INTERNATIONAL STANDARD

ISO 16065-1

Second edition
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Pulps — Determination of fibre length by automated optical analysis —

Part 1: Polarized light method

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

Partie 1: Méthode de la lumière polarisée

Reference number
ISO 16065-1:2014(E)

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

This second edition cancels and replaces the first edition (ISO 16065-1:2001), 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.

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*

Pulps — Determination of fibre length by automated optical analysis —

Part 1: Polarized light method

1 Scope

This part of ISO 16065 specifies a method for determining fibre length using polarized light.

This part of ISO 16065 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 to be included in the results.

NOTE ISO 16065-2 deals with the determination of fibre length using unpolarized 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 degrees C*

ISO 5263-3, *Pulps — Laboratory wet disintegration — Part 3: Disintegration of mechanical pulps at > 85 degrees C*

ISO 7213, *Pulps — Sampling for testing*

ISO/TR 24498, *Paper, board and pulps — Estimation of uncertainty for test methods*

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 a component of a light wave which is vibrating in a particular direction, which is 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

3.5
birefringence
<cellulosic fibres> 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 1 to entry: 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
mean length
L
total length of all fibres counted divided by the number of fibres

Note 1 to entry: See Formula (3).

3.7
length-weighted mean length
L_l
average of the length-weighted fibre-length distribution

Note 1 to entry: See Formula (4).

3.8
mass-weighted mean length
L_w
average of the mass-weighted fibre-length distribution

Note 1 to entry: See Formula (5).

Note 2 to entry: Mass-weighted mean length was formerly called weight-weighted mean length.

3.9
light extinction
difference, expressed as a percentage, in the maximum and minimum light level transmitted through two axially aligned polarizers when one polarizer is rotated 90° about the axis

4 Principle

Fibres suspended in water are routed through a fibre orienting cell (FOC). The projected lengths of individual fibres are measured automatically. A crossed-polarizers setup is used to discriminate between fibres and other materials like air bubbles, which do not rotate the plane of polarization. The numerical and weighted average fibre lengths and fibre-length distributions of pulp are calculated.

5 Apparatus and materials

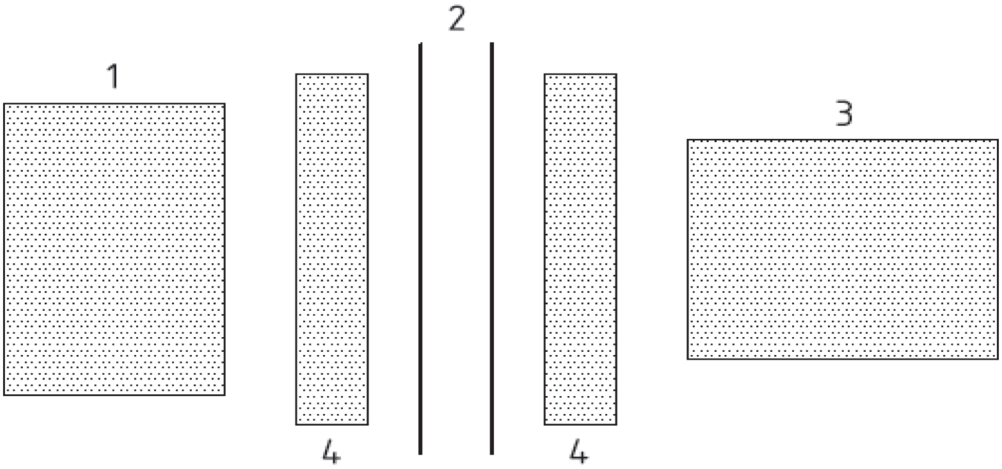
Ordinary laboratory equipment and the following are required.

5.1 Fibre-length analyser, consisting of a measurement section and a sample transport system (see Figure 1).

The measurement section consists of a fibre orienting cell (FOC), through which fibres in a liquid are drawn. There is a uniform light source on one side of the FOC and a photo sensor matrix on the opposite side. There are crossed polarizing filters, on either side of the FOC, between the light source and the photo sensor matrix. The photo sensor matrix senses the length of the image of the fibre from the light that due to the birefringence is transmitted through the second polarizer. The length of this image is converted to fibre length. The flow orientates the fibres into a plane or tube normal to the light path and no thicker than 0,5 mm in the direction of the light path. The resolution of the analyser shall be equal to or better than 100 µm over the range 0 mm to 7 mm.

