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Pulps — Determination of drainability —

Part 2: “Canadian Standard” freeness method

Pâtes — Détermination de l'égouttabilité —

Partie 2: Méthode de mesure de l'indice d'égouttage «Canadian Standard»

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 part of ISO 5267 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 5267-2 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 5267-2:1980) which has been technically revised.

ISO 5267 consists of the following parts, under the general title *Pulps — Determination of drainability*:

— *Part 1: Schopper-Riegler method*

— *Part 2: "Canadian Standard" freeness method*

Annexes A, B and C form a normative part of this part of ISO 5267. Annexes D and E are for information only.

Introduction

The “Canadian Standard” freeness test is designed to provide a measure of the rate at which a dilute suspension of pulp is dewatered under specified conditions. It has been shown that the drainability is related to the surface conditions and swelling of the fibres, and constitutes a useful index of the amount of mechanical treatment to which the pulp has been subjected.

The rate at which a suspension dewaters depends on the conditions of measurement, particularly the geometric characteristics of the instrument. The only practical means of achieving the required degree of accuracy for the measurement of “Canadian Standard” freeness is by the calibration procedure specified in annex C. The reproducibility of this method is entirely dependent on these arrangements being established within and between countries.

Results of this test do not necessarily correlate with the drainage behaviour of a pulp on a commercial paper machine.

A method for the determination of drainability in terms of the Schopper-Riegler number is specified in ISO 5267-1.

NOTE There are two slightly different types of “Canadian Standard” freeness testers in use, as described in annex A. These generally provide similar results, although some differences may occur.

Pulps — Determination of drainability —

Part 2:
“Canadian Standard” freeness method

1 Scope

This part of ISO 5267 specifies a method for determination of the drainability of a pulp suspension in water in terms of the “Canadian Standard” freeness in millilitres.

In principle, this method is applicable to all kinds of pulp in aqueous suspension.

NOTE Treatments which produce a large proportion of fines may induce an anomalous rise in freeness (false freeness), as a rule at values below 100 ml.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 5267. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 5267 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 4094, *Paper, board and pulps — International calibration of testing apparatus — Nomination and acceptance of standardizing and authorized laboratories*

ISO 4119, *Pulps — Determination of stock concentration*

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 14487, *Pulps — Standard water for physical testing*

3 Term and definition

For the purposes of this part of ISO 5267, the following term and definition applies.

3.1
“Canadian Standard” freeness
volume, expressed in millilitres, of the filtrate collected from the side orifice of the “Canadian Standard” freeness tester after correcting for the temperature and stock concentration of the sample under test

4 Principle

Drainage through a fibre mat formed during the test on a perforated screen plate of a given volume of an aqueous pulp suspension into a funnel provided with a bottom and a side orifice. Determination is made of the volume of filtrate discharged from the side orifice. The volume of the discharged filtrate, in millilitres, is the “Canadian Standard” freeness of the pulp.

5 Apparatus

Ordinary laboratory apparatus and:

5.1 “Canadian Standard” freeness tester, as described in annex A.

NOTE Instructions for maintenance of the apparatus are given in annex B. Details of the Calibration Service for the apparatus are given in annex C. Information concerning authorized laboratories is also given in annex C.

5.2 Measuring cylinders, calibrated in millilitres and capable of measuring volume with an error less than 1,0 ml for volumes up to 100 ml; an error less than 2,0 ml for volumes between 100 ml and 250 ml; and an error less than 5,0 ml for volumes above 250 ml.

5.3 Balance, capable of reading accurately to 0,01 g.

NOTE Although for the determination of the mass of the side orifice discharge a balance of 0,1 g accuracy is sufficient, for the determination of stock concentration the necessary accuracy of the balance is 0,01 g.

5.4 Standard water, for physical testing, as described in ISO 14487.

6 Preparation of sample

As the drainage of a pulp suspension is affected considerably by dissolved solids and the pH of the water used in the determination, standard water in accordance with ISO 14487 shall be used throughout the test.

A sample of an aqueous suspension of the disintegrated pulp shall be taken. If the concentration is not known exactly, the suspension shall be thickened or diluted to approximately 0,32 % by mass using standard water, and the stock concentration determined in accordance with ISO 4119. Then the suspension shall be diluted to a stock concentration of 0,30 % ± 0,01 % by mass and the temperature adjusted to 20,0 °C ± 0,5 °C (see note 3). Throughout the preparation of the sample, care shall be taken to avoid the formation of air bubbles in the suspension.

