INTERNATIONAL STANDARD

ISO 5267-2

Second edition
2001-09-15

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»

Reference number
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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

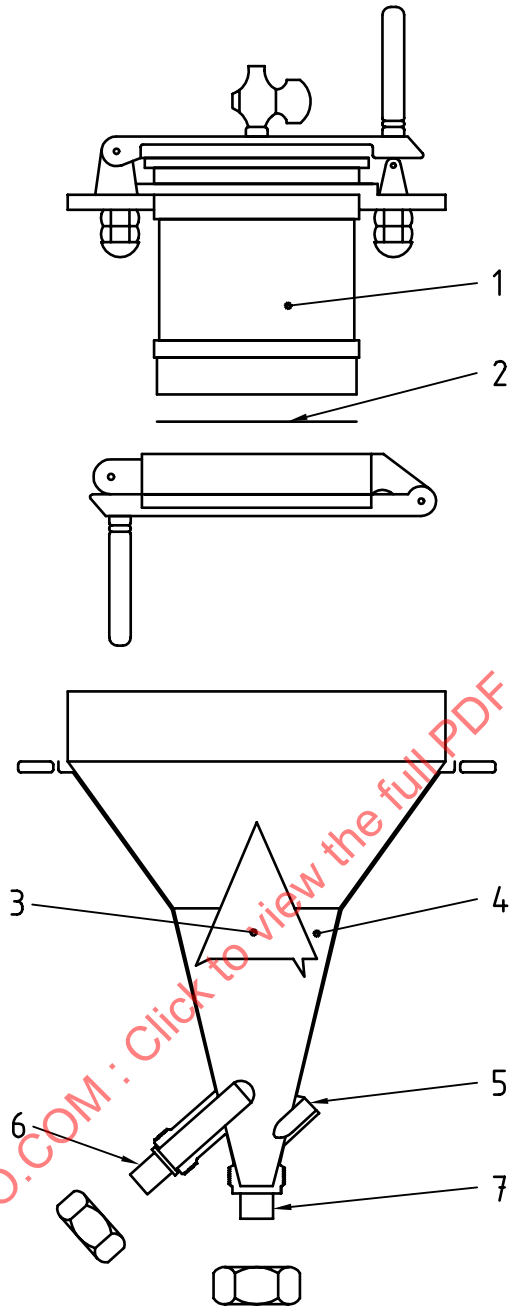
This is a metal cylinder whose bottom is closed by a perforated screen plate and lid, hinged on one side of the cylinder and latched at the other. The lid shall be so fitted that not more than 5 ml of water will flow on opening of the bottom cover at the start of the test.

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.

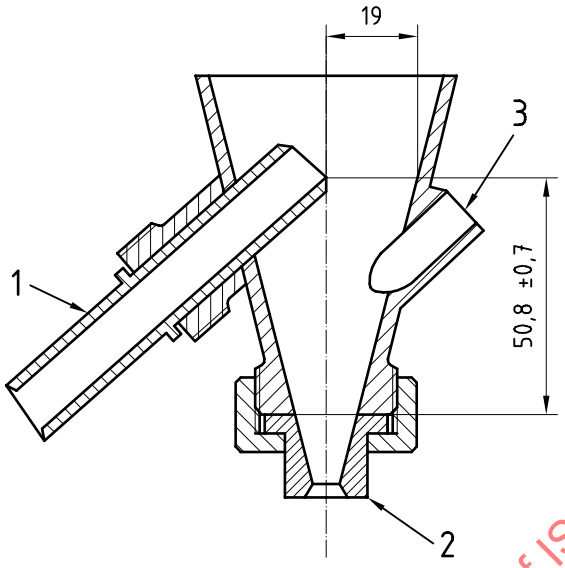


Key

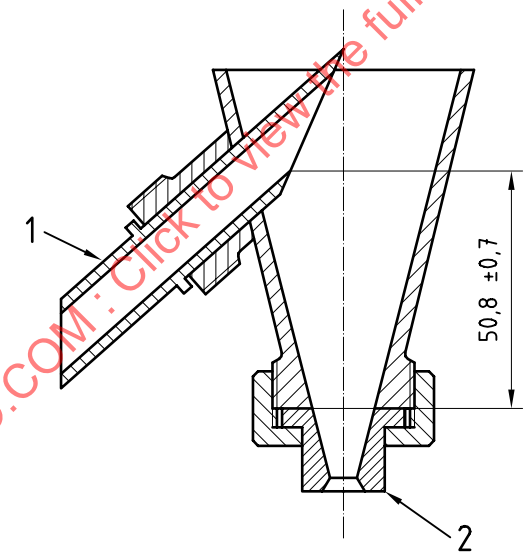
1 Chamber
2 Screen plate
3 Spreader cone
4 Funnel
5 Plug
6 Side orifice
7 Bottom orifice

Figure A.1 — “Canadian Standard” freeness tester

Dimensions in millimetres



Modified design



Original design

NOTE The volume for both designs is 23,5 ml ± 0,2 ml.

