
**Paper, board, pulps and cellulosic
nanomaterials — Determination of
dry matter content by oven-drying
method —**

**Part 1:
Materials in solid form**

*Papiers, cartons, pâtes et nanomatériaux cellulosiques —
Détermination de la teneur en matières sèches par séchage à
l'étuve —*

Partie 1: Matériaux sous forme solide



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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 World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This first edition of ISO 638-1, together with ISO 638-2, cancels and replaces ISO 638:2008, which has been technically revised. The main changes compared to the previous edition are as follows:

- inclusion of cellulosic nanomaterials and paper and board for recycling in the scope;
- splitting of the standard in two parts;
- technical revision of the procedure;
- editorial revision of the document;
- update of precision clause.

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.

Introduction

Determination of dry matter content and moisture content are carried out for different purposes.

This document is used when the dry matter content is needed to calculate the results for chemical analysis or physical testing, or to determine the moisture content of paper, board, and pulp and cellulosic nanomaterials in solid form, which all may be produced from virgin and/or recycled materials. An example of this is where the results of a chemical analysis for cadmium or manganese are required on the basis of the oven-dry mass of the sample.

ISO 638-2^[1] is dedicated to the determination of the dry matter content or water content of cellulosic nanomaterials in the form of suspensions.

ISO 287^[2] should be used for the purpose of determining the average moisture content and the variation in moisture content (maximum and minimum values) of a lot of paper and board. In the converting of paper and board, moisture content is important as it can have an effect on processes such as printing and copying. Moisture content can have an effect on curl and dimensional stability.

ISO 4119^[3] should be used in laboratory procedures or is referred to in other International Standards in which the concentration of an aqueous pulp suspension requires determination.

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Paper, board, pulps and cellulosic nanomaterials — Determination of dry matter content by oven-drying method —

Part 1: Materials in solid form

1 Scope

This document specifies an oven-drying method for the determination of the dry matter content in paper, board, pulp and cellulosic nanomaterials in solid form, which all can be produced from virgin and /or recycled materials.

It is also applicable to the determination of the dry matter content of paper and board for recycling.

The procedure is applicable to paper, board, and pulp and cellulosic nanomaterials which do not contain any appreciable quantities of materials other than water that are volatile at the temperature of $105\text{ °C} \pm 2\text{ °C}$. It is used, for example, in the case of pulp, paper, and board and cellulosic nanomaterial samples taken for chemical and physical tests in the laboratory, when a concurrent determination of dry matter content is required.

This method is not applicable to the determination of the dry matter content of slush pulp or to the determination of the saleable mass of pulp lots.

NOTE 1 ISO 638-2^[1] specifies an oven-drying method for the determination of the dry matter content of suspensions of cellulosic nanomaterials, ISO 287^[2] specifies the determination of the moisture content of a lot of paper and board; ISO 4119^[3] specifies the determination of stock concentration of pulps; ISO 801 (all parts)^[4] specifies the determination of the saleable mass in lots.

NOTE 2 This document determines the total dry matter content of the sample, including any dissolved solids. If only the cellulosic material content free of dissolved solids is desired, dissolved solids are removed prior to measuring the dry matter content e.g. by washing or dialysis, taking care to retain all cellulosic material; in cases where the sample is filterable without loss of cellulosic solids, ISO 4119^[3] can be used to determine the stock consistency (content of cellulosic material in solid form)

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 186, *Paper and board — Sampling to determine average quality*

ISO 7213, *Pulps — Sampling for testing*

EN 17085, *Paper and board – Sampling procedures for paper and board for recycling*

3 Terms and definitions

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

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1 cellulosic nanomaterial CNM

material composed predominantly of cellulose, with any external dimension in the *nanoscale* (3.5)

Note 1 to entry: The terms nanocellulose (NC) and cellulose nanomaterial (CNM) are synonymous with cellulosic nanomaterial (CNM).

Note 2 to entry: Some cellulosic nanomaterials can be composed of chemically modified cellulose.

[SOURCE: ISO/TS 20477:2017, 3.3.1, modified — “or a material having internal structure or surface structure in the nanoscale, with the internal structure or surface structure composed predominantly of cellulose” deleted from the definition; “cellulose” changed to “cellulosic”; Note 3 to entry deleted]

3.2 constant mass

mass of the test piece determined at the equilibrium condition after drying until the difference between two successive dryings and weighings, separated in time by at least half the initial drying period, does not exceed 0,1 % mass fraction of the test piece before drying

3.3 dry matter content

ratio of the mass of a test piece after drying to *constant mass* (3.2) at a temperature of $105\text{ °C} \pm 2\text{ °C}$ under specified conditions, to its mass before drying

Note 1 to entry: The dry matter content is usually expressed as a percentage mass fraction.

3.4 moisture content

content of water in paper or board, i.e. the ratio of the loss of mass of a test piece, when dried at a temperature of $105\text{ °C} \pm 2\text{ °C}$ under specified conditions, to its mass at the time of sampling

Note 1 to entry: The moisture content is normally expressed as a percentage mass fraction.

