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

Part 1:
Cold extraction

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

Partie 1: Extraction à froid

(Revision of ISO 6588:1981)

ICS 85.040; 85.060

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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 6588-1 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This first edition of ISO 6588-1 cancels and replaces ISO 6588:1981. The 1981 edition has been technically revised and split into two parts: 6588-1 for cold extraction and 6588-2 for hot extraction. There are four major differences compared with the previous edition of ISO 6588:1981:

a) the Standard has been divided into two parts; one dealing with cold extraction and the other with hot extraction;

b) the determination is now carried out on an air dry sample without determination of dry matter content, as the amount of sample is not critical;

c) the extract is filtered;

d) a salt solution is added to speed up the measurement.

ISO 6588 consists of the following parts, under the general title *Paper, board and pulps — Determination of pH of aqueous extracts*:

— *Part 1: Cold Extraction*

— *Part 2: Hot extraction*

The work within ISO/TC 6 has shown that the results obtained with the new Standard are equal to those obtained by the first edition (ISO 6588:1981).

Introduction

The kraft fibre is well known to contain ionisable groups that are fixed to or in the fibre wall. In order to fulfil the electro-neutrality, these groups are balanced by an equivalent number of positive charges, which can be either protons or various metal ions. Especially in pulp suspensions at low ionic strengths this can give rise to a marked uneven distribution of mobile ions between the volume held by the fibre wall and the bulk suspension liquor. This means that the fibre acts as an ion exchanger. These ion exchange phenomena can be modelled very well with the Donnan theory [1,2].

If a relatively clean pulp fibre sample, as for example bleached dried pulp, is diluted in deionised water the result will be a pulp suspension with a very low ionic strength. In such a system most of the cations present, including protons, will be concentrated in the water volume held by the fibre wall. If pH is measured, it will be measured in the bulk suspension liquor. By adding salt to this kind of system the ion exchange phenomena will be decreased and the concentration of different cations will be the same in the water held by the fibre wall and in the bulk suspension liquor. Since the process waters always contain a certain amount of ions such a salt addition will give a more realistic environment when measuring the pH of relatively clean pulp samples.

It is necessary to be aware of these effects when interpreting the measured pH-values of highly purified pulps.

Paper, board and pulps — Determination of pH of aqueous extracts — Part 1: Cold extraction

1 Scope

This International Standard specifies a method for the determination of the pH value defined by the electrolytes extractable by cold water from a sample of paper, board or pulp.

The Standard is applicable to all kinds of paper, board and pulp.

As the quantity of extractable ionic material approaches zero, as in the case of highly purified pulps, the precision of the method becomes poor because of the difficulties encountered in making pH measurements on water containing little electrolytic material.

Since the extraction in this Standard is performed with distilled or deionised water, the pH value measured will sometimes be different (e.g. fully-bleached pulp) from the pH value measured under mill process conditions in which various types of process waters, e.g. chemically treated river water containing electrolytes, are used.

ISO 6588-2, Determination of pH of aqueous extracts – Part 2: Hot extraction, differs from this standard only as regards the extraction conditions. No general guidance can be given as to which of the two procedures (hot or cold) is best suited in a particular situation.

NOTE For cellulosic papers for electrical purposes, the method used should be that given in the International standard IEC 60554-2 (see [3] under bibliography).

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.

ISO 186, *Paper and board - Sampling to determine average quality.*

ISO 3696, *Water for analytical laboratory use. - Specification and test methods.*

ISO 7213, *Pulps - Sampling for testing.*

3 Principle

Extraction of a sample of 2 g for 1 h with 100 ml of cold water of high purity. Filtration of the extract and addition of salt solution. Measurement of the pH value of the extract at 20 °C to 25 °C.

4 Reagents

4.1 Water. Distilled or deionised water shall be used throughout the test. The conductivity of the water must not exceed 0,1 mS/m after boiling for 1 h and cooling to between 20 °C and 25 °C. Instructions for the determination of conductivity are given in ISO 3696.

