INTERNATIONAL STANDARD

ISO 3688

Second edition
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Pulps — Preparation of laboratory sheets for the measurement of diffuse blue reflectance factor (ISO brightness)

Pâtes — Préparation des feuilles de laboratoire pour le mesurage du facteur de réflectance dans le bleu (degré de blancheur ISO)

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.

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.

International Standard ISO 3688 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps,* Subcommittee SC 5, *Test Methods and quality specifications for pulp.*

This second edition cancels and replaces the first edition (ISO 3688:1977), of which it constitutes a technical revision.

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Introduction

The reflectance factor depends on the manner of preparation of the laboratory 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 diffuse blue reflectance factor (ISO brightness)

1 Scope

This International Standard specifies two procedures for the preparation of laboratory sheets prior to measuring the diffuse blue reflectance factor (ISO brightness). One is the traditional procedure based on preparation of sheets in a Büchner funnel using a filter paper or a wire screen. In the other procedure, the sheets are prepared in a standard sheet former (conventional or Rapid Köthen).

Details of the subsequent measurement procedure are given in ISO 2470.

It is applicable to all wood pulps and to most other types of pulp. Pulps with very long fibres, such as those made from unshortened cotton, flax and similar materials, shall be reduced to a suitable fibre length before testing by this method.

This International Standard shall be used in conjunction with ISO 2469 and ISO 2470.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2469:1994, *Paper, board and pulps — Measurement of diffuse reflectance factor.*

ISO 2470:—[1], *Paper and board — Measurement of diffuse blue reflectance factor (ISO brightness).*

ISO 5263:1995, *Pulps — Laboratory wet disintegration.*

ISO 5269-1:1998, *Pulps — Preparation of laboratory sheets for physical testing — Part 1: Conventional sheet-former method.*

ISO 5269-2:1998, *Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method.*

3 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 sample is not affected by the water.

1) To be published. (Revision of ISO 2470:1977)

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

3.2 Sulfuric acid, (H_2SO_4), approximately 0,05 mol/l solution, containing 2,8 ml of sulfuric acid (ρ 1,84 g/ml) per litre, or **acetic acid**, w (CH_3COOH) = 10 % containing 95 ml of acetic acid (ρ 1,05 g/ml) per litre.

3.3 Retention aid to be used for recycled and non-wood pulps.

Recycled and non-wood pulps may contain very small particles or fibres, with a colour deviating from the colour of the long fibres. They may affect the brightness value, but they will not be retained by a wire screen. If the brightness value of a pulp including these particles or fibres is to be measured, a retention aid shall be added prior to sheet forming. For example, an addition of 0,4 % of polyacrylamide to the recycled pulp sample has proved effective. The use, kind and amount of retention aid shall be stated in the test report.

4 Apparatus and auxiliary materials

All equipment with which the pulp comes into contact shall be of non-corrosive 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.

4.1 Standard disintegrator, as specified in ISO 5263.

4.2 For sheet-making in a funnel

4.2.1 Büchner funnel or similar of non-corrodible material, the perforated bottom of which shall be flat, and having an internal diameter of 115 mm to 150 mm and a volume of 1 000 ml to 1 500 ml. The funnel is connected to a vacuum pump.

4.2.2 Filter paper, medium hard, fast filtering with a diameter of 110 mm to 150 mm to fit the funnel, and free from fluorescent materials and soluble impurities. Alternatively a **wire screen**, as specified in ISO 5269, can 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. For most kinds of pulp, the brightness value is unchanged regardless of the filtering device, but for some kinds of mechanical or recycled pulp, the difference may be significant. Therefore, it is important that the apparatus and the filtering device used for the production of the sheets be reported.

NOTE When sheets are formed on a filter paper, fine material may stick to the filter paper and may lead to uneven brightness of the sheet. In this case, the wire screen may be preferable.

4.2.3 Blotters, suitable for interleaving and for absorbing water pressed from the test sheets, grammage approximately 250 g/m^2, and free from fluorescent materials and soluble impurities.

4.2.4 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.

4.2.5 Hydraulic disk-press.

4.3 For sheet making in sheet former

4.3.1 Sheet former and auxiliary material, for example, as described in the relevant part of ISO 5269. The material of the sheet former shall be such as not to influence the brightness of pulp.

4.3.2 Device for restricted drying of the laboratory sheets, either by clamping them between drying frames or by keeping them in place on a slightly convex plate by means of a cloth. A number of such frames or plates may be mounted in a cabinet.

