INTERNATIONAL STANDARD

ISO 5264-2

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Pulps — Laboratory beating —

Part 2: PFI mill method

Pâtes — Raffinage de laboratoire —

Partie 2: Méthode au moulin PFI

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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 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 5264-2 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 5264-2:2002), which has been technically revised.

With regard to ISO 5264-2:2002, the following changes have been made:

a) the normative references have been updated;

b) a more precise description of the PFI mill has been given in Annex A;

c) a new reference pulp for internal control beating has been added to Annex C;

d) editorial updating.

ISO 5264 consists of the following parts, under the general title *Pulps — Laboratory beating*:

— *Part 1: Valley beater method*

— *Part 2: PFI mill method*

Introduction

In view of the widespread use of the following beaters:

— Valley beater,

— PFI mill,

it has been decided to provide guidance on the use of these beaters in order to achieve consistency of results with each instrument. Although both beaters show similar trends in the effect on pulp properties, there is no correlation between the actual results obtained with the different types of beaters.

ISO 5264-1 specifies a method of laboratory beating using a Valley beater.

Beating is a preliminary step in the preparation of laboratory sheets for testing the physical properties of pulps. In the PFI mill, each beating is performed separately, i.e. a new test portion of unbeaten pulp is taken for each beating.

NOTE A complete test of physical properties normally comprises unbeaten pulp and several beatings of the same pulp, where the beating is carried out for different numbers of roll revolutions. The number of roll revolutions depends on the type of pulp and the beating load. After beating, the drainability is measured according to ISO 5267-1 or ISO 5267-2, and laboratory sheets are prepared according to ISO 5269-1[1], ISO 5269-2[2] or ISO 5269-3[3]. Physical testing of the laboratory sheets is performed according to ISO 5270[4].

Pulps — Laboratory beating —

Part 2:
PFI mill method

1 Scope

This part of ISO 5264 specifies a method for the laboratory beating of pulp using a PFI mill. The description is limited to the sampling, preparation and beating of the pulp and the beating equipment.

NOTE Beating is a preliminary step in testing the physical properties of pulp.

In principle, this method is applicable to all kinds of chemical and semi-chemical pulps. In practice, the method might not give satisfactory results with certain pulps having extremely long fibres.

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 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 4119, *Pulps — Determination of stock concentration*

ISO 5263-1, *Pulps — Laboratory wet disintegration — Part 1: Disintegration of chemical pulps*

ISO 5267-1, *Pulps — Determination of drainability — Part 1: Schopper-Riegler method*

ISO 5267-2, *Pulps — Determination of drainability — Part 2: "Canadian Standard" freeness method*

ISO 7213, *Pulps — Sampling for testing*

ISO 14487, *Pulps — Standard water for physical testing*

3 Principle

A measured amount of pulp at a specified stock concentration is beaten between a roll with bars and a smooth beater housing, both rotating in the same direction, but at different peripheral speeds.

4 Apparatus and auxiliary materials

Use ordinary laboratory equipment and the following.

4.1 PFI mill, as specified in Annex A.

See Annexes B and C.

4.2 Disintegrator, as specified in ISO 5263-1.

4.3 Balance, capable of weighing the sample with an accuracy of at least ±0,2 g.

4.4 Standard water, purified to have an electrical conductivity ⩽ 0,25 mS/m at 25 °C, as specified in ISO 14487.

4.5 A reference pulp, as described in Annex C, kept for beating-control purposes and stored for a sufficient time for the physical properties to stabilize. The reference pulp should, if possible, be of the same grade as that normally beaten in the beating equipment concerned. Since some pulp grades are not stable, it might be necessary to choose another grade.

To minimize a change in the pulp over time, the reference pulp should be kept at room temperature at a relative humidity range of 40 % to 60 % in a dark and dust-free place.

NOTE Stored under the recommended conditions, the reference pulp will, in most cases, be stable for approximately 10 years. Changes in the level of the tensile strength and tear strength values might be an indication that the reference pulp is no longer stable. The stability can be checked by measuring the viscosity of the reference pulp, e.g. twice a year.

