

Mechanical and chemical pulps

# **Preparation of laboratory sheets for physical testing**

Closed water system

#### 0 Introduction

This SCAN-test Method specifies a procedure for the preparation of laboratory sheets, using a closed water system. The purpose is to carry out subsequent physical tests on these sheets in order to assess the relevant properties of the pulp itself.

### 1 Scope

This Method is applicable to most kinds of pulp. It applies especially for the preparation of laboratory sheets from mechanical pulps, because of the low retention of fines when ISO 5269-1 Pulps – Preparation of laboratory sheets for physical properties – Conventional sheet-former method is used (see 8.3). It is not suitable for some very long-fibred pulps, such as those from unshortened cotton, flax and similar materials.

The standard grammage of sheets used for normal testing of physical properties is 60 g/m<sup>2</sup> calculated on an oven-dry basis. For pulps intended for the manufacture of boards or heavy papers for packages and boxes, this grammage is, however, too low. For testing properties such as bending resistance, compression strength and related properties, laboratory sheets of a higher grammage, 140 g/m<sup>2</sup> calculated on an oven-dry basis, are prepared. See also 6.2, Note 2.

*Note* – The Method is not applicable for the preparation of laboratory sheets for measuring ISO brightness. These sheets shall be prepared according to SCAN-CM 11 (8.1). For the preparation of sheets for the determination of light-scattering and light-

absorption coefficients, opacity and Y-value, SCAN-CM 27 is recommended (8.4).

#### 2 References

- ISO 5263-1 Pulps Laboratory wet disintegration Part 1: Disintegration of chemical pulps (EN ISO 5263-1)
- ISO 5263-2 Pulps Laboratory wet disintegration Part 2: Disintegration of mechanical pulps at 20 °C (EN ISO 5263-2)
- ISO 5263-3 Pulps Laboratory wet disintegration Part 3: Disintegration of mechanical pulps at < 85 °C (EN ISO 5263-3)
- ISO 187 Paper, board and pulps Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples (EN 20187)
- ISO 8787 Paper and board Determination of capillary rise Klemm method

*Note* – SCAN-test has withdrawn a number of test methods and refers instead to the corresponding ISO and/or EN Standards.

### 3 Principle

In a conventional sheet former, equipped with a system to recirculate the water, a circular or rectangular sheet is formed from a pulp suspension on a wire screen under suction. The sheet is pressed twice at a pressure of 400 kPa. It is dried in conditioned air and in contact with a drying plate, to which it adheres so that it does not shrink. *Note* – For mechanical pulps, the physical properties of sheets made according to this method will differ from the properties of sheets made according to ISO 5269-1.

#### 4 Apparatus

4.1 *Sheet former*, consisting of four main parts:

4.1.1 Upper section. Stock container to be filled with pulp suspension to a mark located  $(350 \pm 1)$  mm above a wire screen. The container is furnished with a rubber gasket to prevent leakage. The cross-section of the container shall be rectangular or circular, and constant throughout the height. If the container is rectangular, the shorter side shall not be less than 120 mm and the ratio of the longer to the shorter side shall not exceed 2,5. If it is circular, the container shall be of such a height that water will not splash over the edge when the stirrer is operating.

4.1.2 Lower section. A drainage vessel consisting of an upper and a lower part. The upper part shall have the same cross-section as the stock container (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 it shall be placed symmetrically in relation to the upper part. The lower part shall be fitted with a valve 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  $(800 \pm 5)$  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  $(4,0\pm0,2)$  s. The lower part of the drainage vessel shall be provided with a water inlet tube.

4.1.3 *Frame*, with a perfectly flat, plane-woven wire screen, to be placed horizontally between the stock container and the drainage vessel. The wire screen shall be clean, undamaged and fitted without wrinkles or corrugations. The wire screen shall have a nominal aperture size of 125  $\mu$ m (8.2), and a preferred diameter of the wire of 90  $\mu$ m, with a permissible range between 77  $\mu$ m and 104  $\mu$ m. The wire screen shall be backed by another coarse wire screen, which, in turn, may be backed by a rigid framework.

If the couching method described in Annex B is used, the frame with the wire screen shall be mounted so that it is easily removable.