Key

1 Side orifice
2 Bottom orifice
3 Plug

Figure A.2 — Funnel styles

A.4 The rate-measuring funnel

This has an open top diameter of 203 mm and an overall length of 278 mm. The main cone has an angle of 29,5° ± 0,5° on the inside which flares out into a top cylindrical portion. The bottom (apex) terminates in a carefully machined bottom orifice piece attached to the funnel. The funnel is further provided with a side discharge orifice. A detachable spreader cone is supported in fixed location inside the funnel to prevent splash from directly entering the side orifice.

A.5 The side discharge orifice

This consists of a hollow tube, of internal diameter 12,7 mm, that penetrates the wall of the funnel. This tube is so inserted that the distance between the overflow lip of the tube (inside the funnel) and the bottom of the funnel section is 50,8 mm ± 0,7 mm.

This measurement is extremely critical and is set precisely by the manufacturer. Any adjustment of this dimension will exert a significant effect upon the performance of the tester. Once this distance is set by the calibrations laboratory, IT SHALL NOT BE CHANGED.

A.6 Adjustment of volume

The volume in the bottom section of the cone between the bottom of the funnel and the overflow lip of the side orifice, shall be adjusted to 23,5 ml ± 0,2 ml by means of an optional threaded plug. If this is not sufficient to adjust the volume then shims must be used under the collar of the side orifice. This volume is not critical within the stated limits of the specification, but should not be disturbed.

A.7 The bottom orifice

See Figure A.3.

The bottom orifice has an overall length of 19,6 mm. The diameter of the orifice venturi, nominally given as 3,1 mm in Figure A.3, is so adjusted during calibration that when it is fed with water at a rate of 725 ml/min ± 5 ml/min at 20 °C ± 0,5 °C, it will discharge 529 ml/min to 531,5 ml/min (during calibration). When the calibrated bottom orifice is assembled on the rate-measuring funnel for normal use, this discharge rate should lie within ± 1 % of 530 ml/min.

When the bottom orifice piece is attached to the bottom of the rate-measuring funnel, it shall be concentric, and the two sections shall fit accurately to provide an inside surface that is continuous and uninterrupted.

A.8 Protecting spreader cone

This is detachable and supported inside the funnel to prevent the direct entry of splash into the side orifice during the test.

A.9 Calibration

The screen plates, bottom orifice, side orifice and cone volume are calibrated and adjusted to meet the given specifications. When all of the components are assembled to constitute a complete tester, it may be expected to agree with a master instrument to within ± 2 ml. Any change in the critical adjustments of any of these components will affect the overall calibration of the assembled tester.

NOTE Because the modifications incorporated into the modified tester design required some dimensional changes in the rate-measuring funnel, certain funnel components, such as the side orifice, are not interchangeable with testers made to the original specification without the complete calibration.

Dimensions in millimetres

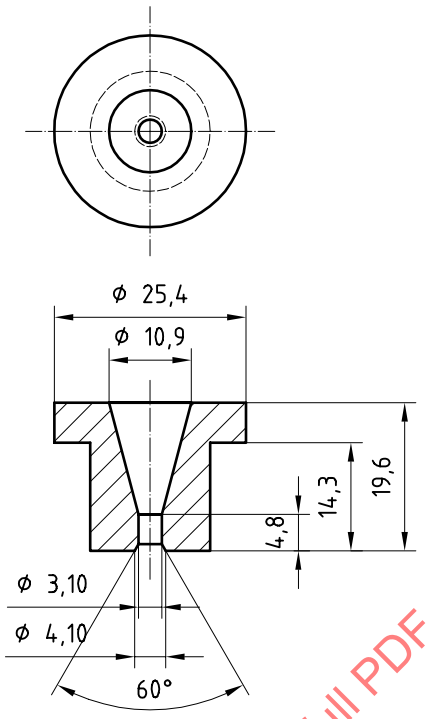


Figure A.3 — Bottom orifice

Annex B
(normative)

Maintenance of the “Canadian Standard” freeness tester

B.1 The instrument shall be mounted in a vibration-free environment, and carefully levelled using a machinist's level placed on the open top of the rate-measuring funnel, in position in the lower bracket. Checking the level of the funnel from side-to-side and back-to-front will indicate when the instrument is mounted in a true level position.

B.2 When the funnel is mounted in this way, the remaining components will be aligned correctly.