5 Sampling

If the beating is being done to evaluate a lot of pulp, the sample shall be selected in accordance with ISO 7213.

If the beating is done on another type of sample, report the source of the sample and, if possible, the sampling procedure used.

Select the test portions so that they are representative of the gross sample received.

6 Preparation of sample

If the sample is wet or air-dry, weigh a test portion for the determination of the dry-matter content in accordance with ISO 638. If the sample is in the form of an aqueous pulp suspension, determine the dry-matter content in accordance with ISO 4119.

Take a test portion of the sample corresponding to (30,0 ± 0,5) g of oven-dry pulp. Do not cut pulp sheets and avoid the use of cut edges. If the sample is in the form of machine-dried sheets or flash-dried slabs, soak the pulp thoroughly in 0,5 litre of standard water (4.4) at room temperature for at least 4 h. Tear the soaked pulp into pieces approximately 25 mm × 25 mm in size. It is essential that the pulp sample be thoroughly softened by soaking, to ensure that the preliminary disintegration leads to only a minimal beating effect. Wet pulps may be disintegrated without soaking.

7 Procedure

For each test portion (each degree of beating), carry out the procedure described in 7.1 to 7.3.

7.1 Disintegration

Disintegrate the initially wet portion or the soaked test portion as described in ISO 5263-1. Use standard water (4.4) at (20 ± 5) °C to give a total volume of (2 000 ± 25) ml. The mass fraction in the disintegrator (4.2) will then be about 1,5 %.

Pulps having an initial mass fraction of dry matter of 20 % or more shall be disintegrated for 30 000 revolutions of the propeller, whereas pulps having an initial mass fraction of dry matter of < 20 % shall be disintegrated for 10 000 revolutions of the propeller.

After disintegration, check visually that the pulp is completely disintegrated. If not, continue the disintegration until complete separation of fibres is achieved.

NOTE A temperature outside the range (20 ± 5) °C can be used for climatic reasons, provided that this is stated in the test report.

7.2 Thickening

After disintegration, drain the pulp suspension on a Büchner funnel or other suitable device to a mass fraction between 11 % and 20 %. To avoid any loss of fibres, refilter the filtrate through the fibre mat, if necessary several times.

Using a balance, dilute the thickened pulp with standard water (4.4) to a total mass of (300 ± 5) g, corresponding to a mass fraction of 10 % stock.

7.3 Beating

7.3.1 Beating conditions

Check that the beating conditions are correct (see A.2).

The beating force per unit bar length shall be (3,33 ± 0,10) N/mm, assuming that only one bar at a time makes contact with the housing. It is important that the distance screw is disengaged during beating, i.e. no fixed gap shall be used.

Experience has shown that, for some pulps, a lower beating force per unit bar length may be needed to be able to evaluate the physical properties of the pulp in a correct way. In such cases, the beating force per unit bar length could be (1,77 ± 0,10) N/mm. This deviation from the standard procedure should be reported.

7.3.2 Beating procedure

Bring the beating elements of the PFI mill (4.1) and the thickened test portion of pulp, prepared in accordance with 7.1 and 7.2, to a temperature of (20 ± 5) °C (see Note to 7.1). Transfer the test portion of pulp to the beater housing, and distribute it as evenly as possible over the wall. A uniform band of pulp will ensure a smooth start-up, thus reducing unnecessary vibrations and attaining a more stable beating. Ensure that no pulp remains on the bottom of the beater housing within an area corresponding to the cross-section of the roll. Insert the roll in the beater housing, and press the cover correctly into position in the housing.

WARNING — When beating for a high number of revolutions, the temperature of the beating elements may increase. If necessary, cool the beating elements with water to bring the temperature within the specified range before the next beating. The temperature can be measured with an infrared (IR) thermometer or similar.

