SLOVENSKI STANDARD
SIST EN 1245:2011

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Nadomešča:
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Lepila - Določevanje pH

Adhesives - Determination of pH

Klebstoffe - Bestimmung des pH-Wertes

Adhésifs - Détermination du pH

Ta slovenski standard je istoveten z: EN 1245:2011

ICS:

83.180	Lepila	Adhesives

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EUROPEAN STANDARD

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English Version

Adhesives - Determination of pH

Adhésifs - Détermination du pH

Klebstoffe - Bestimmung des pH-Wertes

This European Standard was approved by CEN on 10 March 2011.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 1245:2011) 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of an identical text or by endorsement, at the latest by October 2011, and conflicting national standards shall be withdrawn at the latest by October 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1245:1998.

The main modifications regarding the previous version are in the Title, Foreword, 8.4 and Normative References.

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.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination by electrometry of the pH of adhesives, their basic constituents and related products using a pH meter equipped with a glass and silver reference combined electrode.

This standard is applicable to products supplied in an aqueous medium, and of known concentration, and to products which can be dissolved, dispersed or suspended in water. It is not applicable to adhesives that react with water.

NOTE The accuracy of the method decreases at pH values above 11.

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

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

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
pH
log 1/[H+] where [H+] is the hydrogen ion concentration of the test sample in moles per cubic decimetres (mol/dm^3)

4 Principle

Measurement of the potential difference existing between a glass electrode and a reference electrode immersed in the aqueous dispersion maintained at the specified test temperature, and reading of this difference expressed in pH units, directly on the pH meter scale.

5 Apparatus

5.1 pH meter, with input impedance of at least 10^{12} Ω, a resolution of 0,01 pH unit, and equipped for temperature compensation.

5.2 Combined electrode, a glass electrode surrounded concentrically by a silver reference electrode.

The reference electrolyte (6.3) is kept in electrical contact with the sample by a porous diaphragm. The glass electrode used shall be one recommended by the manufacturer as suitable over the pH range to be encountered. Maintain the electrodes in accordance with the manufacturer's instructions.

NOTE 1 While separate electrodes can be used, it is recommended that they are replaced with combined electrodes. For ecological reasons, silver/silver chloride electrodes are preferred to mercury/calomel electrodes.

NOTE 2 The electrodes function linearly between pH 0 and the appearance of the alkaline error, which, depending on the sodium ion concentration, usually does not begin until the pH exceeds 11.

5.3 Thermostatically controlled water bath, controlled to within ± 1 °C of the specified test temperature which shall be stated.

NOTE In the absence of specific temperature requirements, 23 °C is the preferred temperature.

5.4 Thermometer, graduated to 0,1 °C.

6 Reagents

6.1 Deionised or distilled water, complying with EN ISO 3696:1995, grade 3.

6.2 Commercially available analytical grade buffer solutions of known pH shall be used.

In the absence of commercial buffer solutions, prepare the following solutions using only reagents of known analytical grade and carbon dioxide free distilled water, or water of equivalent purity (grade 3 as defined in EN ISO 3696:1995).

6.2.1 Buffer solution of pH 7

Dissolve 3,40 g of potassium dihydrogen phosphate (KH_2PO_4) and 3,55 g of disodium hydrogen phosphate (Na_2HPO_4) in water and dilute to 1 dm^3 in a volumetric flask. The pH of this solution is 6,87 at 23 °C.

6.2.2 Buffer solution of pH 4

6.2.2.1 Dissolve 10,21 g of potassium hydrogen phthalate ($KOOC\text{-}C_6H_4\text{-}COOH$) in water and dilute to 1 dm^3 in a volumetric flask. The pH of this solution is 4 at 23 °C.

6.2.2.2 The solution shall be stored in a glass or polyethylene vessel that is resistant to chemicals. After one month the solution shall be replaced.