4.3.3 Press, capable of pressing the laboratory sheets at the pressure given in the relevant part of ISO 5269.

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

5 Sampling

The sample from which the test pieces are to be taken shall be representative, and precautions shall be taken, during storage, to protect it from heat, light and change in moisture content.

A quantity of pulp sufficient for at least four test sheets having a grammage of approximately 200 g/m^2 is required.

6 Procedure

6.1 Pretreatment of pulp

6.1.1 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 disintegration apparatus (4.1). 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 should not normally be exceeded. Dilute the suspension to a consistency of 4 g/l.

6.1.2 Slush pulp

Determine the pulp concentration and withdraw the appropriate volume.

6.2 Production of laboratory sheets

Using the pH-meter (4.4), check that the pH of the pulp suspension obtained from the pretreatment is between 4,7 and 5,5. If not, adjust the pH to this interval with the sodium hydroxide solution (3.1) or the sulfuric acid or acetic acid solution (3.2).

The brightness of unbleached pulps is affected by pH. For this reason, the measurement of ISO brightness shall usually be made on sheets made from suspensions having a pH between 4,7 and 5,5. However, measurement of brightness at higher pH is sometimes requested, for example when the pulp contains materials which may dissolve at pH 5. In this case, the pulp shall be tested at pH 6,5 ± 0,5, provided that this is stated in the report.

NOTE De-inked pulps have fine particles which may be agglomerated by pH adjustment, thereby affecting brightness.

6.2.1 Funnel procedure

Stir the suspension well and divide it into portions, each portion containing a suitable amount of pulp to give a sheet of approximately 200 g/m^2. Place a filter paper (4.2.2) in the funnel (4.2.1) 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 and by blowing into the stem and catching the sheet on a filter paper (4.2.2). Gently remove the uppermost filter paper and return it to protect the sheet. Mark the side of the sheet that was uppermost in the funnel as the top side.

Proceed in the same way with at least three more portions of the pulp suspension. The number of laboratory sheets needed depends on the opacity of the sheets. It shall be such that the ISO brightness of the pack of sheets will not be changed by increasing its thickness. A pack of four sheets will be sufficient for most types of pulp.

Arrange the pressing plates (4.2.4), blotters (4.2.3) 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;

e) one pressing plate;

f) two dry blotters;

g) the next laboratory sheet covered by filter papers, etc.

Press the pack thus formed in the hydraulic press (4.2.5) for 1 min, applying a pressure of approximately 300 kPa to the sheets (often different from the pressure gauge reading), having verified that the pack is centralized on the pressure platen before applying the pressure.

After pressing, loosen the filter papers from the laboratory sheets, but leave them to protect the sheets. Dry the sheets at room temperature to a moisture content of 5 % to 15 % by hanging them with the filter papers in a current of dust-free air. The drying time shall not exceed 24 h.

Press the dried laboratory sheets, protected by the filter papers, in the press (4.2.5) at a pressure of 300 kPa to 500 kPa for 30 s to make them as flat as possible.

6.2.2 Sheet former procedure

Follow the instructions given in the relevant parts of ISO 5269-1 or ISO 5269-2 but make sheets with a grammage of 200 g/m^2. Use distilled water or water of equivalent purity.

Dry the laboratory sheets in the drying device (4.3.2) in the air at room temperature to a moisture content of 5 % to 15 %. Leave the two nearest blotters in place as a protection, or replace them by new blotters. The drying time shall not exceed 24 h.

As regards the number of laboratory sheets to be prepared, follow the instructions given in 6.2.1.

7 Storage for subsequent brightness measurement

Protect the sheets from contamination and from exposure to light and heat. The ISO brightness of the sheets should be measured immediately, according to ISO 2470, and in no case later than 4 h after drying has been completed.

8 Test report

The test report accompanying the laboratory sheets shall include the following details:

a) precise identification of the sample;

b) a reference to this International Standard;

c) precise identification of the laboratory sheets;

d) the number of disintegrator revolutions in the case of pulp in sheets or slabs;

e) type of apparatus and filter (filter paper or wire screen) used for the production of the laboratory sheets;

f) date and time of sheet making;

g) if a retention aid has been used, the kind and amount of retention aid;

h) if pH has been adjusted to 6,5 ± 0,5, this should be stated;

i) any particular points observed in the course of the sheet making;

j) any departure from this International Standard and any circumstances that may have affected the sheets.