Textiles — Determination of pH of aqueous extract

The European Standard EN 1413:1998 has the status of a British Standard $\,$

ICS 59.080.01



National foreword

This British Standard is the English language version of EN 1413:1998. It supersedes BS 3266:1981, 8.3, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee TCI/25, Chemical properties, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Summary of pages

Amendments issued since publication

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English version

Textiles — Determination of pH of aqueous extract

Textiles — Détermination du pH de l'extrait equeux

Textilien — Bestimmung des pH des wäßrigen Extraktes

This European Standard was approved by CEN on 22 February 1998.

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CEN

European Committee for Standardization Comité Européen de Normalisation Europäisches Komitee für Normung

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

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 248, Textiles and textile products, the Secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by September 1998, and conflicting national standards shall be withdrawn at the latest by September 1998.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

Annex A is a normative part of this European Standard.

Introduction

The pH value of the aqueous extract of textiles affords a useful index to its processing history. In addition, it is becoming more common to demand that the textile, in its various forms, conforms to certain limits in respect of its acidity or alkalinity, often expressed in terms of pH values of aqueous extract.

1 Scope

This standard specifies a method for determining the pH of the aqueous extract of textiles. The method is applicable to textiles in any form.

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 ISO 3696:1987, Water for analytical laboratory use — Specification and test methods.

3 Definition

For the purposes of this standard, the following definition applies:

3.1

pН

the co-logarithm of the hydrogen ion concentration in an aqueous extract

4 Principle

Electrometric measurement of the pH value of the aqueous extract of textiles at room temperature by means of a glass electrode.

5 Reagents

- **5.1** All the reagents used shall be of recognized analytical reagent grade.
- **5.2** Distilled or deionized water, of at least grade 3 in accordance with EN ISO 3696:1987, having a pH value between 5,0 and 7,5.

NOTE The pH value should be verified only the first time. If it is not within the specified range, the water should be redistilled using chemically resistant glassware; acid or organic matters can be removed by distilling water from a solution of 1 g/l potassium permanganate and 4 g/l sodium hydroxide.

Alkalinity (e.g. presence of ammonia) can be suppressed by distilling water from a solution of diluted sulfuric acid.

- **5.3** Potassium chloride solution (0,1 mol/l), prepared using distilled or deionized water (**5.2**).
- **5.4** Buffer solutions, in accordance with annex A, having a pH value similar to that being determined, for calibration of the pH meter before measurement.

6 Apparatus

6.1 Glass or polypropylene stoppered flasks, chemically resistant, for preparation of the aqueous extract.

NOTE The flasks and the beakers should be filled with 0,1 mol/l hydrochloric acid solution two days before use, then washed with distilled water. The glassware used for this test should be set aside for this purpose only and filled with distilled water between tests.

- **6.2** *Mechanical shaker*; providing rotational or reciprocating movement sufficient to obtain a ready exchange of liquid between the interior of the material and the solution used in preparing the extract. A to-and-fro movement at a rate of 60 min⁻¹ or a rotational frequency of 30 min⁻¹ has been found satisfactory.
- **6.3** Beakers, chemically resistant, with a capacity of 150 ml (see note to **6.1**).
- **6.4** *Rods*, chemically resistant (see note to **6.1**).
- **6.5** *pH-meter*, with a glass electrode, capable of measuring to at least 0,1 unit of pH.
- **6.6** Balance, accurate to 0,01 g.
- **6.7** 1 l volumetric flasks, of grade A quality.

7 Preparation of test samples

- **7.1** Take a laboratory sample representative of the bulk of the material and sufficient to provide all the test specimens required. Cut the laboratory sample into pieces having approximately 5 mm sides, or of such a size as to allow the test specimens to wet out rapidly. To avoid contamination, handle the material as little as possible.
- **7.2** Take from the laboratory sample three test specimens of (2 ± 0.05) g.

8 Procedure

8.1 Preparation of the aqueous extract

Prepare the extract in triplicate at room temperature. Place the test specimen and 100 ml of extracting solution [A for water (5.2) or B for potassium chloride solution (5.3)] into a stoppered flask. Agitate the flask for a short period by hand to ensure that the textile is properly wetted out, then shake it mechanically for $2\,\mathrm{h}^{\pm}5\,\mathrm{min}$.

8.2 Measurement of the pH of the aqueous extract

- **8.2.1** Calibrate the pH-meter at the temperature of the extract to be measured. Check the calibration of the pH-meter using two buffer solutions.
- **8.2.2** Immerse the electrode several times in the same solution (A or B) used to prepare the extract until the indicated pH value stabilizes.
- **8.2.3** Decant the first extract into a beaker, immediately immerse the electrode to a depth of at least 10 mm and stir gently with a rod until the pH value stabilizes (do not record the value of this solution).
- **8.2.4** Decant the second extract into a beaker, immediately immerse the electrode, without washing, in the beaker to a depth of at least 10 mm and allow to stand without stirring until the pH value stabilizes. Record this value.
- **8.2.5** Decant the third extract into another beaker. Determine the pH value as described in **8.2.4**.
- **8.2.6** Record the pH values of the second and third extracts as the first and second measurements.

9 Calculation

If the difference between the pH values, expressed to the nearest 0,1 unit, is greater than 0,2, repeat the procedure with other specimens. Calculate the mean value of the two recorded measurements.

10 Test report

The test report shall include the following information:

- a) a reference to this European Standard,
- i.e. EN 1413:1998;
- b) the mean pH value to the nearest 0,1 unit of pH;
- c) the type of extracting solution used (A or B);
- d) the pH of the extracting solution;
- e) the temperature of the solution;
- f) any factor likely to have had an effect on the results, including any resistance to wetting out of the specimen.

11 Precision of the method

Interlaboratory trials were carried out between nine laboratories measuring seven samples. Statistical analysis was carried out and the following results were obtained:

Extracting solution A: Reproducibility extreme

R = 1.7 unit of pH;

Extracting solution B: Reproducibility extreme

R = 1.1 unit of pH.

NOTE Statistical analysis was carried in accordance with ISO 5725-2:1994 Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

Annex A (normative)

Preparation of standard buffer solutions

A.1 All the reagents used shall be of recognized analytical reagent grade. The buffer solutions shall be prepared using water of at least grade 3 in accordance with ISO 3696 and renewed at least once a month.

A.2 pH 4,0 Potassium hydrogen phthalate (0,05 mol/l)

Dissolve 10,21 g of potassium hydrogen phthalate (KHC $_8$ H $_4$ O $_4$) in distilled or deionized water in a 1 l volumetric flask and dilute to the mark. The pH of this solution is 4,00 at 20 °C and 4,01 at 25 °C.

A.3 pH 6,9 Potassium dihydrogen orthophosphate and disodium hydrogen orthophosphate solution (0,02 mol/l)

Dissolve 3,9 g of potassium dihydrogen orthophosphate (KH_2PO_4) and 3,54 g of disodium hydrogen orthophosphate (Na_2HPO_4) in distilled or deionized water in a 1 l volumetric flask and dilute to the mark. The pH of this solution is 6,87 at 20 °C and 6,86 at 25 °C.

A.4 pH 9,2 Disodium tetraborate (0,01 mol/l)

Dissolve 3,80 g of disodium tetraborate decahydrate ($Na_2B_4O_7\cdot 10H_2O$) in distilled or deionized water in a 1 l volumetric flask and dilute to the mark. The pH of this solution is 9,23 at 20 °C and 9,18 at 25 °C.

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