6.2.3 Buffer solution of pH 9

6.2.3.1 Dissolve 3,814 g of sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10\ H_2O$) in water and dilute to 1 dm^3 in a volumetric flask. The pH of this solution when freshly prepared is 9,20 at 23 °C.

6.2.3.2 The solution shall be stored in a glass or polyethylene vessel that is resistant to chemicals and fitted with a soda-lime carbon dioxide trap. After one month the solution shall be replaced.

NOTE Alkaline buffer solutions are unstable; they absorb carbon dioxide from the atmosphere. Where an alkaline buffer has been used for calibration, the accuracy may be verified by means of the buffer solution of pH 4.

6.3 Reference electrolyte, 3 mol/dm^3 solution of potassium chloride saturated with silver chloride.

7 Sampling

Take a significant sample of the adhesive to be tested, in accordance with EN ISO 15605, and prepare it, in accordance with EN 1067.

8 Procedure

8.1 General

Reduction of thermal and electrical hysteresis effects, by ensuring that the temperatures of the test samples, electrodes, demineralised or distilled rinsing water and buffer solutions are as close to one another as possible. The temperatures of the test samples and buffer solutions shall not differ by more than 1 °C.

8.2 Calibration

Calibrate the pH meter in accordance with the manufacturer's instructions. If these are not available, proceed as follows:

8.2.1 Switch on the pH meter and allow the electronic circuit to stabilize.

8.2.2 Select two commercial buffer solutions (6.2). One at pH 7 corresponding to the zero point of the electrode and the other differing from the first by about 3 pH units and of a higher or lower pH in accordance with the sample being tested. Where commercial buffer solutions are not available use the appropriate prepared buffer solutions (6.2.1 and either 6.2.2 or 6.2.3).

8.2.3 Allow the temperature of the buffer solutions, the test sample and the electrode to equilibrate in the water bath (5.3) at the specified test temperature. Record the temperature using the thermometer (5.4) and adjust the temperature correction on the pH meter correspondingly.

8.2.4 Rinse the electrode with water (6.1) and then with the buffer solution of nominal pH 7, so that the liquid runs down the length of the electrode.

8.2.5 Introduce an adequate volume of the same buffer solution into a suitable clean, dry glass or plastic vessel and place in the water bath (5.3). Immerse the electrode in the buffer solution ensuring that the level of the reference electrolyte in the electrode remains about 5 cm higher than the level of the buffer solution (to prevent any contamination of the electrode). Stir gently and allow the reading to stabilize. Adjust the pH meter, using the zero point adjustment control, so that the reading corresponds to the pH of the buffer. Withdraw the electrode and discard the portion of that buffer solution.

8.2.6 Rinse the electrode with water (6.1) followed by the chosen buffer solution as described in 8.2.4.

NOTE Commercial buffer solution pH 10 may be available.

8.2.7 Immerse the electrode in a quantity of the chosen buffer solution as described in 8.2.5. Allow the reading to stabilize before adjusting the meter to the pH of the buffer solution using the gradient adjustment control and without touching the zero point control. Remove the electrode and wash with water (6.1). Discard the portion of the buffer solution.

NOTE Ensure that the electrode gradient is in the range - 55,6 mV/pH to - 61,5 mV/pH unit, i.e. that it is between 95 % and 103 % of the theoretical value (- 58,57 mV/pH unit at 23 °C). If the electrode is outside this range refill with fresh reference electrolyte in accordance with the manufacturer's instructions and repeat the calibration.

8.3 Measurement of the pH of the test sample

8.3.1 Mix the test sample thoroughly to ensure that it is homogeneous.

NOTE Solid, water free or highly viscous adhesives shall be dissolved, dispersed or suspended in water in accordance with the manufacturer’s instructions or, if none are given, in the mass/mass ratio of 1:1.

8.3.2 Rinse the electrode and measuring vessel with water (6.1) and dry with a clean soft tissue. Transfer an adequate volume into the vessel (an additional clean, dry vessel shall be used) and place it in the temperature controlled bath (5.3). Stir gently.