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Pulps — Determination of fibre coarseness by automated optical analysis — Polarized light method

Pâtes — Détermination de la grosseur de fibre par analyse optique automatisée — Méthode de la lumière polarisée

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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 23713 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 5, *Test methods and quality specifications for pulps*.

Pulps — Determination of fibre coarseness by automated optical analysis — Polarized light method

1 Scope

This International Standard specifies a method for determining fibre coarseness using polarized light.

The method is applicable to all kinds of pulp that polarize light. However fibrous particles shorter than 0,2 mm are not regarded as fibres for the purposes of this International Standard and therefore are not to be included in the results.

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, *Pulps — Determination of dry matter content*

ISO 4119, *Pulps — Determination of stock concentration*

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

ISO 5263-2, *Pulps — Laboratory wet disintegration — Part 2: Disintegration of mechanical pulps at 20 °C*

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

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

ISO 7213, *Pulps — Sampling for testing*

ISO 16065-1, *Pulps — Determination of fibre length by automated optical analysis — Part 1: Polarized light method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
unpolarized light
light composed of light waves whose planes of vibration are randomly oriented

3.2
polarizer
material which only transmits that component of a light wave which is vibrating in a particular direction, the direction of polarization of the material

3.3
plane polarized light
light composed of light waves which all vibrate in the same plane

3.4
crossed polarizers
pair of polarizers placed in a light path such that the direction of polarization of one is at right angles to the direction of polarization of the other, thus resulting ideally in none of the light which has passed directly from one polarizer to the other being transmitted through this second polarizer

3.5
birefringence
property of certain materials, such as cellulose fibres, which have a crystalline structure that results in the refractive index varying with the direction of polarization of the light

NOTE This has the effect of rotating the direction of polarization of a plain polarized beam of light resulting in light which has passed through this material being transmitted through the second polarizer of a crossed pair.

3.6
total fibre length
L_T
total length of all fibres in the test portion

See Equation (4)

3.7
fibre coarseness
oven-dry mass of fibres in the test portion divided by the total fibre length of the same test portion

See Equation (5).

3.8
fines
particles shorter than 0,2 mm

4 Principle

A known mass of fibres, suspended in water, is passed through a fibre orienting cell (FOC). The projected lengths of individual fibres are measured automatically. A crossed-polarizer set-up is used to discriminate between birefringent material like fibres with oriented cellulose molecules and non-birefringent material like air bubbles and filler particles, which do not rotate the plane of polarization. The total fibre length and the mean fibre coarseness of the pulp are calculated.

5 Apparatus and materials

Ordinary laboratory equipment and the following are required.

5.1 Fibre length analyzer, as described in ISO 16065-1, consisting of a measurement section and a sample transport system.

5.2 Disintegrator, as described in ISO 5263-1, ISO 5263-2 or ISO 5263-3.

5.3 Sheet former, as described in ISO 5269-1.

5.4 Balance, with $\pm$ 0,1 mg accuracy.

5.5 Balance, with a capacity exceeding 5 kg and with $\pm$ 0,1 g accuracy.

5.6 Vials, for storing test portions, volume 50 ml, with caps and labels.

5.7 A reference pulp[1)].

6 Sampling and preparation of specimen

6.1 Sampling

If the test is being made to evaluate a pulp lot, the sample shall be selected in accordance with ISO 7213. If the test is made on another type of sample, report the source of the sample and, if possible, the sampling procedure used. From the sample received, select specimens so that they are representative of the whole sample.

6.2 Disintegration

6.2.1 Sample in dried form

For samples in dried form, take at least 30 g oven-dry mass and soak for a minimum of 4 h in water. Tear the pieces, do not cut into pieces as this will cause fibre shortening. Disintegrate the pulp according to ISO 5263-1, ISO 5263-2 or ISO 5263-3, depending on the pulp.

Determine the concentration of the disintegrated pulp according to ISO 4119.

6.2.2 Sample in never-dried form

Disintegrate the pulp according to ISO 5263-1, ISO 5263-2 or ISO 5263-3, depending on the pulp.

