



SLOVENSKI STANDARD
SIST EN 13717:2003

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Dc j fý]bg_c`U_hj bY'gbcj]!'8c`c Yj Ub^'df]a UfbY[UŽgY_i bXUfbY[U]b'hYfWUfbY[U
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Surface active agents - Determination of primary, secondary and tertiary amino nitrogen -
Potentiometric titration

Grenzflächenaktive Stoffe - Bestimmung des Gehalts an Primär-, Sekundär- und Tertiär-
Aminstickstoff - Potentiometrische Titration

Agents de surface - Détermination de la teneur en azote sous forme amine primaire,
secondaire et tertiaire - Titrage potentiométrique

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71.100.40 Površinsko aktivna sredstva Surface active agents

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EUROPEAN STANDARD

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Surface active agents - Determination of primary, secondary and tertiary amino nitrogen - Potentiometric titration

Agents de surface - Détermination de la teneur en azote sous forme amine primaire, secondaire et tertiaire - Titrage potentiométrique

Grenzflächenaktive Stoffe - Bestimmung des Gehalts an Primär-, Sekundär- und Tertiär-Aminstickstoff - Potentiometrische Titration

This European Standard was approved by CEN on 23 October 2002.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This document (EN 13717:2002) 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 2003, and conflicting national standards shall be withdrawn at the latest by June 2003.

The annexes A, B and C are informative.

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, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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EN 13717:2002 (E)**1 Scope**

This European Standard specifies the methods for the determination of primary, secondary and tertiary amino 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**3.1 The sum of primary and secondary amino nitrogen**

After reaction of the primary and secondary amino nitrogen with carbon disulfide, the resulting dithiocarbamic acids are titrated with sodium hydroxide standard volumetric solution (see also NOTE in 7.1).

3.2 The sum of secondary and tertiary amino nitrogen

After reaction of the primary amino nitrogen with salicylaldehyde to give a Schiff's base, the sum of secondary and tertiary amino nitrogen is determined by potentiometric titration with hydrochloric acid standard volumetric solution.

3.3 Tertiary amino nitrogen

After reaction of the primary and secondary amino nitrogen with acetic acid anhydride, the tertiary amino nitrogen is determined by titration with standard trifluoromethane sulfonic acid solution.

NOTE Alternatively, a reaction with phenylisothiocyanate and a subsequent titration with hydrochloric acid standard volumetric solution can be carried out.

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

4.2 Carbon disulfide, purity 99,7 % (minimum).

WARNING This substance is highly flammable and toxic. It causes serious damage to health by prolonged exposure through inhalation and is a possible risk of impaired fertility as well as harm to the unborn child. This substance irritates to eyes and skin.

4.3 2-Propanol, purity 99,5 % (minimum).

4.4 Hydrochloric acid standard volumetric solution, c (HCl) = 0,1 mol/l, in 2-propanol.

- 4.5 Salicylaldehyde**, purity 99 % (minimum).
- 4.6 Acetic acid anhydride**, purity 98,5 % (minimum).
- 4.7 Trifluoromethane sulfonic acid standard volumetric solution**, $c(\text{HSO}_3\text{CF}_3) = 0,1 \text{ mol/l}$, in glacial acetic acid.

NOTE Perchloric acid, $c(\text{HClO}_4) = 0,1 \text{ mol/l}$, in glacial acetic acid can also be used.

- 4.8 Glacial acetic acid**, purity 100 %.
- 4.9 Phenolphthalein solution**, $\rho = 1 \text{ g/100 ml}$, in ethanol, as indicator.
- 4.10 Phenylisothiocyanate**, purity 98 % (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**, reading to the nearest 0,1 mg.
- 5.4 Magnetic stirrer**, with hot plate.
- 5.5 Oven**.
- 5.6 Pressure bottles**, encased in plastics, capacity 250 ml, with plastics screw caps and polytetrafluoroethylene (PTFE) seal.
- 5.7 Fume cupboard**.

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 Titration of the primary and secondary amino nitrogen

Weigh into the beaker (5.2) a quantity of the sample (m_1) to be tested which contains about 0,5 mmol to 1 mmol of total base nitrogen to the nearest 0,1 mg. Dissolve the sample in 50 ml of 2-propanol (4.3). Add a few drops of phenolphthalein solution (4.9). Then add sodium hydroxide standard volumetric solution (4.1) until the colour changes to faint pink.

Add 5 ml of carbon disulfide (4.2) and stir for 1 min at room temperature (work in a fume cupboard).

Immerse the electrode, stir and titrate with sodium hydroxide standard volumetric solution (4.1).

