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ISO 9184-1

Second edition
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Paper, board and pulps — Fibre furnish analysis —

Part 1: General method

Papier, carton et pâtes — Détermination de la composition fibreuse —

Partie 1: Méthode générale

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 9184-1:1990), which has been technically revised.

The main changes are as follows:

— addition of a new quantitative testing method in 9.3.3.

A list of all parts in the ISO 9184 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Paper, board and pulps — Fibre furnish analysis —

Part 1:
General method

1 Scope

This document specifies the general procedure for fibre furnish analysis of paper, board and pulps.

It is applicable to all kinds of pulps and to most papers and boards, including those containing more than one kind of fibre, taking into account different pulping processes.

This method is less suitable for heavily impregnated or highly coloured papers and boards, which cannot be dispersed or decoloured without affecting the structure or the staining reactions of the fibres.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 9184-2, *Paper, board and pulps — Fibre furnish analysis — Part 2: Staining guide*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
fibre furnish analysis
determination of the fibre components of paper, board and pulp samples as regards the species of fibres and the method of processing

Note 1 to entry: The fibre furnish analysis may be carried out qualitatively or quantitatively.

3.2
fibre coarseness
c
mass (oven dry) per unit length for a particular type of fibre, generally expressed in milligrams per metre

Note 1 to entry: Fibre coarseness can be determined according to ISO 9184-6 or ISO 23713.

3.3
weight factor
f
ratio of the fibre coarseness of a particular type of fibre to that of a reference fibre

Note 1 to entry: Weight factor can be determined according to ISO 9184-7.

4 Principle

The fibre furnish analysis is carried out under the microscope, or using a fibre analyzer, on a small quantity of stained fibres representative of the sample being tested:

— qualitatively, on the basis of the stain reactions and the morphological characteristics of the fibres;

— quantitatively, by counting the number of crossings of different kinds of fibres with a counting line, or measuring the length of each fibre type within a certain area, and transforming the number of counts or the lengths into the percentages by weight of each type of fibre by the application of weight factors.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Sodium hydroxide, solution, a mass fraction of about 1 %, containing 10 g sodium hydroxide (NaOH) per litre.

5.2 Hydrochloric acid, solution, a mass fraction of about 0,2 %, containing 5 ml of concentrated 36 % to 38 % (mass fraction) hydrochloric acid (HCl) per litre.

5.3 Phosphoric acid, solution, a mass fraction of about 5 %, containing 35 ml of 85 % (mass fraction) phosphoric acid (H_3PO_4) per litre.

5.4 Aluminium sulfate, solution, a mass fraction of about 5 %, containing 50 g of aluminium sulfate [$Al_2(SO_4)_3$] per litre.

5.5 Potassium permanganate, solution, a mass fraction of about 6,5 %, containing 65 g of potassium per-manganate ($KMnO_4$) per litre.

5.6 Oxalic acid, solution, a mass fraction of about 5 %, containing 50 g of oxalic acid ($C_2H_2O_4.2H_2O$) per litre.

5.7 Organic solvents, ethanol (C_2H_5OH), diethyl ether ($C_2H_5OC_2H_5$), ethyl acetate ($CH_3COOC_2H_5$), acetone (CH_3COCH_3), xylene ($C_6H_4(CH_3)_2$), toluene($C_6H_5(CH_3)$), chloroform ($CHCl_3$), tetrachloroethene (C_2Cl_4) and trichloroethane ($C_2H_3Cl_3$).

6 Apparatus

6.1 Microscope, equipped with a mechanical stage and cross-hair, central dot or horizontal line with centre marking eye-pieces.

Illumination: daylight lamp or normal vacuum lamp with daylight filter.

For the identification and counting of fibres a magnification of ×40 to ×120, and for the study of structural details ×200 to ×500 is recommended.

6.2 Fibre Analyzer, shall include the following parts.

— **Imaging system**, including a computer, a microscope, a digital imaging acquisition unit and an image transmission and conversion unit. It shall accurately synchronize the images observed under the microscope lens to the computer monitor for observation and determination

— **Measuring system**, including a counting method mode in which the number of fibres of each particular type crossing a line are accurately counted and a length method mode in which the length of each fibre labelled by the operator is measured to an accuracy of 0,01 mm and the total length of each type of fibre is calculated.

— **Calculation system** that accepts entry of the weight factor for each type of fibre present and calculates the percentage by weight of each type of fibre to an accuracy of 0,1 %.