4.1.4 *Recirculating water system*, consisting of a reservoir placed under the drainage vessel to collect the recirculating water and a pumping system which allows the sheet former to be filled from below the wire and also from above the wire. It is also important to have some

kind of agitation system in the closed water container. All parts of the system that come into contact with the water shall be of a non-corrosive material (plastic or stainless steel).

4.2 Agitation system using compressed air. There shall be at least 8 inlet holes, each having a diameter of  $(1,0 \pm 0,2)$  mm, located at equal distances (max. 70 mm) in the upper section (4.1.1) of the sheet former, so that the distance between the inlet holes and the wire screen is  $(10 \pm 2)$  mm when the sheet former is operating. If the stock container is rectangular, the numbers and locations of the holes in the sides opposite to each other must be the same. 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) will be  $(5 \pm 2)$  mm.

The air pressure is regulated to 1,0 bar above atmospheric pressure. The agitation time is  $(5 \pm 0,5)$  s.

*Note 1* – Alternatively, *a stirrer* may be used, 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 and to minimise swirling during stirring. The total area of the holes (10 mm to 20 mm in diameter) shall be about 30 % of the area of the plate and there shall be a clearance of 2 mm to 3 mm to the stock container. 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.

4.3 *A couch weight* having a plane bottom of the same area as the wire screen (4.1.3) and having a mass corresponding to a pressure of between 1,0 kPa and 5,0 kPa on the surface of the laboratory sheet.

*Note* 2 - Alternatively, an automatic couching system, comprising a diaphragm to which air pressure not greater than 70 kPa is applied, can be used.

*Note* 3 - A new, alternative couching system is described in Annex B.

4.4 *Blotters*, made of fully bleached chemical pulp or of rag pulp, neutral pH, free from sizing agents, chemical additives, visible contraries and fluorescent contaminants.

The blotters shall preferably be of the same dimensions as the laboratory sheets. 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  $(250 \pm 25)$  g/m<sup>2</sup>, the Klemm absorbency, determined according to ISO 8787, shall be  $(70 \pm 20)$  mm, and the dimensional changes caused by soaking shall not exceed 3 % in any direction. Further, the water uptake of the blotter shall be  $(450 \pm 75)$  g/m<sup>2</sup>, see Annex A.

Note 4 – 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, provided that it has been proved that the wet-strength agent will not migrate to the laboratory sheet. If the blotters contain wet-strength agent, this should be mentioned in the report.

Note 5 – The dimensional stability of the blotters is not critical if the couching system given in Annex B is used.

4.5 *Drying plates*, of the same size as the formed sheet, made of corrosion-resistant metal or a suitable plastic material, glazed or polished on at least one side. The surfaces of the drying plates must be such that the surfaces promote adhesion of the wet sheets to the plates. The plate shall be flat and free from any perceptible bulges or distorsions.

4.6 A *template* to facilitate the stacking of laboratory sheets. This shall be designed to fit the shape of the laboratory sheets and ensure that they are placed centrally on each other before they are moved to the press.

4.7 *Press*, capable of exerting an even pressure of  $(400 \pm 10)$  kPa over the area of a laboratory sheet. The press shall be able to reach this pressure within 20 s to 30 s and to hold the pressure constant. The maximum number of laboratory sheets to be pressed simultaneously is determined by the capacity of the press.

4.8 *Means to prevent the laboratory sheets from shrinking* during the entire drying (see clause 6.3).

4.9 *Conditioning cabinet or room*, with adequate air circulation, capable of maintaining 23 °C and 50 % RH, as specified in ISO 187. 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, as far as this method is concerned.

#### 5 **Preparation of sample**

#### 5.1 Disintegration

5.1.1 *Unbeaten pulp*. Disintegrate the pulp in accordance with ISO 5263, Part 1, Part 2 or Part 3, which is relevant.

5.1.2 *Laboratory-beaten pulp*. Prepare the pulp as described in the relevant ISO Standard for laboratory beating.

5.1.3 *Slush pulp* taken from mill streams. No disintegration is necessary if the pulp concentration is less than the concentration stated in ISO 5263-1 or in ISO 5263-2; otherwise disintegrate as described in these methods.

#### 5.2 Dilution

Dilute the stock with water to a concentration of between 0,2 % and 0,5 % (mass/mass). Use the stock for forming sheets with a minimum of delay.

*Note 1* – For pulps that tend to produce flocs, dilute the stock to a concentration between 0,2% and 0,3% (mass/mass).

