INTERNATIONAL STANDARD

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Pulps — Preparation of laboratory sheets for physical testing —

Part 1: Conventional sheet-former method

Pâtes — Préparation des feuilles de laboratoire pour essais physiques —

Partie 1: Méthode de la formette conventionnelle

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 5269-1 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*.

This third edition cancels and replaces the second edition (ISO 5269-1:1998), of which the Introduction, Clauses 1, 2, 5 and 7 and the Bibliography have been revised.

ISO 5269 consists of the following parts, under the general title *Pulps — Preparation of laboratory sheets for physical testing*:

— *Part 1: Conventional sheet-former method*

— *Part 2: Rapid-Köthen method*

Introduction

It has been agreed that the ultimate aim of standardization of the preparation of laboratory sheets should be to develop one method which is internationally acceptable and which, if possible, permits the use of different types of sheet-making apparatus.

For practical reasons, it has not proved possible to achieve this at present. Therefore, as an interim measure, in view of the widespread use of equipment described in this part of ISO 5269, it has been decided to provide agreed guidance on the use of different types of equipment in order to achieve consistency of results with each method.

To avoid creating too many levels of results, the method specified in this part of ISO 5269 should preferably be used with the Valley beater or PFI mill methods of laboratory beating according to ISO 5264-1 and ISO 5264-2, respectively. The method specified in ISO 5269-2[2] (Rapid-Köthen method) should preferably be used with the PFI mill method of laboratory beating according to ISO 5264-2.

Pulps — Preparation of laboratory sheets for physical testing —

Part 1:
Conventional sheet-former method

1 Scope

This part of ISO 5269 specifies a method, using a conventional sheet former, for the preparation of laboratory sheets of pulp for the purpose of carrying out subsequent physical tests on these sheets in order to assess the relevant properties of the pulp itself.

This part of ISO 5269 is applicable to most kinds of pulp. It is not suitable for some pulps with very long fibres, such as those made from unshortened cotton, flax and similar materials.

This method is not suitable for the preparation of laboratory sheets for the determination of diffuse blue reflectance factor (ISO brightness) in accordance with ISO 3688[1].

WARNING — When long-fibred pulp is used in the unshortened form, the sheet formation may not always be satisfactory.

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 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

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 °C*

ISO 5263-3, *Pulps — Laboratory wet disintegration — Part 3: Disintegration of mechanical pulps at* $\geqslant$ *85 °C*

ISO 5264-1, *Pulps — Laboratory beating — Part 1: Valley beater method*

ISO 5264-2, *Pulps — Laboratory beating — Part 2: PFI mill method*

ISO 5635, *Paper — Measurement of dimensional change after immersion in water*

ISO 8787, *Paper and board — Determination of capillary rise — Klemm method*

3 Principle

A circular, square or rectangular sheet is formed from a pulp suspension on a wire screen under suction. The sheet is subjected twice to a pressure of 410 kPa. The sheet is dried in conditioned air and in contact with a drying plate, to which it adheres so that it does not shrink.

4 Equipment

4.1 Sheet former, consisting of three main parts.

4.1.1 Upper section, comprising a stock container, with a mark located 350 mm ± 1 mm above a wire screen (see 4.1.3). It is furnished with a rubber gasket to prevent leakage. The cross-section of the container shall be circular, square or rectangular, and constant throughout the height. If the container is rectangular, the shorter side shall be not less than 120 mm and the ratio of the longer to the shorter side shall not exceed 2,5. If the container is circular, it shall be not less than 158 mm in diameter. It shall be of such height that water will not splash over the edge when the stirrer (4.2) is operating.

4.1.2 Lower section, comprising a drainage vessel, consisting of an upper and a lower part. The upper part shall have the same cross-section as the stock container (see 4.1.1), and its shape shall be such that the flow of liquid through the wire screen is uniform over the whole area. The lower part may be of smaller cross-section but shall be positioned symmetrically in relation to the upper part. The lower part shall be fitted with a valve, which is connected to a draining pipe with a water seal at its lower end. The vertical distance from the top of the wire screen to the overflow of the water seal shall be 800 mm ± 5 mm. The lower part and the drainage valve shall be large enough to permit water in the stock container between the level mark and the wire screen to empty within 4,0 s ± 0,2 s. The lower part of the drainage container shall be provided with a water inlet tube. The design shall incorporate a means of releasing vacuum after the sheet has been formed.

