INTERNATIONAL STANDARD

ISO 9184-1

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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

Reference number
ISO 9184-1:1990(E)

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.

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.

International Standard ISO 9184-1 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

ISO 9184 consists of the following parts, under the general title *Paper, board and pulps — Fibre furnish analysis*:

— *Part 1: General method*

— *Part 2: Staining guide*

— *Part 3: Herzberg staining test*

— *Part 4: Graff "C" staining test*

— *Part 5: Lofton-Merritt staining test (modification of Wisbar)*

— *Part 6: Weight factors by fibre coarseness method*

— *Part 7: Weight factors by comparison method*

Part 1 gives general instructions for the performance of fibre furnish analysis. It should be used in conjunction with the staining guide (see part 2) and the staining tests (see parts 3 to 5).

Additional parts of this International Standard will be published if required by the development of new kinds of fibres or new staining tests.

Annexes A and B of this part of ISO 9184 are for information only.

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Paper, board and pulps — Fibre furnish analysis —

Part 1:
General method

1 Scope

This part of ISO 9184 specifies the general performance of the test for fibre furnish analysis (see 3.1) 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 to 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 reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 9184. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9184 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 9184-2:1990, *Paper, board and pulps — Fibre furnish analysis — Part 2: Staining guide.*

3 Definitions

For the purposes of this part of ISO 9184, the following definitions apply.

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.

The fibre furnish analysis may be carried out qualitatively or quantitatively.

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

3.3 weight factor, f: The ratio of the fibre coarseness of a particular type of fibre to that of a reference fibre.

NOTE 1 Traditionally, cotton staple (rag) fibre was selected as the reference fibre to which all other fibres were compared. The weight factor of cotton fibre was taken as 1,00, and the fibre coarseness of that fibre was determined to be 0,180 mg/m. The weight factor of a particular type of fibre can be derived from its fibre coarseness by the expression

$$f = \frac{c}{0{,}180}$$

where

f is the weight factor;

c is the fibre coarseness, in milligrams per metre.

4 Principle

The fibre furnish analysis is carried out under the microscope 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 various kinds of fibres with the counting line and by transforming the number of

counts into the percentages by weight 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, about 1 % (*m*/*m*), containing 10 g sodium hydroxide (NaOH) per litre.

5.2 Hydrochloric acid, solution, about 0,2 % (*m*/*m*), containing 5 ml of concentrated hydrochloric acid (HCl) per litre.

5.3 Phosphoric acid, solution, about 5 % (*m*/*m*), containing 35 ml of 85 % (*m*/*m*) phosphoric acid (H_3PO_4) per litre.

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

5.5 Potassium permanganate, solution, about 6,5 % (*m*/*m*), containing 65 g of potassium permanganate ($KMnO_4$) per litre.

5.6 Oxalic acid, solution, about 5 % (*m*/*m*), 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_7H_8), chloroform ($CHCl_3$), tetrachloroethene (C_2Cl_4) and trichloroethane ($C_2H_3Cl_3$).

6 Apparatus

Ordinary laboratory equipment and

6.1 Microscope, equipped with a mechanical stage and cross-hair, central dot or horizontal line eyepieces.

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 Dispersers, one for easily dispersible samples (low-speed agitator, etc.) and the other for more resistant samples (ultra-sonic disperser, high-speed macerator, etc.).

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

6.4 Filtering devices

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

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

6.5 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.

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

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

6.8 Dissecting needles

6.9 Petri dishes, or suitable shallow, covered dishes, approximatelly 100 mm to 120 mm in diameter.

6.10 Multiple counter, for recording counted fibres.

6.11 Dropper bottles, 30 ml or 50 ml.

7 Preparation of the test piece

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 7.3.

7.1 Ordinary samples

7.1.1 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 (6.2).

7.1.2 Boiling in sodium hydroxide solution

If the pieces cannot be completely dispersed in accordance with 7.1.1, replace the pieces after filtering (see 6.4) in the test tube or beaker. Boil the pieces in sodium hydroxide solution (5.1) for a few minutes, with occasional stirring.