[SOURCE: ISO 287:2017, 3.1, modified – “at a temperature of $105\text{ °C} \pm 2\text{ °C}$ ” added.]

3.5 nanoscale

length range approximately from 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from larger sizes are predominately exhibited in this length range.

[SOURCE: ISO/TS 80004 1:2015, 2.1]

3.6 solid form

form in which water is held immobile within the cell wall, and/or lumen and/or interstices between the cellulosic materials, and/or is adsorbed at the cellulosic material surface, and which behaves as a discrete or separate unit that does not of itself flow (or as a set of such units)

4 Principle

Test pieces taken from pulp, paper, or board or cellulosic nanomaterial samples in solid form are weighed before and after drying to constant mass.

From the mass of the test piece before and after drying, the dry matter content is calculated.

5 Apparatus

5.1 Balance,

which can be read to the nearest 1 mg, for weighing test pieces of 2 g and less; for larger test pieces, which can be read to 0,05 % mass fraction of the original moisture-containing test piece.

5.2 Containers, water vapour-proof, with tightly fitting lids, and made from a material (e.g. glass or plastic) not affected by the conditions of test, previously dried to constant mass and weighed.

5.3 Drying oven, capable of maintaining the air temperature at $105\text{ °C} \pm 2\text{ °C}$, and suitably ventilated.

5.4 Desiccator.

6 Sampling

If the analysis is being done to evaluate a lot, obtain a representative sample of paper, board, or pulp as described in ISO 186 for paper and board or ISO 7213 for pulps delivered in bales or rolls. For paper and board for recycling, sampling shall be in accordance with EN 17085.

If the tests are made on another type of sample, make sure the test pieces taken are representative of the sample received.

In all cases take special precautions to avoid any change in moisture or water content of the material that will be tested.

7 Preparation of test pieces

7.1 General

Protect the test pieces from evaporation. Do not use bare hands to handle the test pieces. Handle the test pieces and weighing containers with clean and dry plastic or rubber gloves or adapted tools (for example tweezers). For determination of the dry matter content of pulp, paper, board or cellulosic nanomaterial samples as received, place each test piece as soon as obtained in a water-vapour proof container and close it immediately as cellulosic materials and especially cellulosic nanomaterials are highly hygroscopic.

Prepare at least duplicate test pieces for each sample.

7.2 Paper and board for recycling

For paper and board for recycling, the test piece mass shall be in the range from 200 g (for a homogeneous mix of paper and board for recycling) up to 500 g (for a less homogeneous mix of paper and board for recycling), depending on the composition of the grade of paper and board for recycling.

7.3 Paper, board, pulp and cellulosic nanomaterial in solid form

From the paper, board, pulp or “film-form” cellulosic nanomaterial (e.g. “nanopaper”, handsheets, or free-standing films) sample, select test pieces of the required mass which are representative of the sample. The test piece mass depends on the grammage of the sample. The mass can be varied from 1 g to 2 g for low grammage samples (e.g. tissue papers) up to 50 g for high grammage samples, more than 200 g/m² (e.g. pulps or boards).

For freeze-dried flakes, spray-dried powder, dough- or pulp-like materials, select test pieces of the required mass which are representative of the sample. A minimum of 1 g shall be taken.

WARNING — The method specified in this document involves the use of nanomaterials. Care should be taken to ensure observation of the relevant precautions and guidelines for nanotechnology laboratory safety and best practices.

8 Procedure

8.1 General

The following procedures shall be applied to the prepared test pieces.

8.2 Cut, tear or divide test pieces into suitably sized pieces, taking into account the method of test for which the dry matter is to be determined. In handling the test pieces, special precautions shall be taken (test pieces shall be quickly cut and weighed) to avoid any change in dry matter content.

8.3 Carry out all readings according to the required resolution (5.1).

8.4 Place a test piece in a previously dried to constant mass and weighed container (5.2) and close the container. Weigh the test piece and container.

8.5 Calculate the test piece mass in the closed container, the test piece mass before drying.

8.6 Open the container and place it with its lid and the test piece in the drying oven (5.3). Heat at $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ for the initial drying period.

8.7 The initial drying period for sheet form test pieces shall be ≥ 30 min for material of grammage $\leq 200\text{ g/m}^2$ and ≥ 60 min for grammage $> 200\text{ g/m}^2$. For paper and board for recycling, the initial drying period shall be 4 h. For other cellulosic nanomaterials in solid form, the initial drying period shall be ≥ 30 min. The initial drying period, even for highly moist samples, should not be more than 48 h.

8.8 After drying, fit the lid on to the container and allow it and the test piece to cool in the desiccator (5.4).

8.9 After cooling, equalize the air pressures outside and inside the container by quickly half-opening and reclosing the lid.

8.10 Weigh the closed container with the test piece.

8.11 Calculate the test piece mass in the closed container.

8.12 Repeat steps 8.5 to 8.10 with the drying period being at least half the initial drying time, until the test piece is considered to have reached constant mass when the difference between two successive weighings does not exceed 0,1 % of the test piece mass before drying. Until constant mass is reached, no new test pieces shall be placed in the oven. The constant mass is the test piece mass after drying.