NOTE Fibres constrained to flow through a capillary no wider than 0,5 mm have been found to straighten enough to be measured accurately with projected light.

At least 90 % of the transmitted light spectrum shall be within the spectral sensitivity of the detector. The light extinction of the crossed polarizers shall exceed 99 %. The detection efficiency shall be 100 % for all fibres 0,100 mm and longer.



Key

1 light source

2 FOC

3 photo sensor

4 polarizing optics

Figure 1 — An example of the measurement principle

5.2 Disintegrator, as described in ISO 5263.

5.3 Pipette, having a volume of 50 ml ± 0,5 ml, with a tip opening of at least 5 mm for sampling a 50 ml test portion.

5.4 Verification fibres, made of rayon or other suitable material, with suggested lengths of about 0,5 mm, 3,0 mm, and 7,0 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.

5.5 Reference pulp[1].

1) Reference pulp is available, for example, from 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 the product named.

6 Sampling and preparation of sample

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

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 never-dried pulps without disintegration because excessive disintegration can generate fines and reduce fibre length in some pulps.

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. Do not cut the sample as this will cause fibre shortening. Soak the specimen according to ISO 5263.

Disintegrate the specimens, if required (see note).

Soaking time, oven-dry mass of the pulp, the amount of water to be used in the disintegration, and the number of revolutions are specified in ISO 5263. For the removal of latency from mechanical pulps, follow the recommendations given in ISO 5263.

For pulps containing fibre bundles, it can be difficult to measure the fibre length since the fibre bundles can plug the flow cell. If plugging occurs, then a screening is recommended to remove the fibre bundles. Screening can bias the results since it will remove fibre bundles which preferentially contain longer fibres. Very long fibres (e.g. hemp, cotton, flax), if they cause blocking problems of the fibre orientating cell, can require some special sample preparation techniques.

6.3 Stock dilution

After disintegration, make sure that the fibres are properly separated and fully dispersed. Stir and take an aliquot. Dilute the aliquot with water to a volume of 5 l. The concentration shall be 0,010 % to 0,025 % (mass fraction) for softwood pulp and 0,004 % to 0,010 % (mass fraction) for hardwood pulp. Mixed stocks should be treated as hardwood pulps. If necessary, dilute the suspension according to the instructions of the apparatus.

7 Measurement and verification procedures

7.1 Measurement procedure

With a pipette (5.3), take a 50 ml test portion from the continuously agitated dilute sample. The suspension shall be agitated continuously to ensure complete mixing. Move the pipette both horizontally and vertically during sampling. Run the determination procedure according to the instructions of the instrument manufacturer. The minimum number of fibres to measure shall be that at which the average length reaches a steady-state variance of 0,01 mm (i.e. measuring more fibres does not change the average length value by more than 0,01 mm). If the apparatus does not provide continuous fibre-length values during a test, then a minimum of 5 000 fibres is recommended.

7.2 Verification procedure

7.2.1 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.

7.2.2 Calibration check with verification fibres

Run a calibration check using the verification fibres (5.4). Fibres with three different fibre lengths shall be used.

In the check, record data for at least 6 000 fibres or until 1 % coefficient of variation (CV) level is achieved. Prepare a new portion of calibration fibres for each check.

Use only 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, not by circulatory movements but by moving the pipette (5.3) horizontally and vertically, when taking an aliquot from the suspension. Ensure that the fibres do not form flocs. If they do, the calibration check cannot be carried out.

It is very important that the pulp suspension is continuously agitated to prevent 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.

7.2.3 Performance check with reference pulp

The verification 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 (5.5).

Prepare and analyse the reference-pulp sample following the procedure described in this part of ISO 16065. Compare the data achieved with data from previous performance checks. The tolerance limit for 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 reference material is available for future performance checks. If not, select a suitable material for use as a reference pulp and make a measurement to provide a basis for future comparisons.

8 Calculation and expression of results

8.1 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)

$$f_i = \frac{n_i}{\sum n_i} \times 100 \quad (1)$$

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

$$f'_i = \frac{n_i l_i}{\sum n_i l_i} \times 100 \quad (2)$$

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.

8.2 Characteristic distribution values

8.2.1 Lengths

The following characteristic distribution values are commonly calculated (other quantities may also be calculated for particular purposes):

a) The mean length of individual fibres:

$$L = \frac{\sum n_i l_i}{\sum n_i} \quad (3)$$

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 expression is often the length-weighted mean fibre length.

