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Pulps — Preparation of laboratory sheets for the measurement of optical properties

Pâtes — Préparation des feuilles de laboratoire pour le mesurage des propriétés optiques

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 3688:1999), which has been technically revised.

The main changes are as follows:

— D65 brightness, whiteness and colour measurements have been included in the scope;

— recycled pulps have been removed from the scope;

— subclause 7.2 on the preparation of the sheets has been revised;

— pH adjustment has been changed from acid to neutral conditions;

— the number of sheets to be prepared has been updated in order to be consistent with subsequent measurements of optical properties.

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

The reflectance factors of laboratory sheets, and consequently all optical properties derived from diffuse reflectance factor measurements depend on the manner of preparation of those sheets and also on the conditions of measurement, particularly the spectral and geometric characteristics of the instrument used.

Pulps — Preparation of laboratory sheets for the measurement of optical properties

1 Scope

This document specifies two procedures for the preparation of laboratory sheets prior to measuring optical properties. One is the preparation of pads in a Büchner funnel using a filter paper or a wire screen and the other one is the preparation of laboratory sheets in a standard sheet former (conventional or Rapid Köthen).

This document is applicable to all wood pulps and to most other types of pulp.

It is not applicable to pulps with very long fibres, such as those made from unshortened cotton, flax and similar materials, unless they are reduced to a suitable fibre length (about 2 mm) before performing the methods.

It is not applicable to recycled pulps (see ISO 21993).

It is not applicable to opacity measurements or to the determination of light scattering and absorption coefficients.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3689, *Paper and board — Determination of bursting strength after immersion in water*

ISO 5263-1, *Pulps — Laboratory wet disintegration — Part 1: Disintegration of chemical pulps*

ISO 5263-2, *Pulps — Laboratory wet disintegration — Part 2: Disintegration of mechanical pulps at 20 degrees C*

ISO 5269-1, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method*

ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method*

ISO 7213, *Pulps — Sampling for testing*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Reagents

During the sheet preparation, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from colouring matter and from iron and copper ions.

NOTE Distilled water is used to ensure that the brightness of the sheets is not affected by the water.

4.1 Sodium hydroxide, (NaOH), approximately 0,1 mol/l solution, containing 4,0 g of sodium hydroxide per litre.

4.2 Acid, sulfuric acid, (H_2SO_4), approximately 0,05 mol/l solution, containing 2,8 ml of sulfuric acid (1,84 g/ml) per litre, or acetic acid, (CH_3COOH) at a concentration of 10 % containing 95 ml of acetic acid (1,05 g/ml) per litre.

4.3 Retention aid, if necessary, retention aid may be used for non-wood pulps. The kind and amount depend on the pulp to be tested.

NOTE For example, an addition of 0,4 % of polyacrylamide to the pulp sample has been proved effective.

5 Apparatus and auxiliary materials

All equipment with which the pulp comes into contact shall be of non-corrodible material, for example glass, porcelain, plastics and chromium-plated or stainless steel. Iron, copper, brass and bronze particularly shall not be used, since iron and copper ions have a strong tendency to cause colour reversion in pulp.

Ordinary laboratory apparatus and the following equipment shall be used.

5.1 Standard disintegrator, as specified in ISO 5263-1 or ISO 5263-2.

5.2 For sheet-making in a funnel.

5.2.1 Büchner funnel or similar, of non-corrodible material, the perforated bottom of which shall be flat, and have an internal diameter of 115 mm to 150 mm. The funnel shall be connected to a vacuum pump.

5.2.2 Filtering media.

Filter paper, with a diameter adapted to the funnel and free from fluorescent materials and soluble impurities, having a grammage of (84 ± 4) g/m^2, a filtration time for deionized water (20 ± 4) s, tested in accordance with Annex A, and wet bursting strength >30 kPa in accordance with ISO 3689.

NOTE For example, the filter paper Munktell grade 1289[1)] meets these requirements.

Alternatively, a wire screen, as specified in ISO 5269-1 or ISO 5269–2, may be used. The wire screen eliminates the difficulties in separating the sheet from a filter paper, a situation encountered when testing certain kinds of short-fibre pulp. However, there is a risk of losing some fine material when using a wire screen. All optical properties can change as a function of the quantity and distribution of fines in the sheet, and with the level of refining. For some kinds of mechanical pulp, the difference can be significant. Therefore, it is important that the apparatus and the filtering device used for the production of the sheets be reported.

1) Munktell grade1289 is the trade name of a product supplied by Ahlström. It can be obtained at Ahlstrom Germany GmbH, Bärenstein Plant, Niederschlag 1, 09471 Bärenstein, Germany. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

When sheets are formed on a filter paper, fine material can stick to the filter paper and lead to uneven brightness of the sheet. Experience has shown that the support of a thin wire made of plastic helps to avoid marks during dewatering. For this purpose, the use of a plastic wire with a mesh width of about 140 µm and a mesh diagonal of about 190 µm placed under the filter paper is recommended.

