SLOVENSKI STANDARD
SIST EN 13560:2002

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Površinsko aktivne snovi - Določanje dušika, vezanega v amidu - Potenciometrijska titracija

Surface active agents - Determination of amide nitrogen content - Potentiometric titration

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an Amid-Stickstoff - Potentiometrische Titration

Agents de surface - Détermination de la teneur en azote sous forme amide - Titrage potentiométrique

Ta slovenski standard je istoveten z: **EN 13560:2001**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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English version

Surface active agents - Determination of amide nitrogen content - Potentiometric titration

Agents de surface - Détermination de la teneur en azote sous forme amide - Titrage potentiométrique

Grenzflächenaktive Stoffe - Bestimmung des Gehaltes an Amid-Stickstoff - Potentiometrische Titration

This European Standard was approved by CEN on 11 November 2001.

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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 Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This European Standard 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of an identical text or by endorsement, at the latest by June 2002, and conflicting national standards shall be withdrawn at the latest by June 2002.

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

The annexes A, B and C are informative.

1 Scope

This European Standard specifies a method for the determination of amide nitrogen content in surface active agents by potentiometric titration. It is not applicable to other basic substances.

2 Normative reference

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

3 Principle

The sum of amide nitrogen and amine nitrogen as well as soap are determined by potentiometric titration of the test sample, dissolved in acetic acid anhydride, with standard trifluoromethane sulfonic acid solution. The content of amine nitrogen as well as soap are determined by potentiometric titration of the test sample, dissolved in glacial acetic acid, with trifluoromethane sulfonic acid standard volumetric solution. From the difference between the two titrations, the amide nitrogen content in grams per 100 g is calculated.

4 Reagents

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.

4.1 Glacial acetic acid, purity 100 %.

4.2 Acetic acid anhydride, purity 98,5 % (minimum).

4.3 Trifluoromethane sulfonic acid standard volumetric solution, c (HSO_3CF_3) = 0,1 mol/l, in acetic acid (4.1).

NOTE Perchloric acid, c ($HClO_4$) = 0,1 mol/l, in glacial acetic acid can also be used.

4.4 Tris(hydroxymethyl)aminomethane (titrimetric standard), dried at (105 ± 2) °C, purity 99 % (minimum).

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 Potentiometric titrating apparatus, comprising a titrator with a combined pH glass electrode, a 20 ml plunger burette and a stirrer.

NOTE An example of instrument settings is given in annex A.

5.2 Beaker, capacity 150 ml.

5.3 Analytical balance, accurate to 0,1 mg.

6 Sampling

The laboratory sample of the surface active agent shall be prepared and stored in accordance with ISO 607.

7 Procedure

7.1 Factor of the trifluoromethane sulfonic acid standard volumetric solution

Weigh approximately 120 mg (m_1) of tris(hydroxymethyl)aminomethane (4.4) to the nearest 0,1 mg into the beaker (5.2) and dissolve in 80 ml of glacial acetic acid (4.1).

Immerse the electrode, stir and titrate with trifluoromethane sulfonic acid standard volumetric solution (4.3).

Record the consumption at the equivalent (inflection) point (volume V_1) ; (see annex B).

Calculate the factor (f) for this solution from the consumption of trifluoromethane sulfonic acid standard volumetric solution (4.3) by the equation (1) :

$$f = \frac{m \times 1000}{M \times V_1 \times c} \qquad (1)$$

where

m_1 is the mass of tris(hydroxymethyl)aminomethane (4.4), in grams ;

M is the molar mass of tris(hydroxymethyl)aminomethane (4.4), M = 121,14 g/mol ;

V_1 is the consumption of trifluoromethane sulfonic acid standard volumetric solution (4.3), in millilitres ;

c is the concentration of the trifluoromethane sulfonic acid standard volumetric solution (4.3), in moles per litre.

7.2 Titration of the amide nitrogen

Weigh from the sample to be tested a quantity (m_2) which contains about 0,5 mmol to 1 mmol of amide nitrogen to the nearest 0,1 mg into the beaker (5.2).

Dissolve the sample in 80 ml of acetic acid anhydride (4.2), under light heating if needed.

Immerse the electrode, stir and titrate with trifluoromethane sulfonic acid standard volumetric solution (4.3).

Record the consumption at the equivalent (inflection) point (volume V_2) ; (see annex B).

7.3 Titration of the total base nitrogen

Weigh from the sample to be tested a quantity (m_3) which contains about 0,5 mmol to 1 mmol of total base nitrogen to the nearest 0,1 mg into the beaker (5.2).

Dissolve the sample in 80 ml of glacial acetic acid (4.1), under light heating if needed.

Immerse the electrode, stir and titrate with trifluoromethane sulfonic acid standard volumetric solution (4.3).

Record the consumption at the equivalent (inflection) point (volume V_3) ; (see annex B).

8 Expression of results

8.1 Calculation of the amide nitrogen content

Calculate the amide nitrogen content, w_{an}, in grams per 100 g, by the equation (2) :

$$w_{an} = \frac{V_2 \times c \times f \times M \times 100}{m_2 \times 1000} - w_{tbn} \qquad (2)$$

where

V_2 is the consumption of the trifluoromethane sulfonic acid standard volumetric solution (4.3) according to 7.2, in millliitres ;

c is the concentration of the trifluoromethane sulfonic acid standard volumetric solution (4.3), in moles per litre ;

f is the factor of the trifluoromethane sulfonic acid standard volumetric solution (4.3) determined according to 7.1 ;

M is the molar mass of nitrogen, M = 14 g/mol ;

m_2 is the mass of the sample according to 7.2, in grams ;

w_{tbn} is the content of total base nitrogen titrated according to 7.3, in grams per 100 g.

The result shall be given to two decimal places.

8.2 Calculation of the total base nitrogen content

Calculate the total base nitrogen content, w_{tbn}, in grams per 100 g, by the equation (3) :

$$w_{tbn} = \frac{V_3 \times c \times f \times M \times 100}{m_3 \times 1000} \qquad (3)$$

where

V_3 is the consumption of the trifluoromethane sulfonic acid standard volumetric solution (4.3) according to 7.3, in millilitres ;

c is the concentration of the trifluoromethane sulfonic acid standard volumetric solution (4.3), in moles per litre ;

f is the factor of the trifluoromethane sulfonic acid standard volumetric solution (4.3) determined according to 7.1 ;

M is the molar mass of nitrogen, M = 14 g/mol ;

m_3 is the mass of the sample according to 7.3, in grams.

The result shall be given to two decimal places.

9 Precision

9.1 Repeatability limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit, r, in more than 5 % of cases.

Typical precision data obtained in a ring test are given in annex C.

9.2 Reproductibility limit

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit, R, in more than 5 % of cases.

Typical precision data obtained in a ring test are given in annex C.

10 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 (a reference to this European Standard) ;

c) the test results ;

d) details of any operations not specified in this European Standard or in the European Standards to which reference is made, and any operations regarded as optional, as well as any incidents likely to have affected the results.