SLOVENSKI STANDARD
SIST EN 1240:1999

01-maj-1999

Lepila - Določevanje hidroksilnega števila in/ali vsebnosti hidroksilnih skupin

Adhesives - Determination of hydroxyl value and/or hydroxyl content

Klebstoffe - Bestimmung der Hydroxylzahl und/oder des Hydroxylgehaltes

Adhésifs - Détermination de la valeur hydroxyle et/ou de la teneur en hydroxyle

Ta slovenski standard je istoveten z: **EN 1240:1998**

ICS:

83.180 Lepila Adhesives

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EN 1240

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English version

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This European Standard was approved by CEN on 2 January 1998.

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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 Central Secretariat has the same status as the official versions.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Contents

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 193 "Adhesives", the secretariat of which is held by AENOR.

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of an identical text or by endorsement, at the latest by August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

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.

1 Scope

This standard specifies a method to determine the hydroxyl value and/or the hydroxyl content of adhesives, adhesive components, their basic constituents and related products. This method can also be used to determine the hydroxyl value and/or the hydroxyl content of surface protection systems of concrete.

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.

EN 923	Adhesives - Terms and definitions
EN 1066	Adhesives - Sampling
EN 1067	Adhesives - Examination and preparation of samples for testing
EN 1241	Adhesives - Determination of acid value
EN ISO 3696	Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)
EN 21512	Paints and varnishes - Sampling of products in liquid or paste form (ISO 1512:1991)
ISO 385-1	Laboratory glassware - Burettes - Part 1 : General requirements
ISO 648	Laboratory glassware - One-mark pipettes
ISO 1042	Laboratory glassware - One-mark volumetric flasks

3 Definitions

For the purposes of this Standard, the definitions in accordance with EN 923 and the following definitions apply:

3.1 hydroxyl value: The number of milligrams of potassium hydroxide necessary to neutralize the acetic acid which will combine by acetylation with 1 g of the product under test.

3.2 hydroxyl content: Mass fraction in percentage of hydroxyl groups (-OH) contained in the product under test.

4 Principle

Acetylation of the hydroxyl groups is carried out by acetic anhydride on a pyridine solution of the product under test.

The excess acetic anhydride is hydrolysed by the addition of water and the resultant acetic acid titrated with aqueous potassium hydroxide solution.

5 Safety

Persons using this standard shall be familiar with normal laboratory practice.

This standard does not purport to adress all safety problems, if any, associated with its use.

It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European and national regulatory conditions.

6 Reagents

6.1 Acetic anhydride, analytical grade.

6.2 Pyridine, analytical grade.

6.3 Acetylation solution: Add slowly, while mixing, 28 ml of acetic anhydride (see 6.1) to 200 ml of pyridine (see 6.2) and store in a dry atmosphere.

6.4 Water for analytical laboratory use, grade 3 as specified in EN ISO 3696.

6.5 Phenolphthalein (mass concentration ß = 0,1 %) indicator solution: Dissolve 0,1g of phenolphthalein, dilute to 100 ml in pyridine (see 6.2) to 100 ml, and make this solution faintly pink by addition of potassium hydroxide solution.

6.6 Potassium hydroxide, carbonate-free, 1M aqueous solution.

7 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropiate.

7.1 Analytical balance, with a scale division of 0,1 mg.

7.2 Conical flask, capacity 250 ml, with a ground glass stopper.

7.3 One-mark pipette, capacity 20 ml.

7.4 Water-cooled reflux condenser, length 50 cm, inside diameter approximately 9 mm, with ground glass joints to fit the flasks (see 7.2). It shall be fitted on its top with a guard tube containing anhydrous calcium chloride.

7.5 Water bath, controlled at 100 °C.

7.6 Burette, capacity 50 ml, graduated in 0,05 ml.

7.7 Apparatus for potentiometric titration, optional or if necessary, e.g. for dark coloured solutions.

8 Procedure

Take a sample of the product to be tested in accordance with EN 1066. For surface protection systems take a sample in accordance with prEN 21512. Examine and prepare this sample for testing in accordance with EN 1067 or prEN 21512 as appropriate.

Weigh, to the nearest 1 mg, in a 250 ml conical flask (see 7.2) a test portion the mass of which is calculated from the expected hydroxyl value or hydroxyl content by the following formulae:

$$\text{test portion in grams} = \frac{280}{\text{hydroxyl value}} \quad \text{or} \quad \frac{8,5}{\text{hydroxyl content}}$$

NOTE 1: If the approximate hydroxyl content or the approximate hydroxyl value is not known, preliminary tests should be made.

Add to the sample 20 ml of the acetylation solution (see 6.3) with a one-mark pipette (see 7.3) and some dry anti-bumping granules.

NOTE 2: The anti-bumping granules are neutral. In case of doubt, dried the granules should be boiled with water and dried carefully.

Fit the reflux condenser (see 7.4) to the conical flask and ensure the tightness of the joint by moistening with some drops of pyridine (see 6.2).

Place the flask containing the sample in water bath controlled at 100 °C and reflux for 60 min.

Remove the flask from the bath, cool by cold running water to approximately 20 °C and add through the condenser 40 ml of water (see 6.4). Mix thoroughly and cool again.

NOTE 3: The reaction time can be reduced by adding a suitable catalyst for example by the addition of an acid like toluene-4-sulphonic acid to unsaturated polyester resins or basic catalysts like tertiary amines to other polyol compounds.

Comparative tests should be carried out to demonstrate that equivalent values are obtained by the use of the specific catalyst when a reduced reaction time is applied.

Rinse any product adhering to the inner surface of the condenser with water (see 6.4) into the flask. Dissolve any precipitate by addition of 30 ml of pyridine (see 6.2).

Add five drops of phenolphthalein solution (see 6.5) and titrate with the aqueous potassium hydroxide solution (see 6.6) until the colour changes to pink or use a potentiometric apparatus (see 7.7) for the end point detection. Record the volume V_1, in millilitres, of the potassium hydroxide solution used.

Carry out a blank test under the same conditions with the acetylation solution (see 6.3) but without the test portion, and note the volume V_2, in millilitres, of potassium hydroxide solution used.

At least three tests shall be carried out. The mean value shall not differ by more than 0,1% of the hydroxyl content or two units of the hydroxyl value. If this is not the case, further tests shall be carried out until the values of three consecutive tests fulfill the requirement.

NOTE 4: The free acid groups which exist in the product are also neutralized by the potassium hydroxide. Therefore, in the final calculation hydroxyl value and/or hydroxyl content an acid value of the product under test should be taken into account determined in accordance with EN 1241 separately.

9 Expression of results

9.1 Calculation of the hydroxyl value OH_v

From the three test results obtained, calculate the hydroxyl value OH_v by the equation:

$$OH_v = 56{,}1 \cdot M \cdot \frac{(V_2 - V_1)}{m} + A_v$$

where:

M is the molarity of the potassium hydroxide solution;

V_1 is the volume, in millilitres, of the potassium hydroxide solution (see 6.6) required for the determination;