5.2.3 Vacuum device, that allows a pressure difference large enough to enable the drainage of water. A pressure difference ≥60 kPa is recommended.

5.2.4 Blotters, suitable for interleaving and for absorbing water pressed from the test sheets, without colorants and fluorescent whitening agents, having a grammage of 250 g/m^2 ± 25 g/m^2.

5.2.5 Pressing plates, made from chromium-plated metal, stainless steel or rigid plastic [for example of poly (methyl methacrylate)] of the same size as the laboratory sheets.

5.2.6 Hydraulic disk-press.

5.3 For sheet making in a sheet former.

5.3.1 Sheet former and auxiliary material, for example, as described in ISO 5269-1 or ISO 5269-2, depending on the sheet former. The material of the sheet former shall be such as not to influence the optical properties of pulp.

5.3.2 Device for drying of restrained laboratory sheets, either drying frames for clamping the sheet between two frames or a slightly convex plate fitted with a cloth for keeping the sheet in place. A number of such frames or plates may be mounted in a cabinet.

5.3.3 Press, capable of pressing the laboratory sheets at the pressure given in ISO 5269-1 or ISO 5269-2, depending on the sheet former.

5.4 pH-meter, calibrated and adjusted to give pH readings accurate to 0,1 pH unit.

6 Sampling

If the mean quality of a lot is to be determined, sampling shall be in accordance with ISO 7213. Otherwise, the method of sampling shall be reported and care shall be taken to ensure that the test pieces are representative of the sample available. Precautions shall be taken, during storage, to protect the sample from heat, light and change in moisture content.

There shall be a quantity of pulp sufficient for at least five test sheets having a grammage of approximately (225 ± 25) g/m^2.

7 Procedure

7.1 Pretreatment of pulp

7.1.1 Pulp in sheets or compressed slabs

Split the sheets or slabs and tear them into 20 mm to 30 mm pieces. In the case of dry pulp, soak for 0,5 h in water before disintegration so as to facilitate the separation of the fibres.

Weigh out the appropriate quantity of the pulp and disintegrate in water in the disintegrator (5.1). The minimum number of disintegrator revolutions to achieve dispersion of the pulp, free from visible fibre clots or bundles, shall be used for the test. The number of revolutions given in ISO 5263-1 or ISO 5263-2 shall not be exceeded.

Dilute the suspension to a stock concentration of 4 g/l.

7.1.2 Slush pulp

Determine the stock concentration and withdraw the appropriate volume.

7.2 Production of laboratory sheets

7.2.1 Addition of a retention aid

Non-wood pulps can contain small particles or fibres, with a colour deviating from the colour of the long fibres. They can affect the optical properties, but a wire screen will not retain them. If the optical properties of a pulp including these particles or fibres is to be measured, a retention aid shall be added prior to sheet forming.

The use, kind and amount of retention aid shall be stated in the test report.

7.2.2 pH adjustment

Using the pH-meter (5.4), check that the pH of the pulp suspension obtained from the pre-treatment is 7,0 ± 0,3. If not, adjust the pH so it falls within this range using the sodium hydroxide solution (4.1) or the sulfuric acid or acetic acid solution (4.2).

The optical properties of unbleached pulps can be affected by pH. For this reason, the measurement of optical properties is usually made on sheets produced from suspensions having a near neutral pH 7,0 ± 0,3. However, measurement of optical properties at a lower pH is sometimes requested, for example when the pulp is used in acid conditions. In this case, the pulp shall be tested at pH 5,0 ± 0,5, provided that this is stated in the report.

7.2.3 Funnel procedure

Stir the suspension well and divide it into portions, each portion containing a suitable amount of pulp to produce a sheet of (225 ± 25) g/m^2. Place a filter paper (5.2.2) in the funnel (5.2.1) and wet it with water. Alternatively, use a wire screen. Place the funnel so that its bottom is horizontal and pour one portion of the pulp suspension into the funnel. Let the water drain under suction. Avoid drawing any appreciable amount of air through the laboratory sheet that is formed. Remove the sheet by turning the funnel upside down, blowing into the stem and catching the sheet on a filter paper (5.2.2). Gently remove the uppermost filter paper and return it to protect the sheet. Mark the filter paper that is in contact with the side of the sheet that was uppermost in the funnel as the top side. Mark the sheet itself following pressing.

Proceed in the same way with the other portions of the pulp suspension. The number of laboratory sheets needed depends on the number of measurements required by the test procedure and opacity of the sheet. It shall be such that the diffuse reflectance factors of the pack of sheets will not be changed by increasing its thickness, but the number shall also be such that at least 10 valid measurements can be performed. Production of five sheets is sufficient for most types of pulp.

7.2.4 Pressing of the laboratory sheets

Arrange the pressing plates (5.2.6), blotters (5.2.4) and sheets for pressing in the following sequence, commencing from the bottom:

a) one pressing plate;

b) two dry blotters;

c) the laboratory sheet covered by the filter papers;

d) two dry blotters;