Set the beater housing in rotation so that the pulp is slung against the wall and start the roll motor. When both beating elements have attained full speed, apply the required beating force per unit bar length. Apply the beating load at a constant rate over a period of 2 s. At the instant of full application of the load, release the revolution-counter lever arm to engage the counter.

When the required number of roll revolutions has been reached, stop the beating by releasing the beating force. Switch off the motors, and allow the roll and beater housing to come to a complete stop. Lift the lid and bring the roll to its starting position.

Transfer the stock to a measuring cylinder or container with a capacity of at least 2 litres. Rinse the mill with standard water (4.4), and add the rinsings to the cylinder/container. Ensure that all material is included in the beaten test portion.

Dilute the stock with standard water (4.4) to (2 000 ± 25) ml, and disintegrate it for 10 000 propeller revolutions in the disintegrator (4.2). Proceed with the processing and/or testing of the beaten pulp in accordance with the

relevant International Standard. If drainability properties are to be measured, do this within 30 min of the end of the beating. Follow the instructions given in ISO 5267-1 or ISO 5267-2.

After each beating, clean the beating elements thoroughly with water and, if necessary, with a pitch solvent followed by water.

8 Test report

The test report shall include the following information:

a) a reference to this part of ISO 5264, i.e. ISO 5264-2:2011;

b) all information necessary for complete identification of the sample;

c) the date and place of beating;

d) the temperature of the water and the initial temperature of the housing, if either or both were outside the specified range;

e) the number of revolutions used for the preliminary disintegration;

f) the number of roll revolutions;

g) the beating force per unit bar length, if other than (3,33 $\pm$ 0,10) N/mm;

h) the material of the beating elements, if they are made of bronze;

i) the results obtained in the drainability test, if carried out;

j) any unusual features observed in the course of the procedure;

k) any operations not specified in this part of ISO 5264, or in the International Standards to which reference is made, or regarded as optional, which might have affected the results.

Annex A
(normative)

PFI mill

A.1 Description of PFI mill

A.1.1 General

The PFI mill (see Figure A.1) is composed of a roll, a beater housing with a cover and a loading device to provide the beating pressure (beating force per unit bar length). The beating elements are made of stainless steel. The roll and the housing rotate on vertical shafts. The cavity between the bars shall end in a radius of 5 mm and have a smooth surface.

NOTE PFI mills that were manufactured previously had beating elements made of bronze. Beating elements made of stainless steel and beating elements made of bronze will not necessarily give the same beating results.

If beating elements are made of bronze, this shall be stated in the test report.

A.1.2 Roll

The roll has 33 bars, each (50,0 ± 0,1) mm long and (5,0 ± 0,2) mm wide. The bars are arranged radially and are parallel to the roll axis. The diameter of the roll, measured across the bars, shall be between 199,5 mm and 202,0 mm (preferably 201,0 mm) and the depth of the cavities between the bars shall be between 28,0 mm and 31,0 mm (preferably 30,0 mm). The roll is driven by a motor with a rating of approximately 1 kW. The rotational frequency of the roll, when no load is applied, shall be $(24{,}3 \pm 0{,}5)\ s^{-1}$. The number of revolutions is indicated by a counter. The roll is driven in an anti-clockwise direction by means of a timing belt or a belt transmission.

A.1.3 Beater housing

The beater housing shall have an internal diameter of (250,0 ± 0,5) mm and an inner height of (52,1 ± 0,1) mm. The housing is driven by a motor with a rating of approximately 0,4 kW. At zero load and a rotational frequency of the roll of $(24{,}3 \pm 0{,}5)\ s^{-1}$, the difference in peripheral speed between the beating housing and the roll shall be (6,0 ± 0,2) m/s. The rotational frequency of the housing shall be about half of that of the roll and shall be set to comply with those requirements. At a nominal rotational frequency of the roll of $(24{,}3 \pm 0{,}5)\ s^{-1}$, the rotational frequency of the housing shall be $(11{,}8 \pm 0{,}3)\ s^{-1}$. The beater housing is driven in an anti-clockwise direction by means of a timing belt or a belt transmission.