4.1.3 Frame, with a perfectly flat, plain-woven metallic wire screen, to be placed horizontally between the upper section (4.1.1) and the lower section (4.1.2). The screen shall be clean, undamaged and fitted without wrinkles and corrugations. It shall have a nominal size of aperture of 125 µm, according to ISO 3310-1. The preferred diameter of the wire shall be 90 µm with a permissible range between 77 µm and 104 µm. The wire screen is backed by a coarser wire screen, which, in turn, may be backed by a rigid framework.

4.2 Stirrer, made of any non-corroding, rigid material, consisting of a perforated plate and furnished with vanes to keep the plate parallel to the wire screen (see 4.1.3) and to minimize swirling during stirring. The total area of the holes (diameter 10 mm to 20 mm) shall be about 30 % of the area of the plate; the holes shall be evenly spaced. The dimensions of the plate shall be such that there is a clearance of 2 mm to 3 mm between the plate and the stock container (see 4.1.1). All edges shall be rounded and smoothed to avoid the accumulation of fibres. The stirrer shall also have a stop that maintains a distance of about 20 mm between the wire screen and the plate in its lowest position.

An air agitation system may be used, provided that it produces bubbles of sufficient size and that they do not cling to the fibres or cause pin-holes in the sheet.

NOTE The following is an example of an agitation system. It uses compressed air and has at least eight inlet holes, each with a diameter of 1,0 mm ± 0,2 mm and equally spaced (max. 70 mm) in the upper section (4.1.1). The distance between the inlet holes and the wire screen is 10 mm ± 2 mm when the sheet former is operating. The inlet holes are connected to each other by air channels, 8 mm in diameter, located parallel to the sides of the sheet former so that the depth of the inlet holes (wall thickness) is 5 mm ± 2 mm. The air pressure is regulated to 100 kPa above atmospheric pressure. The agitation time is 5,0 s ± 0,5 s.

4.3 Couching equipment, comprising either

a) **a couch weight** having a plane bottom of the same area as the wire screen (see 4.1.3) and having a mass corresponding to a pressure of between 1 kPa and 5 kPa on the surface of the laboratory sheet; or

b) **an automatic couching system,** comprising a diaphragm to which air pressure is applied; or

c) **a couch roll** (mass 13,0 kg, length 178 mm, diameter 102 mm) and a couch plate to protect the sheets.

Unless an automatic couching system is used, a couch plate shall be used to protect the sheet from distortion when the couch weight is placed on it. The total mass of the couch plate and couch weight shall be within the limits given above.

4.4 Blotters, made of fully bleached chemical pulp or rag pulp, having neutral pH, and free from sizing agents, chemical additives, visible contraries and fluorescent contaminant (see Note 1). The blotters shall have the same dimensions as the laboratory sheets or, if the laboratory sheets are circular, neither the length nor width of the blotters shall be less than the sheet diameter nor shall the area of the blotters exceed that of the sheet by more than 35 %. If the sheets are square or rectangular, no blotter dimension in the plane of the blotter shall be less than the corresponding sheet dimension nor shall the area of the blotters exceed that of the sheet by more than 35 %. The grammage of the blotters shall be 250 g/m^2 ± 25 g/m^2; the Klemm absorbency, measured in accordance with ISO 8787, shall be 70 mm ± 20 mm and the dimensional changes caused by soaking, measured in accordance with ISO 5635, shall not exceed 3 % in any direction. The water uptake of the blotters shall be 450 g/m^2 ± 50 g/m^2.

The water uptake is determined as follows. Weigh a conditioned test piece, 40 mm × 40 mm, and immerse it in deionized or distilled water at 23 °C for 2 s. After removal, drain the test piece by holding it vertically from one corner for 30 s and determine the difference in mass before and after immersion. Calculate the water uptake as the mass of water absorbed, in grams per square metre of conditioned blotter.

NOTE 1 For sheets made of highly beaten pulps, the wet strength of the blotters may be insufficient. In such cases, blotters containing wet-strength agents may be used, but only if it has been proved that these agents do not infiltrate the laboratory sheet. If the blotters contain wet-strength agents, this should be mentioned in the test report.

NOTE 2 Practical tests have shown that, in some cases, the blotters can have a wide absorbency variation across the sheet and that this can result in wrinkled sheets.