The fibre analyzer does not identify the types of fibres present. An operator skilled in fibre identification is required.

6.3 Dispersers, one for easily dispersed samples (e.g. a low-speed agitator) and the other for more resistant samples (e.g. an ultra-sonic disperser or high-speed macerator).

6.4 Infra-red lamp or hot-plate capable of being maintained at 50 °C to 60 °C.

6.5 Filtering devices.

6.5.1 Round sieve, diameter 50 mm to 70 mm, with metal or plastic edge of height 5 mm to 10 mm. The sieve bottom shall be made of woven wire cloth, aperture size 60 µm to 80 µm.

6.5.2 Glass filter, 200 ml, with sintered disc of pore size 15 µm to 40 µm.

6.6 Dropper, a glass tube about 100 mm in length and internal diameter 5 mm to 8 mm, with one end carefully smoothed but not constricted, and the other end fitted with a rubber bulb. The dropper shall be designed to discharge approximately 0,5 ml.

6.7 Microscope slides, recommended size 25 mm × 75 mm.

6.8 Rectangular microscope cover glasses, recommended size 22 mm × 32 mm.

6.9 Dissecting needles.

6.10 Petri dishes, or suitable shallow, covered dishes, approximately 100 mm to 120 mm in diameter.

6.11 Multiple counter, for recording counted fibres.

7 Preparation of the test piece

7.1 General

Take a test piece by tearing small pieces from different parts of the sample, about 0,25 g in total. For multilayered samples, take a test piece in accordance with 7.5.

7.2 Ordinary samples

7.2.1 Boiling in water

Place the test piece in a test tube or a small beaker. Boil torn pieces in water for a few minutes, stirring occasionally, and disperse in a disperser (6.3).

7.2.2 Boiling in sodium hydroxide solution

If the pieces cannot be completely dispersed in accordance with 7.2.1, place the pieces after filtering with a filtering device (6.5) in a test tube or beaker. Boil the pieces in sodium hydroxide solution (5.1) for a few minutes, with occasional stirring.

Samples containing wool fibres or natural silk, should not be treated with sodium hydroxide, because wool and silk are soluble in alkali. Boiling in a sodium hydroxide solution can also affect the development of certain stains.

Filter on a glass filter (6.5.2), wash twice with water and neutralize with hydrochloric acid (5.2) for several minutes. Wash several times with water, and disperse in a disperser (6.3).

7.3 Specially treated samples

7.3.1 General

If these treatments do not disperse the pieces, choose one of the treatments described in 7.3.2 to 7.3.4.

7.3.2 Wet-strength Papers

Place test piece of the sample in a beaker, add aluminium sulfate solution (5.4) or phosphoric acid solution (5.3) and boil for 5 min to 30 min, depending on the dispersing velocity. Decant the solution, wash with water and disperse. If the pieces are not dispersible, proceed to 7.3.4.

Hypochlorite bleach has also been found effective for dispersing these products.

7.3.3 Vegetable parchment and papers of highly beaten pulp

Place test piece of the sample in potassium permanganate solution (5.5) in a beaker and allow to stand for 1 h. Decant the solution, wash the pieces, treat with oxalic acid solution (5.6), wash again and disperse.

7.3.4 Impregnated or specially bonded samples having chemically or physically durable inter-fibre bonds

No general rule can be given. Extraction, cold or hot, with organic solvents (5.7) can often facilitate the disintegration. Choose a solvent that does not affect the fibres.

7.4 Coloured samples

In the unlikely event that after disintegration the fibres are still coloured to such an extent as to render their identification difficult, methods depending on the characteristics of the dyestuff may be used to remove the colour. These methods include the extraction, oxidation and reduction treatments with the requisite reagents in normal laboratory use.

7.5 Multilayered samples

When the paper or board sample is expected to be multilayered and two or more layers shall be analysed separately, proceed as follows. From the sample, cut five pieces, about 5 cm x 5 cm in size, and immerse in hot water (about 70 °C) until the pieces can be separated into the component layers. If separation is

difficult, cut another set of five pieces, about 5 cm x 5 cm in size, and use sodium hydroxide solution (5.1) instead of water. If the separated layers seem to contain fibres from the neighbouring layers, try to remove them by rubbing them gently while wet. Treat the layers as separate test pieces and proceed in accordance with 7.2.