*Note 2* – The quality of the water used does not have any influence on the physical properties of the laboratory sheets prepared.

### 6 Procedure

#### 6.1 Preparation of a closed water system

Fill the water reservoir with tap water. Before any sheets are taken for measurements, the water system must be in a state of equilibrium (the retention of fines). At least 8 sheets must be made (and rejected) in order to produce a closed water system. When the water system is in equilibrium, prepare a trial laboratory sheet (oven-dry grammage between 50 g/m<sup>2</sup> and 70 g/m<sup>2</sup>) of known area as described in clause 6.2.

Note 1 - A way of checking that the closed water system is in a state of equilibrium is to measure the dewatering time. During the build-up of the closed water system, the dewatering time normally increases. The system is in a state of equilibrium when the time remains constant even when additional sheets are prepared.

From this trial handsheet, either determine the amount of stock which will produce a laboratory sheet of the desired oven-dry grammage, or adjust the stock concentration so that a sheet of the desired oven-dry grammage can be produced using a vessel of known fixed volume.

### 6.2 Sheet forming

Close the drainage valve of the sheet former. Open the inlet valve and fill the sheet former from below the wire screen until the water is over the wire, then change the valves and fill from above the wire. Let water rise to at least 100 mm above the wire screen.

Normally, add an amount of stock corresponding to the grammage of the ready-made sheet of  $(60,0 \pm 2,0)$  g/m<sup>2</sup>, calculated on an oven-dry basis. If the sheets are intended for evaluating properties such as bending resistance, compression strength and related properties, add an amount of stock corresponding to the grammage of the ready-made sheet of  $(140 \pm 4)$  g/m<sup>2</sup>, calculated on an oven-dry basis.

*Note* 2 – The grammage of the sheets on a conditioned basis are approx.  $65 \text{ g/m}^2$  or  $150 \text{ g/m}^2$  respectively.

Make up to the mark with white water of a temperature of  $(20 \pm 5)^{\circ}$ C so that the final forming consistency of the suspension for 60 g/m<sup>2</sup> sheets is  $(0,020 \pm 0,005)$  % and for 140 g/m<sup>2</sup> sheets  $(0,05 \pm 0,01)$  %. Mix the suspension by activating the air agitation system or, if the manual stirrer (see Note to 4.2) is used, insert the stirrer and move it briskly up and down. The stirrer plate shall remain below the surface during the stirring. Perform the double movement 6 times vigorously enough to ensure thorough mixing, then once more slowly, before gently withdrawing the stirrer.

After the agitation is completed, wait  $(10 \pm 1)$  s and then open the drain valve fully with a rapid movement.

When the water has left the wire screen, let the sheet formed on the wire screen drain for between 5 s and 10 s. Then, disconnect the stock container and close the valve.

Couch the wet sheet as follows:

Place at least two blotters (4.4) centrally over the wet sheet on the wire screen. The machine directions of the blotters shall be marked and parallel and the side having the lowest surface roughness shall be in contact with the laboratory sheet. Place the couch weight (4.3) gently and centrally on the blotters. Remove it again after about 20 s and carefully separate the laboratory sheet, still adhering to the lower blotter, from the wire avoiding any unnecessary bending.

*Note 3* – If an automatic couching system is used, apply a pressure not greater than 70 kPa on the blotters for about 5 s, but not more than 30 s.

Note 4 – Blotters that are in contact with laboratory sheets shall be new. Used blotters, that are flat and in good condition, may be re-used after drying provided they are not placed in contact with a laboratory sheet.

Place the laboratory sheet, attached to the couch blotter (laboratory sheet up) on top of one dry blotter in the stacking template (4.6). Place a 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. The laboratory sheets may be marked while wet with an indelible pencil, parallel to the machine directions of the blotters.

Note 5 – 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.

*Note* 6 – When cutting test pieces, the test pieces shall be cut perpendicular to the MD-direction of the couch blotter.

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

#### 6.3 Pressing

Press the stack of sheets twice. For the first pressing, the complete stack is built up in the following way:

top

drying plate, polished side down	This
laboratory sheet } attached	sequence is
couch blotter $\int attached$	repeated
dry blotter	several times.
bottom	

Place the stack of sheets centrally in the press (4.7) and raise the effective pressure on the sheets to  $(400 \pm 10)$  kPa continuously during 20 s to 30 s. Maintain this pressure for 5 min  $\pm$  15 s, then release it and remove the stack from the press.

