SLOVENSKI STANDARD
kSIST FprEN 1240:2010

01-december-2010

Lepila - Določevanje hidroksilnega števila in/ali deleža 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: **FprEN 1240**

ICS:

83.180	Lepila	Adhesives

kSIST FprEN 1240:2010 **en,fr,de**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD

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FINAL DRAFT
FprEN 1240

September 2010

ICS 83.180

Will supersede EN 1240:1998

English Version

Adhesives - Determination of hydroxyl value and/or hydroxyl content

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

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (FprEN 1240:2010) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by AENOR.

This document is currently submitted to the Unique Acceptance Procedure.

This document will supersede EN 1240:1998.

The main modifications regarding the previous version are in the Foreword, Normative References and 6.5.

SAFETY STATEMENT— Persons using this document should be familiar with the normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory conditions.

ENVIRONMENTAL STATEMENT — It is understood that some of the material permitted in this standard may have negative environmental impact. As technological advantages lead to acceptable alternatives for these materials, they will be eliminated from this standard to the extent possible.

At the end of the test, the user of the standard should take care to carry out an appropriate disposal of the wastes, according to local regulation.

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1 Scope

This European 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

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 923:2005+A1:2008, *Adhesives — Terms and definitions*

EN 1067, *Adhesives — Examination and preparation of samples for testing*

EN 1241, *Adhesives — Determination of acid value*

EN 21512, *Paints and varnishes — Sampling of products in liquid or paste form (ISO 1512:1991)*

EN ISO 1042:1999, *Laboratory glassware — One-mark volumetric flasks (ISO 1042:1998)*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

EN ISO 15605, *Adhesives— Sampling (ISO 15605:2000)*

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*

ISO 648:2008, *Laboratory glassware — One-mark pipettes*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 923:2005+A1:2008 and the following apply.

3.1
hydroxyl value
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 Principles

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 hydrolyzed by the addition of water and the resultant acetic acid titrated with aqueous potassium hydroxide solution.

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5 Reagents

5.1 Acetic anhydride, analytical grade.

5.2 Pyridine, analytical grade.

5.3 Acetylation solution.

Add slowly, while mixing, 28 ml of acetic anhydride (see 5.1) to 200 ml of pyridine (see 5.2) and store in a dry atmosphere.

5.4 Water for analytical laboratory use, grade 3 as specified in EN ISO 3696:1995.

5.5 Phenolphthalein (mass concentration ß = 0,1 %) indicator solution.

Dissolve 0,1g of phenolphthalein, dilute to 100 ml in pyridine (see 5.2), and make this solution faintly pink by addition of potassium hydroxide solution.

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

6 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1:1984, ISO 648:2008 or EN ISO 1042:1999 as appropriate.

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

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

6.3 One-mark pipette, capacity 20 ml.

6.4 Water-cooled reflux condenser, length 50 cm, inside diameter approximately 9 mm, with ground glass joints to fit the flasks (see 6.2).

It shall be fitted on its top with a guard tube containing anhydrous calcium chloride.

6.5 Water bath, controlled at 100 °C.

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

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

7 Procedures

Take a sample of the product to be tested in accordance with EN ISO 15605. For surface protection systems take a sample in accordance with EN 21512. Examine and prepare this sample for testing in accordance with EN 1067 or EN 21512 as appropriate.

Weigh, to the nearest 1 mg, in a 250 ml conical flask (see 6.2) a test portion the mass of which is calculated from the expected hydroxyl value or hydroxyl content by the following formulae:

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

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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 5.3) with a one-mark pipette (see 6.3) and some dry anti-bumping granules.

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

Fit the reflux condenser (see 6.4) to the conical flask and ensure the tightness of the joint by moistening with some drops of pyridine (see 5.2).

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 5.4). 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 5.4) into the flask. Dissolve any precipitate by addition of 30 ml of pyridine (see 5.2).

Add five drops of phenolphthalein solution (see 5.5) and titrate with the aqueous potassium hydroxide solution (see 5.6) until the colour changes to pink or use a potentiometric apparatus (see 6.7) 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 5.3) 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 fulfil 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.

8 Expression of results

8.1 Calculation of the hydroxyl value OH_v

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

$$OH_V = 56{,}1 \times M \times \frac{(V_2 - V_1)}{m} + A_V \qquad (1)$$

where

M is the molarity of the potassium hydroxide solution;

V_1 is the volume, in millilitres, of the potassium hydroxide solution (see 5.6) required for the determination;

V_2 is the volume, in millilitres, of the potassium hydroxide solution (see 5.6) required for the blank test;