SLOVENSKI STANDARD
SIST EN 13926:2003

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Površinsko aktivne snovi - Alkoksilatni derivati - Določevanje hidroksilne vrednosti - Metoda z N-metil imidazolom

Surface active agents - Alkoxylated derivatives - Determination of hydroxyl value - N-methyl imidazole method

Grenzflächenaktive Stoffe - Alkoxylierte Derivate - Bestimmung der Hydroxylzahl - N-Methylimidazol-Verfahren

Agents de surface - Dérivés alkyloxylés - Détermination de l'indice d'hydroxyle - Méthode a la N-méthylimidazole

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71.100.40	Površinsko aktivna sredstva	Surface active agents

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English version

Surface active agents - Alkoxylated derivatives - Determination of hydroxyl value - N-methyl imidazole method

Agents de surface - Dérivés alkyloxylés - Détermination de l'indice d'hydroxyle - Méthode à la N-méthylimidazole

Grenzflächenaktive Stoffe - Alkoxylierte Derivate - Bestimmung der Hydroxylzahl - N-Methylimidazol-Verfahren

This European Standard was approved by CEN on 29 November 2002.

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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
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Foreword

This document (EN 13926:2003) 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of an identical text or by endorsement, at the latest by September 2003, and conflicting national standards shall be withdrawn at the latest by September 2003.

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, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of hydroxyl value of aliphatic and alicyclic hydroxyl compounds such as polyols, sorbitan esters, plasticisers and surface active agent alcohols and alkoxylates with hydroxyl values greater than 20 mg KOH/g.

2 Normative references

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.*

ISO 4314, *Surface active agents – Determination of free alkalinity or free acidity – Titrimetric method.*

3 Term and definition

For the purposes of this European Standard, the following term and definition apply.

3.1
hydroxyl value *I*(OH)
number of milligrams of potassium hydroxide needed to neutralize the acetic acid required to esterify the hydroxyl groups in 1 g of the material, or the number of milligrams of potassium hydroxide corresponding to the hydroxyl groups in 1 g of the material

4 Principle

A known mass of test sample is esterified with an excess of acetic anhydride/tetrahydrofuran solution using N-methyl imidazole as a catalyst. The excess of acetic anhydride is hydrolysed and then the formed acetic acid istitrated potentiometrically with ethanolic potassium hydroxide standard volumetric solution. The hydroxyl value is calculated from the difference in titration volumes of a blank test and the test sample.

5 Reagents

5.1 General

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.

WARNING — Tetrahydrofuran and N-methylimidazole being hazardous chemicals, all the operations shall be conducted under a well ventilated fume hood.

5.2 Tetrahydrofuran, C_4H_8O.

5.3 Acetic anhydride, $C_4H_6O_3$.

5.4 N-methyl imidazole, $C_4H_6N_2$.

5.5 Potassium hydrogen phthalate, $C_8H_5O_4K$, $w(C_8H_5O_4K) = 99$ % dried at (105 ± 1) °C.

5.6 Ethanol, C_2H_5OH.

5.7 Potassium hydroxide standard volumetric solution in ethanol, *c*(KOH) = 0,5 mol/l.

Weigh 66,0 g of potassium hydroxide with a purity of 85 % (*m/m*) to the nearest 0,1 g and dissolve it in 250 ml freshly boiled water. Then transfer the solution into a 2 l volumetric flask and complete to the mark with ethanol (5.6). Allow the solution to stand for 24 h before standardization.

5.8 Acetylation reagent

Carefully mix 92 ml of tetrahydrofuran (5.2) and 8 ml of acetic anhydride (5.3). This solution is stable for up to one month.

6 Apparatus

6.1 General

Ordinary laboratory apparatus and the following.

6.2 Potentiometer, comprising a titrator with a combined glass/calomel electrode, automatic burette assembly and a magnetic stirrer.

6.3 Water bath, capable of maintaining a temperature of (45 ± 1) °C.

6.4 Air-cooler, with a length of 1 m.

6.5 Analytical balance, with an accurate to 0,000 1 g.

7 Sampling

Sample the material to be tested and store in accordance with ISO 607.

8 Procedure

8.1 Standardization of the ethanolic potassium hydroxide standard volumetric solution

Weigh approximately 1 g of dried potassium hydrogen phthalate (5.5) to the nearest 0,1 mg into a 150 ml beaker and dissolve it in 80 ml of freshly boiled water.