NOTE 2 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 hy-

droxide solution can also affect the development of certain stains.

Filter on a glass filter (6.4.2), wash twice with water and neutralize with hydrochloric acid (5.2) for several minutes. Wash several times with water and disperse in a disperser (6.2).

7.2 Specially treated samples

If these treatments do not disperse the pieces, choose one of the treatments described in 7.2.1 to 7.2.4.

7.2.1 Wet-strength papers

Place torn pieces 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 7.2.3.

NOTE 3 Hypochlorite bleach has also been found effective for dispersing these products.

7.2.2 Vegetable parchment and papers of highly beaten pulp

Place torn pieces 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.

7.2.3 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) may often facilitate the disintegration. Choose the solvent so that is does not affect the fibres.

7.2.4 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.

7.3 Multilayered samples

When the paper or board sample is expected to be multilayered and two or more layers are to be analysed separately, proceed as follows. From the sample, cut five pieces, about 5 cm × 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, 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 7.1.

8 Staining and preparation of fibre slides

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

NOTE 4 While the stains recommended in ISO 9184-2 have proven effective for differentiating various fibre types, there are a number of other stains which can be usefully employed in certain cases. These stains are described in a number of published references, many of which are given in annex B.

8.1 Staining on a slide

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

8.1.1 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 % (m/m). By means of a dropper (6.5), transfer about 0,5 ml of the suspension on to a clean, grease-free microscope slide (6.6), and disperse the fibres evenly with a dissecting needle (6.8) or by tapping the slide gently. Dry the fibre slide on the hot-plate or under the infra-red lamp (6.3) and allow to cool.

Apply the stain according to the relevant method and put on a cover glass (6.7) 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.

8.1.2 Preparation from the fibre pad

Filter one-half of the dispersed fibre suspension (see clause 7) on a sieve (6.4.1) or on a glass filter (6.4.2). Place the filtered fibre pad in a small covered dish (6.9) 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 according to the prescribed method, and distribute the fibres evenly with dissecting needles (6.8). Apply a cover glass (6.7) 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.

8.2 Staining in a test tube

Take a specimen from the filtered fibre pad (see 8.1.2) and perform the staining in a test tube according to the relevant method. After staining prepare the fibre slide according to 8.1.1 or 8.1.2 using water instead of stain.

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

9 Procedure

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

9.1 Qualitative analysis

Place the stained fibre slide on the mechanical stage of the microscope (6.1). 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 (see annex B) and the colours obtained by staining (see the staining tests).

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[1].

9.2 Quantitative analysis

With 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 slowly and systematically traverse the slide, either horizontally or vertically, line by line, so that the entire field is examined. Count the fibres of each kind in the lines as follows (see note 5):

Using a multiple counter (6.10), count and record 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. Ignore parenchyma and other small cells if they are few as in softwood pulp (see note 6). Count each fibre in a bundle separately.

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. The number of fibre crossings counted should be not less than 600. This may be achieved by counting on not less than two slides.

NOTES

5 If the colour difference between the different kinds of fibre is insufficient, counting should be done partly or, in some cases, entirely on the basis of the morphological characteristics.

6 If the parenchyma cells are numerous, mentally count them as fractions so that when four cells of the same kind of fibre have been observed in the same line, they are added together to give a whole number.

10 Expression of results

10.1 Qualitative analysis

Combine the results obtained by microscopic anaylsis (clause 9) and report the species of fibre and the methods of processing in accordance with clause 11.

Report as one group fibres which are difficult to distinguish from each other, as well as closely related fibres which are present in minor quantities.

10.2 Quantitative analysis

The percentage by weight of each fibre component X_i, is given by the equation

$$X_i = \frac{100 f_i n_i}{\sum_{i=1}^{i=k} f_i n_i}$$

where

f_i is the weight factor;

n_i is the total number of each kind of fibre crossings;

k is the number of fibre components.

Report the percentages by weight of the various kinds of fibres to the nearest whole number.

