SLOVENSKI STANDARD
SIST EN 13716:2002

01-junij-2002

Površinsko aktivne snovi - Določanje celotnega bazičnega dušika - Potenciometrijska titracija

Surface active agents - Determination of total base nitrogen - Potentiometric titration

Grenzflächenaktive Stoffe - Bestimmung des Gehalts an Gesamt-Basenstickstoff - Potentiometrische Titration

Agents de surface - Détermination de la teneur en azote total basique - Titrage potentiométrique

Ta slovenski standard je istoveten z: **EN 13716:2001**

ICS:

71.100.40 Površinsko aktivna sredstva Surface active agents

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English version

Surface active agents - Determination of total base nitrogen - Potentiometric titration

Agents de surface - Détermination de la teneur en azote total basique - Titrage potentiométrique

Grenzflächenaktive Stoffe - Bestimmung des Gehalts an Gesamt-Basen-Stickstoff - Potentiometrische Titration

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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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of an identical text or by endorsement, at the latest by June 2002, and conflicting national standards shall be withdrawn at the latest by June 2002.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 total base nitrogen content in surface-active agents by potentiometric titration.

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 total base nitrogen, in grams per 100 g, is determined by potentiometric titration with trifluoromethane sulfonic acid in glacial acetic acid.

NOTE The content of total base nitrogen is the sum of the nitrogen in primary, secondary and tertiary amines.

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

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

4.3 Tris(hydroxymethyl)aminomethane (titrimetric standard), dried at (105 ± 2) °C, purity 99,9 % (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 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 and preparation of the sample

The laboratory sample of 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 (4.3) 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 (4.2).

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

Calculate the factor, *f*, of the trifluoromethane sulfonic acid standard volumetric solution (4.2) by the equation (1):

$$f = \frac{m_1 \times 1000}{M \times V_1 \times c} \quad (1)$$

where

m_1 is the mass of tris(hydroxymethyl)aminomethane (4.3), in grams;

M is the molar mass of tris(hydroxymethyl)aminomethane (4.3), M = 121,14 g/mol;

V_1 is the volume of trifluoromethane sulfonic acid standard volumetric solution (4.2) used, in millilitres;

c is the concentration of trifluoromethane sulfonic acid standard volumetric solution (4.2), in moles per litre.

7.2 Titration of the total base nitrogen

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

Dissolve the sample in 80 ml of glacial acetic acid (4.1) by slight heating if needed.

Immerse the electrode, stir and titrate with trifluoromethane sulfonic acid standard volumetric solution (4.2).

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

8 Expression of results

Calculate the total base nitrogen content, w, in grams per 100 g, by the equation (2):

$$w = \frac{V_2 \times c \times f \times M \times 100}{m_2 \times 1000} \quad (2)$$

where

V_2 is the volume of trifluoromethane sulfonic acid standard volumetric solution (4.2) used, in millilitres;

c is the concentration of trifluoromethane sulfonic acid standard volumetric solution (4.2), in moles per litre;

f is the factor of trifluoromethane sulfonic acid standard volumetric solution (4.2) determined in 7.1;

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

m_2 is the mass of the sample, in grams.

The results 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 Reproducibility limit

The absolute difference between two 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.

Annex A
(informative)

Instrument settings

Data given are settings for the Metrohm Titroprocessor 682[1)] and are intended to act as a guideline, only.

Pause 1	20 s
Electrical input	1
Titration rate	1,50 ml/min
Anticipation	40
Stop volume	15,00 ml
Temperature	25 °C
Equivalent point criterion	1

1) Metrohm Titroprocessor 682 is the trade name of on instrument 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 the instrument named.