Immerse the electrode and a magnetic stir bar into the beaker, stir and titrate with the potentiometer (6.2) with the ethanolic potassium hydroxide standard volumetric solution (5.7).

Record the volume V_o of the ethanolic potassium hydroxide standard volumetric solution (5.7) at the first inflection point.

Carry out the standardization in triplicate.

8.2 Test portion

Calculate the quantity of the laboratory sample to be weighed, m, expressed in grams, by the equation (1):

$$m = \frac{168}{I(\mathrm{OH})_{\mathrm{E}}} \qquad (1)$$

where

$I(OH)_E$ is the expected hydroxyl value, in milligrams KOH per gram of the material.

8.3 Determination

Fill the calculated mass of the test portion (8.2), m, weighed to the nearest 0,1 mg and not exceeding 10 g into a clean dry 150 ml stoppered conical flask.

Introduce with a pipette 10 ml of the acetylation reagent (5.8).

Add 2 ml of N-methyl imidazole (5.4) into the flask and dissolve the test portion.

Stop up the flask or connect the air cooler (6.4).

Place the flask in a water bath (6.3) at 45 °C for 15 min.

At the end of the reaction time, add 10 ml of water (taking care of washing the stopper or the air-cooler). Homogenise and allow to stand for 2 min.

Introduce the electrode and a magnetic stir bar into the flask, add enough ethanol to cover the tip of the electrode.

Titrate with the potentiometer (6.2) with the ethanolic potassium hydroxide standard volumetric solution (5.7) and note the volume (V_1) at the first inflection point.

Perform the laboratory sample analysis in duplicate.

8.4 Blank tests

Carry out two blank tests at the same time at the determination, using the same reagents, but without the test portion and note the volume at the first inflection as V_2.

9 Expression of results

9.1 Standardisation factor of the ethanolic potassium hydroxide standard volumetric solution

The standardisation factor, f, of the ethanolic potassium hydroxide standard volumetric solution (5.6) is calculated by the equation (2):

$$f = \frac{m_0 \times w_0 \times 10}{V_0 \times M \times c} \qquad (2)$$

where

m_o is the mass of potassium hydrogen phthalate (5.5), in grams;

w_o is the purity of potassium hydrogen phthalate (5.5), in percent (*m/m*);

V_o is the volume of the ethanolic potassium hydroxide standard volumetric solution (5.7) used for the standardisation (8.1), in millilitres;

M is the molar mass of potassium hydrogen phthalate (5.5), M ($C_8H_5O_4K$) = 204,23 g/mol;

c is the concentration of the ethanolic potassium hydroxide standard volumetric solution (5.7), c(KOH) = 0,5 mol/l.

Take as the standardization factor the mean of three titrations performed according to 8.1.

9.2 Hydroxyl value

The experimental hydroxyl value, $I(\mathrm{OH})_o$, in milligrams of KOH per gram of the test portion, is calculated by the equation (3):

$$I(\mathrm{OH})_o = (V_2 - V_1) \times c \times f \times \frac{56{,}11}{m} \qquad (3)$$

where

V_2 is the mean volume of the ethanolic potassium hydroxide standard volumetric solution (5.7) used for the blank tests (see 8.4), in millilitres;

V_1 is the volume of the ethanolic potassium hydroxide standard volumetric solution (5.7) used for the determination (see 8.3), in millilitres;

c is the concentration of the ethanolic potassium hydroxide standard volumetric solution (5.7), c(KOH) = 0,5 mol/l;

f is the mean of standardisation factor of ethanolic potassium hydroxide the standard volumetric solution (5.7) from 9.1;

m is the mass of the test portion (see 8.3), in grams.

For samples containing free acidity, the hydroxyl value, I(OH), is calculated by the equation (4):

$$I(\mathrm{OH}) = I(\mathrm{OH})_o + AV \qquad (4)$$

where

$I(\mathrm{OH})_o$ is the experimental hydroxyl value, in milligrams of KOH per gram of the test portion;

AV is the acidity value of the sample in accordance with ISO 4314 or equivalent, in milligrams of KOH per gram of the test portion.

For samples containing free alkalinity, the hydroxyl value, I(OH), is calculated by the equation (5):

$$I(\mathrm{OH}) = I(\mathrm{OH})_o - BV \qquad (5)$$

where

$I(\mathrm{OH})_o$ is the experimental hydroxyl value, in milligrams of KOH per gram of the test portion;

BV is the alkalinity value of the sample in accordance with ISO 4314 or equivalent, in milligrams of KOH per gram of the test portion.