B.3 The instrument shall at all times be kept clean, free from pulp accumulations, pitch, oil or grease. After each test, the chamber shall be rinsed out with clean water. It is particularly necessary to ensure that no pulp is retained in the holes of the screen plate. Before any tests are started, the chamber and funnel shall be well wetted with clean water within $\pm$ 1 °C of the temperature of the stock to be tested.

B.4 If the instrument is to remain out of use, it shall be carefully and thoroughly washed so that no pulp will dry on it. The bottom lid shall be left opened. The top lid shall be partially closed, but unlatched. This will prevent dirt and other particles from entering. Before the instrument is re-used, it shall be well rinsed with clean water.

B.5 The practice of keeping an extra standard screen plate for a reference standard is strongly recommended. The screen plate in current use may then be checked at intervals. With careful use, a screen plate has a long life, but under normal mill conditions it may become dirty with an accumulation of resin which may be removed with an organic solvent or by gentle brushing in a mild detergent, free from carboxymethyl cellulose, phosphates or bleach, followed by thorough washing with hot water. Except where the materials of construction permit, the instrument and its components shall not be cleaned with acid. Bent or damaged screen plates shall be discarded.

B.6 When the screen plate is being replaced, care shall be taken when tightening the collar to avoid squeezing the chamber out of round. To perform this task a special holding device for the collar of the bottom cover assembly and a strap wrench with a 38 mm wide non-slip belting is satisfactory.

B.7 The instrument shall be cleaned with an organic solvent or detergent, followed by hot water. More drastic cleaning may destroy the calibration of the bottom orifice. If the flow exceeds that specified, the bottom orifice shall be replaced.

B.8 The certificate of inspection issued for each tester gives a value (using 1 000 ml distilled water at 20 °C instead of pulp) for the side orifice discharge which may be used as a field check (water test) on the bottom orifice. The test is described in the certificate. The side orifice discharge should not differ from the value in the certificate of inspection by more than 5 ml. If the difference exceeds this value, the bottom orifice shall be replaced.

Annex C
(normative)

Calibration Service

C.1 International calibration of “Canadian Standard” freeness testers is carried out in accordance with ISO 4094. The standardizing laboratory shall maintain a group of identical standard screen plates (hereafter referred to as ISO reference standards of level 1) for the ISO reference instrument.

C.2 The standardizing laboratory shall maintain a group of checking and working standard screen plates of level 2 which differ from the level 1 standards by less than ± 2 ml when compared with them under standard drainage conditions. The level 2 standard screen plates shall be used as comparisons in the calibration of screen plates of level 3 for sale and shall be subjected to comparison with the level 1 standard every 6 months.

C.3 Any agency that has, upon agreement with ISO/TC 6, been nominated as an authorized laboratory for the calibration of CS-freeness screen plates for sale, shall conform to the procedure described in a) to f) below.

a) The standardizing laboratory shall initially provide this authorized laboratory with five ISO reference standards of level 2. These shall be distinguished using some readily identifiable numbering system. At six-monthly intervals, one of these plates shall be returned to the standardizing laboratory in rotation for replacement by a new, numbered ISO reference standard of level 2. The standardizing laboratory shall maintain records to show the replacement schedule of each authorized laboratory.

b) The authorized laboratory shall use the ISO reference standards of level 2 obtained from the standardizing laboratory as one checking standard and four working standards to be used in the calibration of screen plates of level 3 for sale. ISO reference standards of level 2 shall not differ from the mean drainage value of the level 1 standard by more than ± 2 ml at a freeness level of 100 ml ± 25 ml with stone groundwood.

c) Screen plates offered for sale as Canadian Screen Plates shall fall within ± 2 ml of the mean drainage value of the ISO reference standard of level 2 when compared with a working standard in the standard CS-freeness apparatus.

d) Each authorized laboratory shall number each of its screen plates offered for sale with an easily identifiable number system and maintain records to indicate the exact date of calibration.

e) It is recommended that every tenth numbered plate at level 3 be set aside and shipped to the standardizing laboratory for comparison with the standardizing laboratory's standards. A report shall be made to the agency, and the plate returned.

f) The nominated authorized laboratories shall use, in their calibration procedures, only apparatus which agrees with the specifications of the “Canadian Standard” freeness tester, as described in this part of ISO 5267.

NOTE In case of disagreement at level 3, the standardizing laboratory may request the authorized laboratory to have its apparatus checked by the standardizing laboratory. This checking shall be charged to the standardizing laboratory.

A list of names and addresses of standardizing and authorized laboratories is maintained by the ISO/TC 6 secretariat, for the issue of ISO reference standards of level 3.