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Record the amount consumed at the equivalent (inflexion) point (volume V_1) ; (see Annex B).

NOTE To avoid the use of carbon disulfide, the test method in accordance with EN 13716 can be alternatively applied to determine the content of total base nitrogen, w_t , the primary amino nitrogen content is calculated by the equation $w_1 = w_t - w_2$ when w_2 is determined in 7.2.

7.2 Titration of the secondary and tertiary amino nitrogen

Weigh into the beaker (5.2) an amount of the sample (m_2) to be tested which contains about 0,5 mmol to 1 mmol of total base nitrogen to the nearest 0,1 mg. Dissolve the sample in 50 ml of 2-propanol (4.3).

Add 5 ml of salicylaldehyde (4.5) and heat at 80 °C for 15 min (work in a fume cupboard). Allow the solution to cool to room temperature.

Immerse the electrode, stir and titrate with hydrochloric acid standard volumetric solution (4.4).

Record the consumption at the equivalent (inflexion) point (volume V_2) ; (see Annex B).

7.3 Titration of the tertiary amino nitrogen (acetic acid anhydride method)

Weigh into a pressure bottle (5.6) a quantity of the sample (m_3) to be tested which contains about 0,5 mmol to 1 mmol of total base nitrogen to the nearest 0,1 mg. Dissolve the sample in 20 ml of glacial acetic acid (4.8). Then add 30 ml of acetic acid anhydride (4.6).

Seal the pressure bottle (5.6) securely and heat at 60 °C in the oven (5.5) for 30 min.

Allow to cool to room temperature; open the pressure bottle and rinse down the cap and the sides of the bottle with glacial acetic acid (4.8).

Immerse the electrode, stir and titrate with trifluoromethane sulfonic acid standard volumetric solution (4.7).

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

7.4 Titration of the tertiary amino nitrogen (phenylisothiocyanate method)

Weigh into the beaker (5.2) an amount of the sample (m_4) to be tested which contains about 0,5 mmol to 1 mmol of total base nitrogen to the nearest 0,1 mg. Dissolve the sample in 50 ml of 2-propanol (4.3). Then add 5 ml of phenylisothiocyanate (4.10) and heat the solution at 60 °C for 30 min (work in a fume cupboard). Allow the solution to cool to room temperature.

Immerse the electrode, stir and titrate with hydrochloric acid standard volumetric solution (4.4).

Record the consumption at the equivalent (inflexion) point (volume V_4) ; (see Annex B).

8 Expression of results**8.1 Calculation of the sum of primary and secondary amino nitrogen**

Calculate the sum of primary and secondary amino nitrogen, w_1 , expressed as grams per 100 g, by the equation (1) :

$$w_1 = \frac{V_1 \cdot c_1 \cdot f_1 \cdot M \cdot 100}{m_1 \cdot 1000} \quad (1)$$

where

V_1 is the volume of sodium hydroxide standard volumetric solution (4.1) according to 7.1, in millilitres ;

c_1 is the concentration of the sodium hydroxide standard volumetric solution (4.1), in moles per litre ;

f_1 is the factor of the sodium hydroxide standard volumetric solution (4.1) ;

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

m_1 is the mass of sample according to 7.1, in grams.

8.2 Calculation of the sum of secondary and tertiary amino nitrogen

Calculate the sum of secondary and tertiary amino nitrogen, w_2 , expressed as grams per 100 g, by the equation (2) :

$$w_2 = \frac{V_2 \cdot c_2 \cdot f_2 \cdot M \cdot 100}{m_2 \cdot 1000} \quad (2)$$

where

V_2 is the volume of hydrochloric acid standard volumetric solution (4.4) according to 7.2, in millilitres ;

c_2 is the concentration of hydrochloric acid standard volumetric solution (4.4), in moles per litre ;

f_2 is the factor of the hydrochloric acid standard volumetric solution (4.4) ;

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

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

8.3 Calculation of the tertiary amino nitrogen

a) Calculate the tertiary amino-nitrogen content, w_3 , expressed as grams per 100 g, by the equation (3) :

$$w_3 = \frac{V_3 \cdot c_3 \cdot f_3 \cdot M \cdot 100}{m_3 \cdot 1000} \quad (3)$$

where

V_3 is the volume of trifluoromethane sulfonic acid standard volumetric solution (4.7) according to 7.3, in millilitres ;

c_3 is the solution concentration of the trifluoromethane sulfonic acid standard volumetric solution (4.7), in moles per litre ;

f_3 is the factor of the trifluoromethane sulfonic acid standard volumetric solution (4.7) ;

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.