4.5 Drying plates, of the same size as the formed sheet, made of corrosion-resistant metal or another suitable material, such as rigid plastic, glazed or polished on at least one side. The surfaces of the drying plates should be such that the wet sheets adhere easily to them. The plates shall be flat and free from any perceptible bulges or distortions.

4.6 Template, to facilitate the stacking of laboratory sheets. This shall be designed to fit the shape of the laboratory sheets and to ensure that they are placed centrally on each other in the press (4.8).

4.7 Separating plates, of the same size as the blotters (4.4) or larger, made of corrosion-resistant material or plastic, to separate laboratory sheets of different kinds. The use of separating plates is optional.

4.8 Press, capable of exerting a uniform pressure of 410 kPa ± 10 kPa over the area of a laboratory sheet and of maintaining this pressure for 5 min. The maximum number of laboratory sheets to be pressed simultaneously shall be adjusted to the capacity of the press.

4.9 Means to keep the test sheets in close contact with the drying plates (4.5) during the entire drying so that the laboratory sheets do not shrink. (See also Note 1 in 6.3.)

4.10 Conditioning cabinet or **suitable room,** with adequate air circulation, capable of maintaining the same atmospheric conditions, specified in ISO 187, as those under which the sheets will be tested. During the period when the sheets are still wet, the relative humidity may be allowed to exceed the limit and the temperature may be allowed to fall a few degrees below the limit.

5 Preparation of sample

Unbeaten pulps shall be disintegrated in accordance with ISO 5263. If the sample is a chemical pulp, disintegrate in accordance with ISO 5263-1, if the sample is a mechanical pulp not exhibiting any latency, disintegrate in accordance with ISO 5263-2, and if the sample is a mechanical pulp that exhibits latency, disintegrate in accordance with ISO 5263-3. Laboratory beaten pulps shall be treated as specified in the relevant International Standard (ISO 5264-1 or ISO 5264-2). Slush pulps taken from mill streams do not require any pretreatment.

Obtain the stock and dilute it with water to a mass fraction of between 0,2 % and 0,5 %. Mix thoroughly and prepare a trial laboratory sheet (oven-dry grammage 50 g/m^2 to 70 g/m^2) of known area as specified in 6.1. From this trial laboratory sheet, either determine the amount of stock which will produce a laboratory sheet of the desired oven-dry grammage or adjust the mass fraction of the stock so that a sheet of the desired oven-dry grammage can be produced using a vessel of known fixed volume. The preparation of this trial laboratory sheet on the sheet former for the determination of the mass fraction of the stock eliminates the need for making a correction for the loss of fibres through the woven metallic wire screen. Use the stock for forming sheets with a minimum of delay.

For pulps that tend to produce flocs, dilute the stock to a mass fraction of 0,2 % to 0,3 %.

6 Procedure

6.1 Sheet forming

Close the drainage valve of the sheet former (4.1). Open the inlet valve to wash the wire. Clamp the upper section (4.1.1) in position. Let the water rise to at least 50 mm above the wire screen (see 4.1.3).

Add, to the sheet former, an amount of stock which contains an accurate mass of the pulp to be evaluated so as to produce a laboratory sheet of the required grammage. For sheets to be used for testing the general physical properties, this grammage is 60,0 g/m^2 ± 2,0 g/m^2, calculated on an oven-dry basis. If the sheets are to be used for tests requiring another grammage, make sheets of the required grammage to an accuracy of ± 3 %.

Dilute to the mark with tap water having a temperature of 20 °C ± 5 °C. To mix the suspension, carry out either of the following operations.

a) Insert the stirrer (4.2) and move it briskly up and down. The stirrer plate shall remain below the surface during the stirring. Perform the double movement six times, vigorously enough to ensure thorough mixing, then once more, slowly, before gently withdrawing the stirrer.

b) Activate the air agitator (see 4.2).

In either case, 10 s ± 1 s after agitation is completed, open the drainage valve fully with a rapid movement.

When the water has left the wire screen, let the sheet formed on the wire screen drain under reduced pressure for a period that is about 10 % of the draining time, but not less than 5 s.