Determine the concentration of the disintegrated pulp according to ISO 4119.

NOTE It is preferable to measure never-dried pulps with minimal disintegration, because excessive disintegration may generate fines and reduce fibre length in some pulps.

6.3 Removal of fines and preparation of test portion

6.3.1 Removal of fines

Place approximately 0,50 g oven-dry mass of the disintegrated pulp into a laboratory sheet former and form a wet sheet as described in ISO 5269-1.

NOTE 1 Forming a 0,50 g oven-dry laboratory sheet ensures that most fines are washed out.

Visually inspect the wet laboratory sheet for debris (i.e. shives, fibre bundles, contaminants). If debris is found (e.g. in recycled pulp, in mechanical pulps), then carefully remove 1 g (approx. 50 mg oven-dry) of wet pulp from parts of the wet sheet that do not contain debris. Any debris removal shall be mentioned in the test report.

NOTE 2 Debris introduces inaccuracy in the mass measurement, which in turn causes inaccuracy in the final coarseness result.

Place the 1 g debris-free wet pulp into a tared vial (5.6) and determine the mass of the wet pulp to an accuracy of $\pm$ 0,1 mg.

1) Reference pulp is available, for example, from the National Institute of Science & Technology (NIST), Gaithersburg, MD, USA, or the Pulp and Research Institute of Canada (Paprican), Pointe Claire, QC, Canada. The reference pulp is provided in sheet form. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

Determine the dry matter content of the remaining wet laboratory sheet, after removing the debris, as described in ISO 638. Make sure that the dry matter content over the wire area is uniform. Use this value to calculate the oven-dry mass of fibre in the vial. Record the result as the oven-dry fibre mass in the vial ($= m_1$).

If the debris-free wet pulps are to be stored, place them in a refrigerator at 4 °C ± 2 °C until testing begins. Never allow the sample to freeze, since fibre damage will change the results.

If some other standardized or well-established method is used for fines removal, the method shall be mentioned in the test report.

The precision statements in 8.3 are specific only to the method described in 6.3.1. If other methods of removing fines are used, these precision statements are not valid.

6.3.2 Preparation of the test portion by mass and dilution

To obtain at least 3 test portions for coarseness testing from the fibres in the debris-free pulp (from 6.3.1), perform the following procedure:

Tare a clean 5 litre container to an accuracy of 0,1 g. Pour the contents of the vial into the tared container. Rinse the vial and cap to ensure that all the fibres have been transferred to the container. Add about 4 500 ml of distilled, or deionised, water to the container and weigh the contents ($= m_2$).

NOTE 1 Plastic containers, with handles, are recommended to facilitate handling.

The concentration c_A expressed in milligrams per gram, is calculated using the equation

$$c_A = \frac{m_1}{m_2} \tag{1}$$

where

m_1 is the oven-dry mass of fibres in the vial, in milligrams;

m_2 is the mass of the fibres and water mixture in the container, in grams.

Tare a clean beaker, to within 0,1 g. The beaker, or other container, should conform to the manufacturers' requirements.

NOTE 2 Typically a 600 ml beaker is used.

Calculate the mass (m_3), expressed in grams, of the fibre/water mixture to be transferred to the beaker using the equation

$$m_3 = \frac{c_B M}{c_A} \tag{2}$$

where

M is the final, total suspension mass in the beaker, in grams;

c_B is the gravimetric concentration as specified by the manufacturer of the analyzer, in milligrams per gram.

NOTE 3 Typically for softwoods the fibre concentration $c_B = 0{,}002\ 4$ mg/g, and for hardwoods $c_B = 0{,}001\ 0$ mg/g. Treat mixed stocks as hardwood samples. M depends on the beaker volume. For example: the value of M for a 600 ml beaker would be up to 600 g.

Place the empty tared beaker on the balance.