A.1.4 Loading device

The loading device, to enable the beating force per unit bar length to be determined, shall work according to one of the following alternative principles:

— by means of a load applied by a lever that presses the roll against the wall of the beater housing;

— by means of pneumatic cylinders pressing the roll against the wall of the beater housing.

A.1.5 Device for regulating the gap

This device regulates the gap, i.e. the distance between the roll and the housing, including a distance screw, when grinding-in and conditioning the mill.

A.2 Operating conditions

A.2.1 To ensure reproducible beating, the following conditions shall be fulfilled.

A.2.2 The mill shall be set and levelled on a sturdy vibration-free base. Levelling can be accomplished, after the roll has been raised and swung to the side in the locked position, by placing a spirit level across the top of the bedplate and adjusting the levelling feet.

A.2.3 The roll and the housing shall run at the correct speeds.

A.2.4 There shall be no slippage of the belts. When the beating force is applied, the rotational frequency of the roll will normally decrease by between 0,3 s^{-1} and 0,6 s^{-1}, whereas the rotational frequency of the housing will increase slightly.

NOTE If timing belts are used, no slippage will occur.

A.2.5 The correct beating load is applied.

A.2.6 All parts shall move freely, so that the whole of the load applied is transmitted as beating pressure.

A.2.7 The distance screw shall be disengaged during beating.

A.2.8 The roll and the housing shall be clean and free from deposits. Pitch deposits shall be removed with a noncorrosive solvent.

A.2.9 The general mill conditions shall be checked from time to time by beating a reference pulp (4.5). Beat the reference pulp for the standard number of revolutions which has previously been shown to produce drainage values of about 50 Schopper-Riegler units (see ISO 5267-1) or 200 “Canadian Standard” freeness, in millilitres (see ISO 5267-2). The drainage value obtained shall be within ±5% of that established initially.