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

$$L_l = \frac{\sum n_i l_i^2}{\sum n_i l_i} \quad (4)$$

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

$$L_w = \frac{\sum n_i l_i^3}{\sum n_i l_i^2} \quad (5)$$

NOTE 2 In the interpretation of the length-weighted mean length, it is assumed that all fibres have identical coarseness. In the interpretation of the mass-weighted mean length, it is assumed that fibre coarseness is proportion to the length. This proportionality does not hold for mechanical pulps.

8.2.2 Coefficient of variation

Calculate the coefficient of variation (%) from the frequency distribution using Formula (6)

$$\mathrm{CV} = \frac{s}{L} \times 100 \tag{6}$$

where the standard deviation s, in millimetres, is given by Formula (7)

$$s = \left(\frac{\sum (l_i - L)^2 n_i}{\sum n_i} \right)^{1/2} \tag{7}$$

However, if the values L and L_l are calculated, the coefficient of variation (%) may be calculated using Formula (8)

$$\mathrm{CV} = 100 \left(\frac{L_l}{L} - 1 \right)^{1/2} \tag{8}$$

8.2.3 Expression of the frequency distribution

If a length distribution graph is required, express it

— by a frequency diagram representing the number and/or the percentage of the fibres in each length interval employed, expressed as a function of the length and/or

— by a cumulative frequency diagram indicating the percentage as a function of fibres greater than a given length, expressed as a function of the length.

9 Test report

The test report shall give the following information:

a) reference to this part of ISO 16065 (i.e. ISO 16065-1)

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 length-weighted mean length and mass-weighted mean length of the fibres, and, if required, the mean length;

g) the frequency diagram and the cumulative frequency diagram, if a distribution graph is required;

h) the class intervals used, if required;

i) the number of fibres in each class, if required;

j) any operations not specified in this part of ISO 16065 or in the International Standards to which reference is made or regarded as optional, which might have affected the results.

Annex A
(informative)

Precision

A.1 General

The precision data are based on three pulp samples (two whole pulps and one R-14 Bauer-McNett fraction) tested by 11 different laboratories, using either the FQA or the FS-200 commercial apparatus, according to this part of ISO 16065.

The whole pulps were commercial, dried, bleached kraft produced from softwood or hardwood. The R-14 Bauer-McNett fraction of the softwood pulp was also tested. The samples were provided as slurries of known fibre concentrations. Similar testing procedures were followed by each laboratory. Ten measurements were made on each sample.

The calculations have been made according to ISO/TR 24498.

The repeatability and reproducibility limits reported are estimates of the maximum difference which should be expected in 19 of 20 instances, when comparing two test results for material similar to those described under similar test conditions. These estimates could not be valid for different materials or different test conditions.

NOTE Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where $2,77 = 1,96\sqrt{2}$.

The pooled repeatability and reproducibility data are presented in Tables A.1 and A.2, respectively.

A.2 Repeatability

Table A.1 — Estimation of the repeatability of the test method

Sample	Number of laboratories	Length-weighted mean fibre length (mm)	Standard deviation s_r (mm)	Coefficient of variation $C_{V,r}$ (%)	Repeatability limit r (mm)
Hardwood, whole pulp	11	0,65	0,009 6	1,48	0,027
Softwood, whole pulp	11	2,22	0,022	0,97	0,060
Softwood, R-14 fraction	11	3,09	0,024	0,79	0,068

A.3 Reproducibility

Table A.2 — Estimation of the reproducibility of the test method

Sample	Number of laboratories	Length-weighted mean fibre length (mm)	Standard deviation s_R (mm)	Coefficient of variation $C_{V,R}$ (%)	Reproducibility limit R (mm)
Hardwood, whole pulp	11	0,65	0,020	3,1	0,057
Softwood, whole pulp	11	2,22	0,063	2,8	0,18
Softwood, R-14 fraction	11	3,09	0,093	3,0	0,26

Bibliography

[1] Clark J. D'A. *Pulp Technology and Treatment for Paper*. Chapter 17. Miller Freeman Publications Inc, San Francisco, Second Edition, 1985

[2] Ilvessalo-Pfäffli M-S., Alfthan, G. *The measurement of Fibre Length With a Semi-Automatic Recorder*. In Paperi ja Puu 39:11 1957, pp 509 to 516

[3] Olson J.A., Robertson A.G., Finnigan T.D., Turner R.H.R. *An Analyzer for Fibre Shape and Length*. In JPPS, 21:11 1995, pp J367 to J373

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