Ensure that the fibres in the fibre/water mixture are well dispersed when drawing a test portion for testing. Draw the test portion and fill the beaker on the balance with the amount of fibre/water mixture needed (m_3) to within 10 %. Record the mass of fibre/water mixture in the beaker to an accuracy of 0,1 g, and calculate the mass of oven-dry fibre in the beaker:

NOTE 4 A recommended procedure is to pour the fibre/water mixture rapidly back and forth, without splashing, between two clean 5 litre containers. After the last transfer and before the fibres have a chance to settle, add a mass of about m_3 of the mixture to the beaker.

Calculate and record the mass of oven-dry fibre in the beaker (m_4), expressed in milligrams, using the equation

$$m_4 = c_{\mathrm{A}} m_3 \tag{3}$$

Prepare at least two more beakers, using the remainder of the fibre/water mixture in the 5 litre container, as described above. The test portions should be tested soon after preparation.

7 Measurement and verification procedures

7.1 Measurement procedure

For the most precise results, all fibres in each test portion shall be detected and analysed.

Add water to the fibre/water mixture in the beaker until the correct mass (M) of the suspension in the beaker is reached, so that the concentration is equal to or less than that required by the manufacturer for coarseness testing (cf. concentration c_{B}). Follow the instructions to enter the oven-dry mass of the test portion into the analyzer and then start the test.

7.2 Verification procedure

Check the performance of the analyzer regularly using a reference pulp and always after cleaning. A verification procedure shall include a calibration check every week, and a performance check every month. Follow the procedures as presented in ISO 16065-1.

8 Calculation and expression of results

8.1 Total fibre length

The total length of fibres in the test portion L_{T}, expressed in metres, is calculated using the equation

$$L_{\mathrm{T}} = \sum l_i \tag{4}$$

where l_i is the length of the ith fibre, in metres.

8.2 Fibre coarseness

The fibre coarseness of the test portion C_k, expressed in milligrams (oven-dry mass) per metre, is calculated using the equation

$$C_k = \frac{m_4}{L_{\mathrm{T}}} \tag{5}$$

where

C_k is the fibre coarseness of the Kth test portion

m_4 is the mass of the oven-dry fibres in the test portion (from Equation (3)), in milligrams

Calculate the mean fibre coarseness C, using the equation

$$C = \frac{\sum C_k}{n} \tag{6}$$

where n is the number of test portions tested.

8.3 Precision

8.3.1 General

A precision statement for this International Standard is based on work published in a peer reviewed journal (see reference [3]). The estimates of precision are based on hardwood and softwood reference pulps available from NIST.

There is no indication that the precision should be different between chemical and mechanical pulps, because the fines are washed out in 6.3.1 (see reference [3]).

Eleven laboratories participated with 17 instruments representing the different manufacturers whose apparatus met the apparatus specifications of ISO 16065-1.

8.3.2 Repeatability

The hardwood and softwood pulps were tested in 11 different laboratories according to this International Standard. The pooled repeatability was determined and the results are shown in Table 1.

Table 1 — Pooled repeatability for determination of mean fibre coarseness

Sample	Mean fibre coarseness mg/m	Coefficient of variation %
Hardwood	0,085	4,3
Softwood	0,140	4,0

8.3.3 Reproducibility

The hardwood and softwood pulps were tested in 11 different laboratories according to this International Standard. The results are shown in Table 2.

Table 2 — Reproducibility for determination of mean fibre coarseness

Sample	Mean fibre coarseness mg/m	Coefficient of variation %
Hardwood	0,085	10,5
Softwood	0,140	5,1

9 Test report

The test report shall give the following information:

a) reference to this International Standard;

b) the date and place of testing;

c) all information for complete identification of the sample;

d) the type of instrument used;

e) the total amount of fibres;

f) the total fibre length (the other measures, length-weighted and mass-weighted fibre length are not defined in this method);

g) the mean sample coarseness;

h) debris removal, if relevant;

i) any operations not specified in this International Standard, e.g. measurement with fines retained, or in the International Standards to which reference is made, or regarded as optional, which might have affected the results.

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[3] ROBERTSON, A.G., OLSON, J.A., ALLEN, P., CHAN, B., SETH, R. *Measurement of fiber length, coarseness, and shape with the fiber quality analyzer*. In Tappi J., **82**:10 (1999), pp. 93 to 98

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