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ISO 16065-2

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Pulps — Determination of fibre length by automated optical analysis —

Part 2: Unpolarized light method

Pâtes — Détermination de la longueur de fibre par analyse optique automatisée —

Partie 2: Méthode de la lumière non polarisée

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

ISO 16065 consists of the following parts, under the general title *Pulps — Determination of fibre length by automated optical analysis*:

— *Part 1: Polarized light method*

— *Part 2: Unpolarized light method*

This second edition cancels and replaces the first edition (ISO 16065–2:2007), of which it constitutes a minor revision with the following changes:

— a new precision statement that complies with the requirements of ISO/TR 24498 has been added.

Pulps — Determination of fibre length by automated optical analysis —

Part 2: Unpolarized light method

1 Scope

This part of ISO 16065 specifies a method for determining fibre length by automated optical analysis using unpolarized light.

The method is applicable to all kinds of pulp. However, fibrous particles shorter than 0,2 mm are not regarded as fibres for the purposes of this part of ISO 16065 and, therefore, are not included in the results.

NOTE ISO 16065-1 deals with the determination of fibre length using polarized light.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 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 > 85 °C*

ISO 7213, *Pulps — Sampling for testing*

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
mean length
L
total length of all fibres counted divided by the number of fibres

Note 1 to entry: See Formula (3).

3.3
length-weighted mean length
L_l
average of the length-weighted fibre-length distribution

Note 1 to entry: See Formula (4).

3.4
mass-weighted fibre length
L_w
average of the mass-weighted fibre-length distribution

Note 1 to entry: See Formula (5).

4 Principle

The fibres suspended in the water are routed through a measuring cell, where the lengths of the individual fibres are measured. A suitable unpolarized light source is used to create the high-contrast image between the fibres and the background. The numerical and weighted average fibre lengths and fibre-length distributions of the pulp are calculated.

5 Apparatus and auxiliary materials

Ordinary laboratory equipment and the following are required.

5.1 Fibre-length analyser, consisting of a measurement section and a sample transport system.

The measurement section consists of a measuring cell, through which the fibres in the water are drawn. There is a uniform unpolarized light source on one side of the cell and a sensitive detector [for example, a charge coupled device (CCD) camera] on the same or on the opposite side of the measuring cell. The flow orientates the fibres into a focal depth no thicker than 0,5 mm normal to the sample flow. The sample flow can be stopped when the CCD camera takes a picture. The detector indicates the fibre length from the image of the fibre. The analyser shall have a resolution that is equal to or better than 100 µm over the range 0 mm to 7 mm, and an accuracy of at least 0,01 mm.

5.1.1 Measuring system, constructed so that air bubbles and non-fibrous particles greater than 0,2 mm do not have any effect on the results.

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

5.3 Verification fibres, made of rayon with suggested lengths of about 0,5 mm, 3,0 mm, and 7,0 mm, with an accuracy of 0,01 mm.

The fibres shall be provided by the producer of the analyser, together with statistical data showing the mean length and the length distribution of each type of verification fibre.

5.4 Reference pulp[1)], a commercial reference pulp is available. A quantity of pulp for which the length-weighted fibre length has previously been determined, using this part of ISO 16065.

NOTE In-house reference pulp can also be used.

1) Reference pulp is available, for example, from the device supplier or the National Institute of Science & Technology, Gaithersburg, MD, USA (NIST). The reference pulp is provided in sheet form. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

5.5 Dilution water, free of non-fibrous material and air bubbles greater than 5 µm, and having an evaporation residue less than 50 mg/l.

Non-fibrous particles can be removed from the water by filtering (pore size of 5 µm) and the air bubbles can be removed by the application of heat, vacuum, and/or a settling time.

6 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 the specimens so that they are representative of the whole sample.

7 Preparation of sample

7.1 Disintegration

If the sample is in dried form, determine the dry matter content, in accordance with ISO 638. If the sample is in slush form, determine the stock concentration, in accordance with ISO 4119.

NOTE It is preferable to measure the never-dried pulps without disintegration, because excessive disintegration can generate fines and reduce the fibre length in some pulps.

7.1.1 Dry pulp samples

If the sample is in dried form, tear the specimens into pieces before soaking. Tear the pieces evenly throughout the thickness of the pulp sheet. By visual assessment, ensure that the fibres are properly separated. Do not cut the sample, as this will cause fibre shortening. Soak the specimen, according to the relevant part of ISO 5263.

Disintegrate the specimens, if required (see the note in 7.1), using the apparatus described in the relevant part of ISO 5263. If the disintegrator described in ISO 5263 is used, the soaking time, the oven-dry mass of the pulp, the amount of water to be used in the disintegration, and the number of revolutions are specified in the relevant part of ISO 5263. Use the dilution water (5.5). For the removal of latency from the mechanical pulps, follow the recommendations given in ISO 5263-3.

For the pulps containing the fibre bundles [e.g. Canadian high-freeness thermo-mechanical pulp (TMP) and chemithermo-mechanical pulp (CTMP)], it can be difficult to measure the fibre length, since the fibre bundles can plug the flow cell. If plugging occurs, then screening is recommended to remove the fibre bundles. Screening can bias the results since it removes the fibre bundles, which preferentially contain the longer fibres. After screening, ensure that the fibres are completely separated and fully dispersed.

