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

Part 2:
Hot extraction

Papier, carton et pâtes — Détermination du pH des extraits aqueux — Partie 2: Extraction à chaud

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This third edition cancels and replaces the second edition (ISO 6588-2:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

- addition of the last four paragraphs in the introduction to clarify the differences, in the role and mode of addition of a salt solution, between this document and ISO 29681[5].

A list of all parts in the ISO 6588 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

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[2][3].

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 the 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.

Based on the foregoing, two ISO Standards, this document and ISO 29681, are available for determination of the pH of hot aqueous extracts of paper, board, or pulps. In ISO 29681, a salt solution is added prior to extraction; however, in this document, the extraction is carried out with distilled or deionized water. ISO 29681 is specifically applicable to bleached pulps from virgin fibres and to pulp samples having a low ionic strength for which the pH-value will give more realistic results related to mill conditions than those obtained with this document.

It is necessary to be aware that the results will not be the same when measuring pH according to this document and to ISO 29681. The difference can be significant especially when measuring pulps having a low ionic strength.

ISO 6588-1 differs from this document, in relation to 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.

It should be noted that in this document, addition of a salt solution is performed after extraction and filtration only for the purpose of obtaining a more stable and accurate pH measurement.

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

Part 2:
Hot extraction

1 Scope

This document specifies a method for the determination of the pH-value defined by the electrolytes extractable by hot water from a sample of paper, board or pulp.

This document 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 document 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. In such cases, ISO 29681 can be used instead, as it is specifically applicable to bleached pulps from virgin fibres and to pulp samples having a low ionic strength for which the pH value will give more realistic results related to mill conditions than those obtained with this document. For cellulosic papers used for electrical purposes, the method used can be that given in IEC 60554-2[4].

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 7213, *Pulps — Sampling for testing*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

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

5 Reagents

5.1 Water, distilled or deionised water shall be used throughout the test. The conductivity of the water shall not exceed 0,1 mS/m after boiling for 1 h and cooling in an acid-free atmosphere (e.g. free of CO_2, SO_2, H_2S) to a temperature between 20 °C and 25 °C. The pH of the water should be in the range of 6,8 to 7,3. Instructions for the determination of conductivity are specified in ISO 3696.

5.2 Standard buffer solutions, with known pH-values of about 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.

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

6 Apparatus and equipment

Use the following, in addition to ordinary laboratory apparatus and equipment.

6.1 Glassware of chemically resistant glass, flasks with ground-glass joints, stoppers, beakers, reflux condenser and fritted glass filter. All glassware shall be cleaned with an acid cleaning solution, without the use of soap or detergent, and they shall be carefully rinsed with water (5.1) and allowed to dry before use.

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

7 Sampling and preparation of sample

7.1 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, and ensure that the specimen taken in 7.1 is representative of the sample received.

Wear clean protective gloves when handling the sample.

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

7.2 Preparation of sample

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

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

8 Procedure

Run the procedure in duplicate.

8.1 Weighing

Weigh 2,0 g ± 0,1 g of air-dry sample.

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.

8.2 Extraction

Add 100 ml of water (5.1) to a 250 ml flask (6.1) and heat to boiling. Add the weighed sample, attach the reflux condenser (6.1) fitting the flask, and continue boiling gently for 1 h ± 5 min on an electric heater.

Cool the flask rapidly to a temperature between 20 °C and 25 °C with the condenser in place. Filter the extract through a coarse, fritted glass filter into a small beaker (6.1). Immediately add 2 ml of potassium chloride solution (5.3) and continue with the measurement.

8.3 Determination of pH

Operate the pH-meter in accordance with the manufacturer's instruction. Wash the electrodes with water (5.1); allow the water to drain from the electrodes, but do not wipe them. Calibrate the pH-meter (6.2), at a temperature between 20 °C and 25 °C, with two different buffer solutions (5.2) having pH-values such that the pH of the extract is between the pH-values of the buffer solutions. The first buffer solution shall 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.

If the pH-meter fails to show the correct pH-value for the second buffer solution, consult the manufacturer's manual. A deviation of, or exceeding, 0,2 pH-units, rounded to the nearest 0,1 pH unit, indicates a faulty electrode. In addition, a slow but continuous increase or decrease in the reading indicates a faulty electrode.

After calibration, rinse the electrodes several times with water (5.1) 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 s.

Before measuring the next sample, rinse the electrodes carefully with water (5.1) to remove any traces of sample or buffer solution.

At the end of a series of measurements, check the electrodes with the same buffer solutions. The results for both solutions should agree with their correct value to within 0,1 pH unit. Otherwise repeat the procedure in this subclause (8.3).

9 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.

NOTE See Annex B for precision.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 6588-2;

b) the date and place of testing;

c) all the information necessary for complete identification of the sample;

d) the result, expressed as indicated in Clause 9;

e) any unusual features observed in the course of the test;

f) any departure from the procedure described in this document, or any other circumstances which may have affected the result.

Annex A
(informative)

Preparation of some standard buffer solutions

All the reagents used shall be of recognized reagent grade. The buffer solutions shall be renewed at least once a month. The anhydrous salts in A.1 and A.2 shall be dried at 120 °C.

A.1 Buffer solution pH 4,0: Potassium hydrogen phthalate, 0,05 mol/l solution

Dissolve 10,21 g of potassium hydrogen phthalate ($KHC_8H_4O_4$) in water (5.1) in a 1 l volumetric flask and dilute to the mark.

The pH-value of this solution is 4,00 at 20 °C and 4,01 at 25 °C.

A.2 Buffer solution pH 6,9: Potassium dihydrogen phosphate and disodium hydrogen phosphate solution

Dissolve 3,39 g of potassium dihydrogen phosphate (KH_2PO_4) and 3,54 g disodium hydrogen phosphate (Na_2HPO_4) in water (5.1) in a 1 l volumetric flask and dilute to the mark.

The pH-value of this solution is 6,87 at 20 °C and 6,86 at 25 °C.

A.3 Buffer solution pH 9,2: Disodium tetraborate solution

Dissolve 3,80 g of disodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) in water (5.1) in a 1 l volumetric flask and dilute to the mark.

The pH-value of this solution is 9,23 at 20 °C and 9,18 at 25 °C.