1) A catalogue listing the reference pulps available can be obtained from the TAPPI Library, Institute of Paper Science and Technology, 575 4th Street, N.W., Atlanta, GA, 30318, USA.

Report percentages less than 2 as "traces".

10.3 Precision

The ideal precision of a quantitative fibre count is primarily a function of the number of fibres counted, and can be calculated for the 95 % confidence limits from the expression

$$p \pm 1{,}96\sqrt{\frac{p(1-p)}{N}}$$

where

p is the fraction of one fibre type;

N is the total number of fibres counted.

The ideal confidence limits for counts where $N = 600$ and $N = 1200$ are given in table 1.

Table 1 — Confidence limits

Proportion of given fibres in total furnish	Confidence limits (±) 95 %	
%	$N = 600$	$N = 1200$
2	1,0	0,8
5	1,7	1,2
10	2,4	1,7
20	3,2	2,3
30	3,7	2,6
50	4,0	2,8
70	3,7	2,6
80	3,2	2,3
90	2,4	1,7
95	1,7	1,2
98	1,0	0,8

Because the actual precision of a quantitative fibre furnish analysis depends on the type of pulp, the colour differences obtained, the accuracy of the weight factors used, and the judgement of the analyst, no generally valid confidence limits can be stipulated, but must be developed for each staining method and counting procedure used.

Where statistical data are available, the number of counts required to attain a desired level of precision should be specified in the relevant standard. Where no precision data are available, not less than 600 fibres should be counted to achieve a precision which may approximate that given in table 1, under ideal conditions.

11 Test report

The test report shall include the following particulars:

a) reference to this part of ISO 9184;

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

c) in the case of a multilayered product, for example a board, clear indications necessary for the identification of the layers analysed separately;

d) the staining procedure used;

e) results from the qualitative analysis, including

- species of fibre,
- pulping processes,
- bleaching;

f) results of the quantitative analysis, in accordance with 10.2, including

- the number of fibres of each type counted,
- the weight factors used, and the source of these factors,
- if parenchyma cells were included;

g) estimate of precision, if available;

h) any unusual features observed in the course of the test;

i) any operations not specified in this part of ISO 9184, which might have affected the results.

Annex A
(informative)

Weight factors

The weight factor varies with the kind of fibre and with the type of pulping. Ideally, separate weight factors should be determined for each kind of fibre present in the furnish under examination. If this is not possible, the values given in table A.1 will serve as a guide.

Table A.1 — Weight factors

Origin of fibres	Weight factor
Cotton staple (rag)	1,0
Softwood chemical pulps	
bleached (most species)	0,9
unbleached (most species)	1,0
douglas fir, inland variety	0,9
douglas fir, coastal variety	1,4
southern yellow pine	1,4
larch	1,1
cedar	0,7
radiata pine	1,2
Softwood dissolving grade pulps	0,85
Softwood semi-chemical sulfite	1,4
Softwood mechanical pulps[1]	
groundwood	1,3
thermo-mechanical pulp	1,7
Softwood chemi-mechanical pulp (many species)	2,0
Hardwood chemical pulps	
birch, aspen, poplar, beech	0,5
maple, willow, hickory	0,4
"gums" : sweet gum, black tupelo, tulip poplar	0,8
eucalyptus, oak	0,45
Hardwood semi-chemical pulps	
birch	0,9
"gums"	1,3
Hardwood mechanical pulps[1]	0,9
Cotton linters	1,25
Bagasse pulp for paper grades	0,75
Esparto, bleached pulp	0,50
Abaca and jute pulps	0,55
Sisal pulp	0,60
Straw pulp for board grades	0,60
Straw, bleached pulp	0,35
Bamboo pulp	0,55
Wool hair	3,1
Flax pulp	0,8
Flax shives	0,4
Synthetic fibres	(see annex B, ref. 11)

1) In particular, the weight factors for mechanical pulps depend on the analyst's personal experience of counting fibre fragments, and may thus also be influenced by the fineness of the pulp.

Annex B
(informative)

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