4.2 Standard buffer solutions, with known pH values near 4, 7 and 9. Such buffer solutions are commercially available. Some examples of suitable buffer solutions are given, and their preparation is described in Annex A.

4.3 Potassium chloride solution, about 1M. Dissolve about 7,4 g of KCl, analytical grade, into 100 ml of freshly boiled, distilled water. Prepare a fresh solution every week.

5 Apparatus and equipment

In addition to ordinary laboratory apparatus and equipment.

5.1 Glassware of chemical resistant glass, flasks with ground glass joints, stoppers, beakers and fritted glass filter. All glassware shall be carefully rinsed with water (4.1) and allowed to dry before use.

5.2 pH meter, fitted with glass and calomel electrodes or with a combined electrode, capable of being read to at least 0,05 pH unit.

6 Sampling and preparation of sample

6.1 Sampling

The sampling procedure to be followed depends on the particular circumstances in each case. If the analysis is being made to evaluate a lot or a consignment of pulp, paper or board, the sample shall be taken in accordance with ISO 7213 or ISO 186, as relevant. If the analysis is made on another type of sample, report the origin of the sample and, if possible, the sampling procedure.

Wear protective gloves when handling the sample.

NOTE – Some gloves are powdered to prevent them from sticking to one another, and this powder may cause contamination of the sample.

6.2 Preparation of sample

Do not touch the sample with bare hands and ensure that it has been placed only on clean surfaces. Cut or tear the sample into pieces approximately 1 cm^2 in size. Split samples of heavy board.

Mix the pieces thoroughly. Store the pieces in clean, covered containers.

7 Procedure

Run the procedure in duplicate.

7.1 Weighing

Weigh 2,0 g ± 0,1g of air dry sample (6.2) in a 250 ml flask (5.1).

NOTE – Since the amount of sample is not critical, there is no need to determine the dry matter content to adjust for minor differences in moisture content.

7.2 Extraction

Add 100 ml of water (4.1) into the flask (5.1) containing the sample pieces. Check that all pieces are soaked. Seal the flask with its ground glass stopper and leave it to stand for 1 h at 20 °C to 25 °C. Shake the flask at least once during this time.

NOTE If the ionic strength in the diluted sample is low, ion-exchange phenomena will occur and the pH in the bulk suspension liquor and the pH in the water held by the fibre wall will not be the same.

Filter the extract through a coarse, fritted glass filter into a small beaker (5.1). Immediately add 2 ml of potassium chloride solution (4.3) and continue with the measurement.

7.3 Determination of pH

Operate the pH meter in accordance with the manufacturer's instruction. Wash the electrodes with water (4.1); allow the water to drain from the electrodes, but do not wipe them. Calibrate the pH meter (5.2), at a temperature between 20 °C and 25 °C, with two different buffer solutions (4.2) having pH values such that the pH of the extract is between the pH values of the buffer solutions. The first buffer solution should be chosen so that the pH value of the buffer solution is in the same region as the electric zero point of the pH meter (usually = 7). The reading for the second buffer solution should agree with its correct value to within 0,1 pH unit.

NOTE – If the pH meter fails to show the correct pH value for the second buffer solution, consult the manufacturer's manual. A deviation exceeding 0,2 pH units indicates a faulty electrode. Also a slow but continuous increase or decrease in the reading indicates faulty electrodes.

After calibration, rinse the electrode several times with water (4.1) and once in a small quantity of the extract. Check that the temperature of the extract is between 20 °C and 25 °C. Immerse the electrodes in the extract. Record the pH when there is no measurable drift, within 30 seconds.

Before measuring the next sample, rinse the electrodes carefully with water (4.1) to remove any traces of sample or buffer solution.

At the end of a series of measurements, check the electrodes with buffer solutions.

8 Calculation

Calculate the mean of the duplicate determinations.

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