NOTE 1 With time, an aqueous pulp suspension withdrawn from the stock preparation system or laboratory pulp evaluation equipment can undergo a change in freeness. To avoid the effect of this reversion phenomenon, pulp suspensions subjected to testing more than 30 min after sampling shall first be treated in the disintegration apparatus for 6 000 revolutions of the propeller at 1,2 % to 1,5 % concentration.

NOTE 2 The test result is sensitive to the quantity of pulp fines, or “crill”, in the suspension. Thickened pulp samples may lose some of this fibre fraction. To avoid such losses during the course of thickening, the filtrate shall be recirculated through the pulp pad until the filtrate is clear and the pulp redispersed by disintegration, as described in note 1. This procedure shall be used to concentrate dilute pulp suspensions to the stock concentration required for the freeness test.

NOTE 3 Where necessary (e.g. process control), a temperature other than 20 °C may be used but it is not in accordance with this part of ISO 5267 and must be reported. The correlation tables presented in this part of ISO 5267 (informative annexes D and E) were developed from groundwood freeness evaluation studies. The accuracy of the correction tables for chemical pulp freeness evaluation has not been determined.

NOTE 4 In some applications, e.g. process control, it may be convenient to accept larger deviations than 0,01 % for stock concentration and ± 0,5 °C for temperature. The volume measured should be corrected with the aid of correction tables given in annexes D and E. The result so obtained does not conform to this part of ISO 5267.

7 Procedure

Clean the funnel and drainage chamber of the “Canadian Standard” freeness apparatus (5.1) thoroughly, and finally rinse with water. Place the drainage chamber in position. Adjust the temperature of the apparatus by rinsing with water at 20,0 °C ± 0,5 °C (see note 3 in clause 6).

Place the measuring cylinder (5.2) or a tared beaker (see note in 5.3) in position to receive the discharge from the side orifice.

Whilst stirring, transfer 1 000 ml ± 5 ml of homogeneous pulp suspension to a clean measuring cylinder.

Close the bottom of the chamber of the freeness tester and open the top lid and the air-cock. Mix the sample by closing the top of the cylinder with the hand and invert the cylinder through 180° three times, without losing the stock. As much as possible, avoid introducing air into the stock at this stage.

Pour the stock gently but as rapidly as possible into the chamber. At the end of the pouring, the stock should be almost motionless in the chamber. This can be achieved by pouring the stock around the inside of the chamber and finishing the pouring in the centre. Immediately close the top lid and the air-cock and open the bottom lid. Allow 5 s to elapse from the time of opening the bottom lid, then open the air-cock in a single motion to start the flow.

When the discharge from the side orifice has stopped, read the volume of this discharge to the nearest 1 ml for values below 100 ml, to the nearest 2 ml for values between 100 ml and 250 ml, and to the nearest 5 ml for values exceeding 250 ml. For greater accuracy, weigh the tared beaker and its contents to the nearest 0,1 g and convert the mass to volume (ml).

Combine, in a 2 000 ml beaker, the pulp from the chamber, side orifice discharge and bottom orifice discharge and drain the slurry in a sheet machine in accordance with ISO 5269-1 or ISO 5269-2 with a wire, or on a filter paper. For pulps with a high fines content, it is recommended to drain the pulp slurry on a tared filter paper in a Buchner funnel. Oven-dry the pad to a constant weight and record. Use this weight to calculate the stock concentration.

Carry out two determinations on each sample.

8 Expression of results

Report the mean of two determinations as the “Canadian Standard” freeness (ml). Duplicate determinations differing by more than 2 % from their mean value shall be repeated.

9 Test report

The test report shall include the following particulars:

a) reference to this part of ISO 5267, i.e. ISO 5267-2;

b) date and place of testing;

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

d) the test temperature if different from the standard;

e) the test concentration if different from the standard;

f) the mean result;

g) the type of funnel used in the test (modified or original design);

h) any unusual features observed during the test;

i) any operations not specified in this part of ISO 5267 or in the International Standard to which reference is made or regarded as optional, which might have affected the results.