Dimensions in millimetres

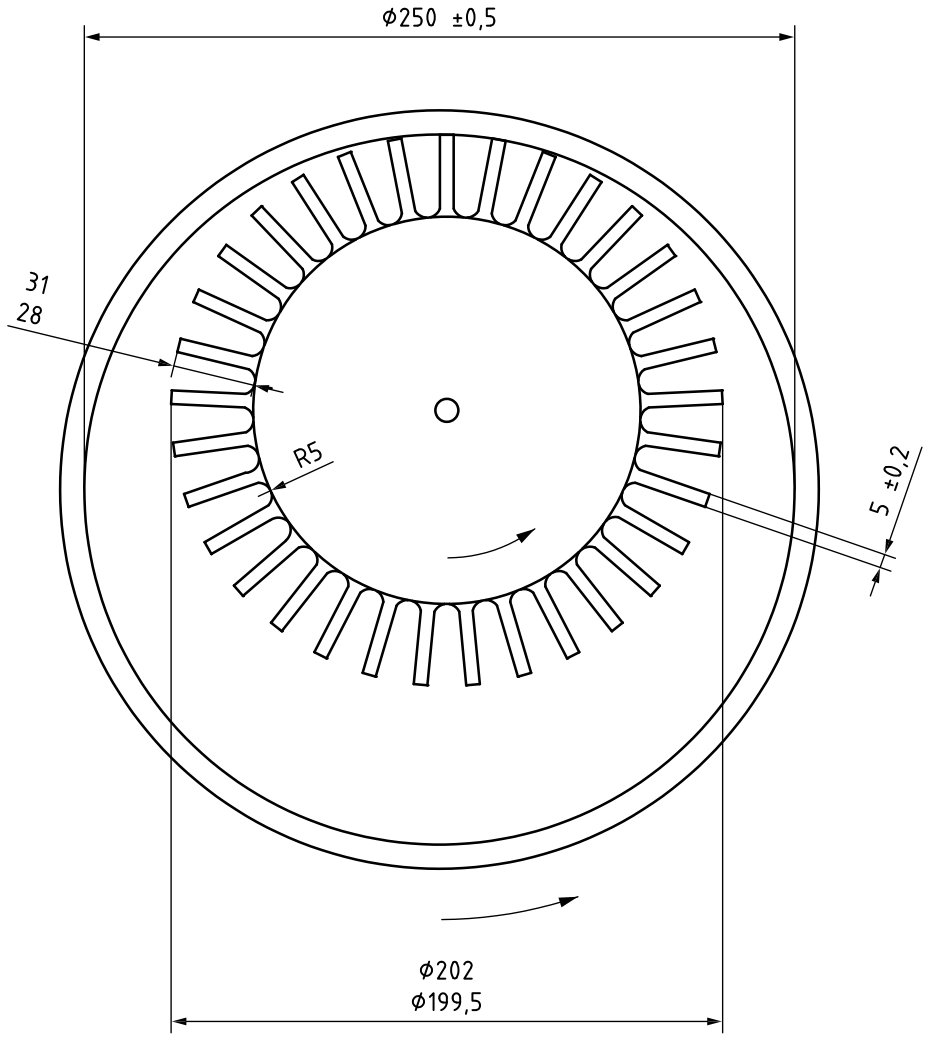

Figure A.1 — Roll and beater housing dimensions of the PFI mill

Annex B
(normative)

Control and maintenance of the PFI mill

B.1 General

To check the beating effect of the PFI mill (from time to time), keep a reference pulp (4.5) for beating purposes. How often the control beatings have to be carried out depends on the use of the mill and thus is a matter of experience. Under normal conditions, the beating effect should be maintained for a long period.

If the beating effect has decreased, i.e. the mill beats slower than normal, or if the beating surfaces have been damaged, restore the beating effect by using the procedures described in B.2 and B.3. Always start that procedure with the fine-grinding procedure described in B.3. If this is not enough, use the rough-grinding procedure described in B.2 followed by the fine-grinding procedure described in B.3.

If the beating effect has increased, i.e. the mill beats faster than normal, restore the beating effect by using the conditioning procedure described in B.4.

B.2 Rough-grinding procedure

Reverse the direction of rotation of the motor driving the beater housing. The housing shall now rotate clockwise. Determine the zero position of the adjusting screw by following the instructions given by the PFI mill producer.

Set the distance screw to obtain a gap of approximately 2,0 mm between the beating elements. Set the desired number of roll revolutions (5 000 to 6 000) and check the beating force per unit bar length (3,33 N/mm). Charge the beater housing with 15 g of silicon carbide powder (grade: passes through a 90 µm aperture) suspended in 50 ml of soluble oil diluted with 50 ml of water. Ensure that the silicon carbide powder, the cutting oil and the water are well mixed before the beater housing is set in motion.

WARNING — If the components are not well mixed, the silicon carbide powder has a tendency to remain near the bottom of the housing and will round off the bottom edges of the bars.

WARNING — To obtain a homogeneous mixture, temperature control is very important. See the recommendations given in the manual for the PFI mill. In an inhomogeneous mixture, there is a risk that globules of oil and water will form, and these will influence the grinding process.

Set the beater housing in rotation, so that the powder suspension is flung against the housing. Stop the housing. Ensure that the cover is in position in the bracket, insert the roll in the beater housing and press the cover into position.

Immediately start both beating elements, apply the load, and carefully reduce the gap between the elements by means of the distance screw until the sound of grinding can be heard. When the sound has diminished appreciably, further reduce the gap. The gap should not be less than 0,03 mm at any time. Continue this process of reducing the gap and then grinding until the damage has been rectified.

Finally, clean the beating elements and the cover with soap and water. Ensure that no oil or grinding powder remains.

B.3 Fine-grinding procedure

When the rough grinding is completed, carry out the fine-grinding procedure once or twice using silicon carbide powder (grade: passes through a 45 µm aperture). Perform the procedure as described in B.2, except for the grade of silicon carbide powder.