WARNING — Very long fibres (e.g. hemp, cotton, and flax) can require some special sample preparation techniques if they are so long that part of the fibres are outside the measuring window, or if they cause blocking of the fibre-orientating cell.

7.1.2 Wet pulp samples

Dilute to the consistency required for accurate measurements, following the instructions in 7.2.

The wet pulp samples can contain the fibre bundles, and if plugging occurs, follow the instructions given in 7.1.1.

7.2 Stock dilution

Stir and take an aliquot from the uniformly dispersed specimen from 7.1. Dilute by serial dilution, to the concentration recommended by the instrument's manufacturer, or to that determined by the tests over

the concentration range. When taking the sample, the diluted suspension shall be agitated continuously. Do not stir with a rotary movement because the sample dilution will not be homogenous.

NOTE Pouring the test sample back and forth will achieve the desired agitation.

The recommended mass fraction to be used in this method is 0,010 % to 0,025 % for softwood pulp and 0,004 % to 0,010 % for hardwood pulp. Mixed stocks should be treated as hardwood pulps. In order to get accurate measurements, the suspension shall be diluted using the dilution water (5.5).

8 Procedure

8.1 Measurement procedure

The suspension shall be agitated continuously to ensure complete mixing. Take at least a 50-ml test portion from the continuously agitated dilute sample. Carry out the test according to the instructions of the instrument manufacturer. The minimum number of fibres to measure shall be that at which the mean length reaches a steady-state variance of 0,01 mm (i.e. measuring more fibres does not change the mean-length value by more than 0,01 mm). If the apparatus does not continuously provide fibre-length values during a test, a minimum of 5 000 fibres shall be measured.

8.2 Verification procedure using verification fibres

8.2.1 General

Check the performance of the analyser regularly and always after cleaning. A verification procedure shall include a calibration check every week and a performance check every month. If the analyser is used infrequently, check the calibration prior to each use.

8.2.2 Calibration check with verification fibres

Run a calibration check using the verification fibres (5.3).

In the check, record the data for at least 5 000 fibres or until a 1 % coefficient of variation (CV) level on the mean-length value is achieved. Prepare a new portion of calibration fibres for each check.

Use only the verification fibres that have been dispersed on the same day as the calibration check is made, since rayon fibres tend to flocculate.

Stir the fibre suspension, when taking an aliquot from the suspension. Ensure that the fibres do not form flocks. If they do, the calibration check cannot be carried out.

It is very important that the pulp suspension is continuously agitated, to prevent the fibres from settling.

Compare the fibre-length data obtained with the data provided for the verification fibres by the manufacturer. If the results of the calibration check lie outside the tolerance limits given, clean the system and run a new calibration check. If the new data still lie outside the tolerance limits, follow the recommendations given by the manufacturer of the analyser.

8.2.3 Performance check using reference pulp

The calibration check is not sufficient to give a true picture of the functioning of the analyser. Check the performance of the analyser every month using fibres from a reference pulp (5.4).

Prepare and analyse the reference-pulp sample, following the procedure described in this part of ISO 16065. Compare the data achieved with the specifications of the supplier of the reference pulp, or with the previous performance checks if an in-house reference pulp is used. The tolerance limit for a length-weighted fibre length of chemical pulps is ±1,5 %.

If the check data lie outside the tolerances given, clean the analyser and run a new check. If the data are still outside the tolerances given, contact the manufacturer of the analyser for service.

Ensure that the reference material is available for future performance checks. If not, select a suitable material for use as a reference pulp and determine its length-weighted fibre length, using this part of ISO 16065 to provide a basis for future comparisons.

9 Calculation and expression of results

9.1 Method of calculation

The number of fibres (n_i) in each class of length (l_i) is counted.

For each class, the percentage frequency by number, f_i, is calculated using Formula (1)

$$f_i = \frac{n_i}{\sum n_i} \times 100 \tag{1}$$

and the percentage length-weighted frequency, f'_i, is calculated using Formula (2)

$$f'_i = \frac{n_i l_i}{\sum n_i l_i} \times 100 \tag{2}$$

where

n_i is the number of fibres in the ith class;

l_i is the central length of the ith class, in millimetres;

$\sum n_i$ is the total number of fibres in all classes;

$\sum n_i l_i$ is the sum of the products, $n_i \times l_i$, for all classes.

9.2 Characteristic distribution values

9.2.1 Lengths

Formulae (3), (4), and (5) are used for calculating the required quantities and the mean fibre length (other quantities can also be calculated for particular purposes).

a) The mean length of the fibres:

$$L = \frac{\sum n_i l_i}{\sum n_i} \tag{3}$$

NOTE 1 The numerical mean fibre length is not always the most meaningful indicator of the fibre length, because the effect of short fibres is emphasized. A better formula is often the length-weighted mean fibre length.

b) The length-weighted mean length of the fibres (L_l):

$$L_l = \frac{\sum n_i l_i^2}{\sum n_i l_i} \tag{4}$$

c) The mass-weighted fibre length of the fibres (L_w):

$$L_w = \frac{\sum n_i l_i^3}{\sum n_i l_i^2} \tag{5}$$