*Note* 7 – 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 shall be firmly attached to the drying plates, and any sheets that are not firmly attached, shall be rejected. Before the second pressing, the order of the laboratory sheets shall be reversed and all the blotters shall be replaced. Proceed as follows: Place the top drying plate from the first pressing with the laboratory sheet attached (laboratory sheet up) on a dry blotter using the template (4.6). Place a new dry blotter, with the side having the lowest surface roughness in contact with the laboratory sheet, followed by the next drying plate with its laboratory sheet attached. Continue the second stacking until it contains all the laboratory sheets from the first stack. The complete stack is now built up in the following way:

top ▲ new dry blotter	the side with the lowest surface roughness in contact with the sheet	This sequence is repeated
laboratory sheet drying plate	} attached	several times.
dry blotter		

#### bottom

Place the stack in the press, raise the pressure rapidly to  $(400 \pm 10)$  kPa. Maintain the pressure for 2 min  $\pm$  15 s and then release it and remove the stack from the press.

Note 8 – 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 and the attached laboratory sheets from the wet blotters. If necessary, a dry blotter may be used to protect the sheets during the drying.

Either clamp the sheets attached to the drying plates in specially designed drying frames or on a non-heated, slightly convex metal plate where they are kept in place by means of a cloth. A number of such drying plates can be mounted in a conditioning cabinet.

Dry the sheets in the conditioning cabinet or room (4.9). It is essential that the laboratory sheets are restrained by adhesion to the drying plate so that they do not shrink during the entire drying period since shrinkage will influence the result achieved.

The drying period will vary depending on the equipment used, the kind of pulp, the number of sheets prepared etc. For each arrangement for drying and conditioning, the time required shall be checked.

With normal air circulation in a conditioning room, the sheets will 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 and, if they have adhered properly to the drying plates, they should be uniformly glazed.

*Warning* – Minimize bending of the laboratory sheets when removing them from the drying plates, since incautious removal may influence the physical properties (e.g. bending stiffness) of the sheets.

#### 7 Report

The report shall include reference to this SCAN-test Method and the following particulars:

- (a) date and place of sheet preparation;
- (b) all the indications necessary for complete identification of the sample;
- (c) statement of the disintegration and beating given to the sample in the laboratory;
- (d) any unusual features observed in the course of test;
- (e) any departure from the standard procedure and any other circumstances that may have affected the results.

### 8 Literature

8.1 SCAN-CM 11 Pulps – Preparation of laboratory sheets (optical properties) – Part 1: Sheets for measuring ISO brightness

8.2 ISO 3310-1 Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth

8.3 ISO 5269-1 Pulps – Preparation of laboratory sheets for physical testing – Part 1: Conventional sheet-former method

8.4 SCAN-CM 27 Pulp – Preparation of laboratory sheets for determination of light-scattering and light-absorption coefficients, opacity and Y-value

## Annex A Determination of water uptake of blotters

The water uptake is determined as follows:

Weigh a conditioned (50 % RH, 23 °C) test piece of the blotter, 40 mm  $\times$  40 mm in size. Immerse the test piece in distilled or deionized water at 20 °C for 2 s. After removal from the water, drain the test piece by holding it in one corner for 30 s, reweigh it 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 the conditioned blotter.

## Annex B An alternative couching system

B.1 *Equipment*. If this equipment is used, its use must be reported since experience regarding any differences compared with results from conventional couching is limited.

In this alternative couching system, *Figure 1*, the whole frame has to be removed from the lower section of the sheet former for each sheet to be prepared. Thus, the couching procedure is considered to be more complicated to standardise.

This couching system may preferably be used for preparation of sheets from pulp grades having low wet tensile strength, such as mechanical pulps with a low CSF value (Canadian Standard Freeness) having a high content of fines.

B.2 *Procedure.* Place two pre-wetted blotters (4.4) on the curved plate covered with a felt. The machine directions shall be marked and parallel and the side having the lowest surface roughness shall come into contact with the laboratory sheet. Remove the wire screen with the wet sheet from the sheet former, place one of the edges of the wire screen down against the left-hand side of the blotters and press the laboratory sheet gently against the blotters with a rolling movement.

*Note* – The blotters can be pre-wetted by placing them under running tap water.



Figure 1. A couching system comprising a curved plate covered with a felt.

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