6.2 Transfer of the sheet

Disconnect the upper section of the sheet former (4.1) and close the drainage valve (see 4.1.2). Place two blotters (4.4), wire side up, centrally over the wet sheet on the wire. To couch the sheet either

a) place the couch weight [4.3 a)] gently and centrally on the blotters, protected with a couch plate, and remove it again after 20 s; or

b) use the automatic couching system [4.3 b)] to apply a pressure not greater than 70 kPa on the blotters for about 5 s, but not more than 30 s; or

c) lay the couch plate (see 4.3) centrally on the blotters and place the couch roll [4.3 c)] gently in the middle of the couch plate. Move the roll backwards and forwards across the couch plate, applying no additional pressure, to within 6 mm of the edges of the couch plate. Make five complete rolls in about 20 s and lift up the couch roll from the middle of the plate.

NOTE Blotters that have been used in this procedure and are flat and in good condition may be re-used after drying, provided that they are not placed in contact with a laboratory sheet. Blotters that are used in contact with laboratory sheets should be new.

Carefully separate the laboratory sheet, still adhering to the lower blotter, from the wire. Avoid any unnecessary bending. Place the laboratory sheet, attached to the couch blotter (4.4), laboratory sheet up, on top of one dry blotter in the stacking template (4.6). Place the drying plate (4.5), with its polished side down, on top of the laboratory sheet, followed by another dry blotter ready to receive the next couch blotter and laboratory sheet. Ensure that the laboratory sheets are placed centrally on each other by means of the template (4.6). The laboratory sheets may be marked while wet with an indelible pencil.

It is essential to keep the drying plates completely clean and free from wax, oil or anything that prevents adhesion of the wet sheet to the polished surface.

Empty the drainage vessel and prepare the sheet former for the next sheet.

6.3 Pressing

The complete stack then consists of dry blotter, couch blotter and laboratory sheet with drying plate, repeated several times. Place a single dry blotter on top of the top drying plate.

NOTE 1 If the stack contains laboratory sheets from different types of pulp, these may be separated by inserting a separating plate (4.7).

It is also recommended to use a separating plate at the bottom of the stack, so that the blotters carrying the laboratory sheet are not in direct contact with the plate of the press.

Ensure that the stack (see 4.6) is centrally in the press (4.8) and continuously raise the effective pressure on the sheets to 410 kPa ± 10 kPa within 25 s ± 5 s from the moment at which the first increase in pressure is registered. Maintain this pressure for 5 min ± 15 s, then release it and remove the stack from the press.

NOTE 2 The specified pressure is that applied to the laboratory sheets and may differ from the reading on the pressure gauge.

After the first pressing, the laboratory sheets should be firmly attached to the drying plates and any sheets that are not shall be rejected. A second pressing is next carried out, for which the order of the laboratory sheets is reversed and all the blotters are replaced. To do this, place the top drying plate from the first pressing with laboratory sheet attached (laboratory sheet up) on a dry blotter (if the top side can be identified it should be placed in contact with the sheet) using the template (4.6).

The complete stack then consists of a dry blotter, drying plate with laboratory sheet, and a dry blotter, repeated several times. Press the laboratory sheets by raising the pressure rapidly to 410 kPa ± 10 kPa. Maintain the pressure for 2 min ± 15 s and then release it and remove the stack from the press.

NOTE 3 There is no need to fix the time period for attaining the specified pressure in the second pressing, since the risk of sheet rupture is negligible and the platen movement due to compression is very much less than in the first pressing.

6.4 Drying and conditioning

Separate the drying plates together with the attached laboratory sheets from the blotters and mount them in a suitable manner (see 4.9) in the conditioning cabinet or suitable room (4.10) so that the laboratory sheets remain in contact with their drying plates during the entire drying period to prevent shrinkage. With normal air circulation, the sheets will therefore be conditioned and ready for testing the day after preparation. In cabinets where the air circulation is rapid, the drying period can be reduced. The dry sheets should separate readily from the drying plates; if they have properly adhered to the drying plates, they should be uniformly glazed. Any sheets which have separated from the drying plate during drying, or are not fully and uniformly glazed on one side, or leave some fibres on the surface of the drying plate, shall be rejected.

It is essential that the laboratory sheets do not shrink during drying.

NOTE Shrinkage can be prevented, for example, by clamping the laboratory sheets between specially designed drying frames. Another method is to dry the laboratory sheets with their drying plates on a non-heated, slightly convex