8 Staining and preparation of fibre slides

8.1 General

The method of staining and preparation of slides depends on the stain used. Choose the appropriate stain from the staining guide in ISO 9184-2 and perform the staining of the fibres on the slide or in the test tube.

NOTE While the stains recommended in ISO 9184-2 have proven effective for differentiating various fibre types, there are a number of other stains that can be usefully employed in certain cases.

The fibre slide for staining can be prepared either from a dilute fibre suspension or from a filtered fibre pad.

8.2 Staining on a slide

8.2.1 Preparation from the fibre suspension

Dilute about one-half of the dispersed fibre suspension (see Clause 7) in a beaker to a concentration of about 0,05 % (mass fraction). By means of a dropper (6.6), transfer about 0,5 ml of the diluted suspension on to a clean, grease-free microscope slide (6.7), and disperse the fibres evenly with a dissecting needle (6.9) or by tapping the slide gently. Dry the fibre slide on the hot-plate or under the infra-red lamp (6.4) and allow to cool.

Apply the stain in accordance with the relevant method and put on a cover glass (6.8) avoiding air bubbles. Allow to stand for 1 min to 2 min and drain off the surplus stain preferably by tilting the long edge of the slide into contact with a blotter.

8.2.2 Preparation from the fibre pad

Filter one-half of the dispersed fibre suspension (see Clause 7) on a sieve (6.5.1) or on a glass filter (6.5.2). Place the filtered fibre pad in a small covered dish (6.10) and keep it from drying during the analysis. Transfer a small amount of the fibre pad to the slide and remove excess water with blotting paper. Apply the stain in accordance with the prescribed method, and distribute the fibres evenly with dissecting needles (6.9).

Apply a cover glass (6.8) and remove excess solution with blotting paper, taking care to avoid flocculation of the fibres. The best result is obtained if the fibre slide is tilted and blotted edgewise.

8.3 Staining in a test tube

Take a specimen from the filtered fibre pad (see 8.2.2) and perform the staining in a test tube according to the relevant method. After staining prepare the fibre slide according to 8.2.1 or 8.2.2 using water instead of stain.

More detailed instructions for staining and preparation are given in the relevant staining methods.

9 Procedure

9.1 General

Because the colours developed by certain stains are unstable, the analysis should be carried out shortly after the slide has been prepared.

9.2 Qualitative analysis

Place the stained fibre slide on the mechanical stage of the microscope (6.1) or fibre analyzer (6.2). Slowly and systematically traverse the slide, either horizontally or vertically, line by line, so that the entire fibre field is examined. Identify the species of fibres and the methods of processing on the basis of the morphological characteristics and the colours obtained by staining.

Examine at least two slides. In cases where there are fibres that are difficult to identify, examine one or more additional slides.

Previous experience and knowledge of the stain reactions and of the structural details of the most common papermaking fibres are essential for the identification.

9.3 Quantitative analysis

9.3.1 General

Weight factors are used for quantitative analysis and they can be determined or those given in Annex A may be used. The origin, growth period, pulping method (yield) and bleaching process have considerable influence on the weight factors.

9.3.2 Counting method

9.3.2.1 Measurement using a microscope

Place the stained fibre slide on the mechanical stage of the microscope (6.1). Using the mechanical stage, move the fibre slide so that the centre marking of the eyepiece is 3 mm to 5 mm from the top corner of the cover glass. Then systematically traverse the slide, analysing each fibre, until the whole slide has been analysed as follows:

Slowly traverse the slide either horizontally or vertically. Using a multiple counter (6.11), count and record according to species and method of processing, each fibre or broken fibre as it passes the centre marking. If a fibre passes the centre more than once, count it each time. If a fibre follows the centre for some time, count it only once. Ignore very fine fibre fragments, but keep in mind larger fragments such as split fibres, so that when two or three of the same kind of fibre are observed in the same line, record them as one.

If there is difficulty in counting each kind of fibre during one pass, make repeated counts along the same line, until all the fibres are counted. Take care not to move the slide from the original line during the subsequent counts and return to the original line if any movement occurs.

When every fibre in the line has been counted, move the slide about 5 mm to a new line and count the fibres as described above. Repeat this process until the entire slide has been examined. The total number of fibre crossings counted shall be at least 600, which can require two or more slides.

NOTE If the colour difference between the different species of fibre is insufficient to distinguish them, counting can be done partly or, in some cases, entirely on the basis of the morphological characteristics.