Using a fine-honing stone and then a polishing stone, remove any rough edges which may appear on the trailing edge of the bars. Clean the roll thoroughly.

Reverse the direction of rotation of the motor driving the beater housing. The beater housing shall now rotate anticlockwise.

B.4 Conditioning procedure

After grinding according to B.2 and/or B.3, the beating surfaces are usually too coarse. They have to be smoothened to the required beating effect (SR/CSF level) at a desired number of roll revolutions by beating a pulp mixed with silicon carbide powder (grade: passes through a 45 µm aperture) using the trial-and-error method.

Set the gap between the beating elements to approximately 2 mm and set the number of roll revolutions (which will vary depending on the degree of the coarse grinding). A suitable number may be 10 000 roll revolutions.

Charge the housing with a mixture of 30 g of pulp and 15 g of silicon carbide grinding powder (grade: passes through a 45 µm aperture). Beat this mixture using the normal procedure (see 7.3).

After the beating, clean the beating elements very thoroughly with soap and water. Take care that no remnants of powder and oil are left on the roll and the housing.

Disengage the distance screw.

Remove any remnants of powder by beating a pulp for a period corresponding to 10 000 roll revolutions.

B.5 Control beating

After grinding or conditioning, carry out a control beating with the reference pulp (4.5) to check the Schopper-Riegler or “Canadian Standard” freeness (SR/CSF) level.

If the SR value is too high (the CSF value is too low), repeat the conditioning procedure described in B.4. If, on the other hand, the SR value is too low (the CSF value is too high), repeat the grinding procedure described in B.2 and B.3.

Annex C
(informative)

Checking the stability of the PFI mill

C.1 General

It is recommended that periodic checks of the performance of the beater be made. Several procedures exist for verifying the performance of PFI mills. The following describes procedures that are commonly used. It is recommended that the laboratory choose the procedure(s) most appropriate for its application.

C.2 Reference pulp material for internal control beating

The laboratory may choose to check the repeatability of beating of the PFI mill on a regular basis (e.g. monthly) by running an internal control beating using a reference pulp of the same grade as the pulp samples that are most frequently used with the beater. Select a reference pulp that is stable with respect to its physical properties.

Reference pulps (hardwood and softwood) are available from the Norwegian Paper and Fibre Research Institute[1)] (PFI, having known calibration values and tolerances for physical properties after beating in the PFI mill).

Reference pulps (hardwood and softwood) are also available from FPInnovations[1)], having known calibration values and tolerances for physical properties after beating in the PFI mill. The beating conditions stated for FPInnovations reference pulp, however, are those of PAPTAC Standard C.7[5], which specify that the beating shall be performed with a pulp charge of 24 g and a beating gap of 0,2 mm and, therefore, are not in accordance with those stated in this part of ISO 5264.

C.3 Inter-laboratory comparison tests

In addition to internal control beating, the laboratory is recommended to check the reproducibility of beating of the PFI mill by taking part, on a regular basis, in inter-laboratory comparison tests where the performance of the beater is checked in comparison with other laboratories.

NOTE Both internal control beating and inter-laboratory testing can be performed using a hardwood pulp and a softwood pulp. Some laboratories even designate separate PFI mills for hardwood pulp and softwood pulp, since different pulp grades may have a different influence on the beating rate of the PFI mill.

1) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this supplier.

Bibliography

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

[2] ISO 5269-2, *Pulps — Preparation of laboratory sheets for physical testing — Part 2: Rapid-Köthen method*

[3] ISO 5269-3, *Pulps — Preparation of laboratory sheets for physical testing — Part 3: Conventional and Rapid-Köthen sheet formers using a closed water system*

[4] ISO 5270, *Pulps — Laboratory sheets — Determination of physical properties*

[5] PAPTAC Standard C.7, *Laboratory Processing of Pulp (PFI Mill)*

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