Annex A
(normative)

The “Canadian Standard” freeness tester

A.1 The “Canadian Standard” freeness tester

This comprises a drainage chamber and a rate-measuring funnel, mounted on a suitable support (see Figure A.1). The rate-measuring funnel shown in Figure A.1, represents a modified design, adopted by the Canadian Pulp and Paper Association Technical Section (now known as the Pulp and Paper Technical Association of Canada) as a standard in 1964. The original design side orifice, with the angle cut that is still being used in some countries, fulfils the requirement of this standard. Work carried out at Paprican in 1993 comparing the two funnel designs shown in Figure A.2, with chemical pulp ranging in CS-freeness from 215 ml to 696 ml, showed no difference between the two funnels.

The dimensional and flow specifications given below are those required for the modified instrument to provide freeness results that are in complete agreement with those obtained from the original CS-freeness tester design. Test equipment made to the original design (without the centred side orifice or volume adjusting plug) may require adjustment to somewhat different values for calibration, and these values should be stated by the manufacturer. Where careful calibration procedures are followed, CS-freeness results — from either tester design — can be made to agree within the limits prescribed in clause A.5.

The calibration of the rate-measuring funnel requires that two critical dimensions be adjusted within specified limits: (1) the head of water in the funnel, which influences the flow through the bottom orifice; (2) the volume of water in the bottom section, from the bottom of the cone to the side orifice overflow level, given as 23,5 ml in the standard procedure. In this procedure the side orifice position is adjusted to provide the required volume, and early funnels in which the head above the bottom orifice falls within the specification are acceptable.

NOTE This caused occasional rejection of funnels which did not meet both requirements. It was also demonstrated that, with the side orifice tube cut at an angle to the tube axis, variation in the flow of water from the side orifice could result, when the funnel was rotated through 180° and the tester was not perfectly level. The design that was adopted in 1964 utilises a side orifice tube which is cut at 90° to the tube axis, and which is mounted so that the overflow from the funnel occurs at the centreline. This was intended to facilitate adjustment of the head over the bottom orifice, and permit rotation of the rate-measuring funnel without affecting the discharge rate. The bottom section of the cone is fitted with a threaded plug, which can be adjusted during calibration to provide the necessary volume of 23,5 ml, independent of the head adjustment. No further alteration should be made after the initial calibration. The modifications to the rate-measuring funnel were checked against the standard instrument maintained at the Pulp and Paper Research Institute of Canada. There was no evidence of any effect on instrument performance or level of test result. Rate-measuring funnels of this design can be expected to provide the same result as the original funnels. All testers incorporating these modifications had the letter “M” following the serial number. The angles of the funnels were not changed in 1964 and they conformed with the specifications given in A.4.

A.2 The chamber

This is a metal cylinder whose bottom is closed by a perforated screen plate and lid, hinged on one side of the cylinder and latched at the other. The lid shall be so fitted that not more than 5 ml of water will flow on opening of the bottom cover at the start of the test.

The upper end of this cylinder is closed by a similar lid, attached to the shelf bracket in which the cylinder is held when in use. The hinge and latching mechanisms are so designed that they provide an airtight closure by means of a rubber gasket on the inside of the lid. An air-cock is inserted in the centre of the upper lid for the admission of air into the cylinder at the start of the test.

The cylinder has an internal diameter of 101,6 mm ± 0,2 mm with an internal height of 127 mm ± 0,2 mm (from the upper surface of the screen plate to the rim). The diameter and height are critical dimensions. These dimensions provide a capacity which slightly exceeds 1 000 ml above the screen plate. The air-cock bore is 4,8 mm. This dimension is not critical but should not be subjected to substantial reduction.

A.3 The screen plate

This is cut into a circular shape, of diameter 112 mm to 112,5 mm and thickness 0,5 mm ± 0,05 mm and has perforations of diameter 0,5 mm spaced at 97 per cm^2. As it has not been possible to standardize the performance of these plates by reference to the dimensions or spacing of the perforations, all plates are calibrated according to documented in-house procedures at Paprican so that their performance matches that of master plates maintained by Paprican, or sub-master plates maintained by other centres (see annex C). The plates shall be mounted with the burr side of the perforations downwards.