

International Standard



6588

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Paper, board and pulps — Determination of pH of aqueous extracts

Papier, carton et pâtes — Détermination du pH des extraits aqueux

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6588 was developed by Technical Committee ISO/TC 6, *Paper, board and pulps*, and was circulated to the member bodies in August 1978.

It has been approved by the member bodies of the following countries :

ISO 6588:1981

Australia	France	New Zealand
Austria	Germany, F. R.	Norway
Belgium	Hungary	Poland
Brazil	India	Romania
Canada	Italy	South Africa, Rep. of
Chile	Kenya	Spain
China	Korea, Rep. of	Sweden
Czechoslovakia	Libyan Arab Jamahiriya	Switzerland
Egypt, Arab Rep. of	Mexico	United Kingdom
Finland	Netherlands	USSR

The member body of the following country expressed disapproval of the document on technical grounds :

USA

Paper, board and pulps — Determination of pH of aqueous extracts

1 Scope

This International Standard specifies a method for the determination of the pH of an aqueous extract of paper, board or pulps. The pH value is not a quantitative measure of the total content of acid or base in the material extracted.

2 Field of application

This method is applicable to all kinds of paper, board and pulps yielding an extract having a conductivity exceeding 0,2 mS/m when determined in accordance with ISO 6587, except for papers used for electrical purposes. For high purity papers used for electrical purposes, the method used should be that given in IEC Publication 554-2.

3 References

- ISO 186, *Paper and board — Sampling for testing*.
- ISO 287, *Paper and board — Method for the determination of moisture content of paper (oven-drying method)*.
- ISO 638, *Pulps — Determination of dry matter content*.
- ISO 6587, *Paper, board and pulps — Determination of conductivity of aqueous extracts*.¹⁾
- IEC Publication 554-2, *Specification for cellulosic papers for electrical purposes — Part 2 : Methods of test*.

4 Principle

Extraction of a sample of 2 g for 1 h with 100 ml of cold or boiling distilled water. Measurement of the pH of the extract at 20 to 25 °C.

5 Reagents

5.1 Distilled or deionized water

Distilled or deionized water shall be used throughout the test. The conductivity of the water shall not exceed 0,1 mS/m after boiling and cooling as specified in 8.1.2. (See note 2.)

NOTES

1 Usually both distillation and deionization are required. Unless great care is exercised when distilling, and with the materials employed in the condenser and subsequent surfaces with which the condensed vapour may come in contact, the distillate may not reach the required level of conductivity.

If deionization is applied without distillation, there may be a buffering action due to organic acids present, unless the deionization process includes the use of strongly basic ion-exchange material.

2 When it is not possible to obtain water of the specified purity, water with a higher conductivity may be used, but the conductivity of the water used shall be stated in the test report.

5.2 Standard buffer solutions, having pH values such as 4,0, 6,9 and 9,2, commercially available or prepared as described in the annex.

6 Apparatus

Ordinary laboratory apparatus and

6.1 Glassware of chemically resistant glass, flasks with ground glass joints, stoppers, beakers and water-cooled reflux condensers. All glassware shall be carefully rinsed with boiling distilled water (5.1) and allowed to dry before use.

6.2 Electric heater, adjustable to at least 200 W.

6.3 pH meter, fitted with glass and calomel or combined electrodes, capable of being read to at least 0,05 in pH.

6.4 Thermometer.

7 Sampling and preparation of the sample

7.1 Sampling

Sampling of paper and board shall be carried out in accordance with ISO 186.

Sampling of pulp shall be as representative as possible.

1) At present at the stage of draft.

7.2 Preparation of sample

Cut or tear the sample into pieces approximately 5 mm × 5 mm in size from portions that have not been touched by bare hands and ensuring that the sample is placed only on clean surfaces. Mix the pieces thoroughly. The sample shall not at any time be touched with the bare hands. Clean protective gloves shall be worn at all times to protect the sample and the pieces prepared from it. Store the prepared samples in clean covered containers.

7.3 Determination of dry matter content

Determine the dry matter content in accordance with ISO 287 for paper and board and ISO 638 for pulps.

8 Procedure

8.1 Preparation of aqueous extract

8.1.1 Weighing

Weigh, to the nearest 0,1 g, exactly 2 g (oven-dry basis) of the sample (7.2) into a flask of suitable size (6.1) and proceed as specified in 8.1.2 or 8.1.3.

8.1.2 Hot extraction

With a pipette, measure 100 ml of water (5.1) into a separate flask of similar size to that containing the pieces (6.1). Attach the reflux condenser (6.1) and heat the water to almost boiling. Remove the condenser, add the almost boiling water to the flask containing the pieces, attach the condenser to this flask, and boil gently for 1 h.

Cool rapidly to 20 to 25 °C with the condenser in place. Let the fibres settle and then decant the extract into a small beaker (see 6.1). Prepare the extract in duplicate.

8.1.3 Cold extraction

With a pipette, measure 100 ml of water (5.1) into the flask (6.1) and add the pieces (8.1.1). Seal the flask with a ground glass stopper, and leave to stand for 1 h at 20 to 25 °C. Shake the flask at least once during this time. Decant the extract into a small beaker (see 6.1).

8.2 Determination of pH

Calibrate the pH meter (6.3) with two buffer solutions (5.2) having pH values such that the pH of the extract is between the pH values of the buffer solutions. After calibration, rinse the electrodes several times with water (5.1), and once in a small quantity of the extract (8.1.2 or 8.1.3). Check that the temperature of the extract is 20 to 25 °C. Immerse the electrodes in the extract and measure the pH value.

NOTE — The hot extract may be used in the determination of the conductivity in accordance with ISO 6587, provided that the sample is drawn prior to the pH determination. During the measurement of the pH, potassium chloride will diffuse from the calomel electrode thus affecting the conductivity.

Repeat the determination with the duplicate extract.

9 Expression of results

Express the pH to the nearest 0,1 unit as the mean of two determinations. The individual results should not differ by more than 0,2; if they do, repeat the determination on two additional extracts, and report the mean and the range of all measurements.

10 Test report

The test report shall include the following particulars :

- a) all the indications necessary for complete identification of the sample;
- b) reference to this International Standard;
- c) the extraction procedure used (hot or cold);
- d) the results of the test;
- e) the conductivity of the water used, where this is greater than 0,1 mS/m;
- f) any unusual features observed in the course of the test;
- g) any operations not specified in this International Standard or in the International Standards to which reference is made, or regarded as optional, which might have affected the results;
- h) the number of determinations, if more than two.

Annex

Preparation of standard buffer solutions

All the reagents used shall be of recognized analytical reagent grade. The buffer solutions shall be renewed at least once a month.

A.1 pH 4,0

Potassium hydrogen phthalate, 0,05 mol/l solution.

Dissolve 10,21 g of potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) in distilled water in a 1 litre one-mark 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.2 pH 6,9

Potassium dihydrogen orthophosphate and disodium hydrogen orthophosphate solution

Dissolve 3,39 g of potassium dihydrogen orthophosphate

(KH_2PO_4) and 3,54 g of disodium hydrogen orthophosphate (Na_2HPO_4) in distilled water in a 1 litre one-mark 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.3 pH 9,2

Disodium tetraborate, 0,01 mol/l solution.

Dissolve 3,80 g of disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10 \text{H}_2\text{O}$) in distilled water in a 1 litre one-mark 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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