloric acid standard volumetric solution until the third inflection point, in millilitres;

V_2 is the volume consumed of the perchloric acid standard volumetric solution until the second inflection point, in millilitres;

M is the molar mass of the betaine in grams per mole;