8.13 Repeat steps 8.2 to 8.11 to carry out two determinations or as many as are stated in the method of test for which the dry matter content is to be determined.

8.14 The results of the parallel determinations of dry matter content should not deviate by more than 0,5 % mass fraction from their mean. Otherwise, it is recommended to use test pieces of a larger mass or a balance of better accuracy, and repeat steps 8.2 to 8.12.

NOTE This document is a reference method for determination of dry matter content based on oven dryness at 105 °C. Other drying techniques (e.g. microwave and infrared drying) are available on the market, but results from those techniques do not accord with this document.

9 Calculation and expression of results

Calculate the dry matter content, w_{dm} , expressed as a percentage mass fraction, according to [Formula \(1\)](#):

$$w_{\text{dm}} = \frac{m_1}{m_0} \times 100 \quad (1)$$

where

m_0 is the mass, in grams, of the test piece before drying;

m_1 is the mass, in grams, of the test piece after drying to constant mass.

Calculate the mean dry matter content of the determinations, $\bar{w}_{\text{dm}}$, and express the result to the first decimal place.

Moisture content can be calculated as described in [Annex A](#).

10 Precision

The repeatability and reproducibility of this method were determined by conducting an interlaboratory comparison study with several types of samples. A description of the samples used in this study and the interlaboratory comparison results are presented in [Annex B](#).

11 Test report

The test report shall contain at least the following information:

- a) a reference to this document, i.e. ISO 638-1:2021;
- b) the date and place of testing;
- c) all information for the complete identification of the sample, including its grade if the sample is a paper or board for recycling (according to EN643^[5] for example);
- d) the dry matter content, expressed as a percentage mass fraction;
- e) any unusual features observed during the test;
- f) any deviations from this document which may have influenced the test result.

Annex A (informative)

Calculation of the moisture content

Calculate the moisture content, $w_{\text{H}_2\text{O}}$, expressed as a percentage mass fraction, according to [Formula \(A.1\)](#):

$$w_{\text{H}_2\text{O}} = \frac{m_0 - m_1}{m_0} \times 100 \quad (\text{A.1})$$

where

m_0 is the mass, in grams, of the test piece before drying;

m_1 is the mass, in grams, of the test piece after drying to constant mass.

Calculate the mean moisture content of the determinations and express the result to the first decimal place.

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Annex B (informative)

Precision

B.1 General

The calculations were made according to ISO/TR 24498^[6].

The repeatability standard deviation reported is the “pooled” repeatability standard deviation, that is, the standard deviation is calculated as the root-mean-square of the standard deviations of the participating laboratories. This differs from the conventional definition of repeatability in ISO 5725-1^[7].

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 may 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 $2,77 = 1,96\sqrt{2}$.

B.2 Pulp, paper and board

Results reported in [Tables B.1](#) and [B.2](#) are issued from interlaboratory tests performed for the previous version of this document. Different kinds of pulp, paper and board samples were tested in 11 different laboratories according to this document.

Results reported in [Tables B.3](#) and [B.4](#) represent additional results for pulp samples. They are issued from interlaboratory tests performed in the first quarter of 2020 by 5 different laboratories according to the procedure specified in this document.

Table B.1 — Repeatability for determination of dry matter content in pulps, papers and boards

Sample	Mean value %	Coefficient of variation $C_{V,R}$ %
Bleached kraft pulp	94,6	0,08
Unbleached kraft pulp	94,5	0,14
Copy paper	96,1	0,04
Uncoated paper	95,9	0,10
Carton board 1	94,0	0,08
Carton board 2	93,8	0,15

Table B.2 — Reproducibility for determination of dry matter content in pulps, papers and boards

Sample	Mean value %	Coefficient of variation $C_{V,R}$ %
Bleached kraft pulp	94,6	0,27
Unbleached kraft pulp	94,5	0,32
Copy paper	96,1	0,23

Table B.2 (continued)

Sample	Mean value %	Coefficient of variation $C_{V,R}$ %
Uncoated paper	95,9	0,38
Carton board 1	94,0	0,36
Carton board 2	93,8	0,34

Table B.3 — Repeatability for determination of dry matter content in pulps

Sample	Mean value %	Number of laboratories	Repeatability standard deviation s_r %	Repeatability limit r %	Coefficient of variation $C_{V,R}$ %
Pulp 01 (Softwood)	35,6	4 ^a	0,08	0,21	0,21
Pulp 02 (Hardwood)	28,5	4 ^a	0,08	0,21	0,27

^a : One outlier for repeatability value exceeding critical values.

Table B.4 — Reproducibility for determination of dry matter content in pulps

Sample	Mean value %	Number of laboratories	Reproducibility standard deviation s_R %	Reproducibility limit R %	Coefficient of variation $C_{V,R}$ %
Pulp 01 (Softwood)	35,6	4 ^a	0,14	0,40	0,40
Pulp 02 (Hardwood)	28,5	4 ^a	0,22	0,62	0,78

^a : One outlier for repeatability value exceeding critical values.

B.3 Paper and Board for recycling

Results reported in [Tables B.5](#) and [B.6](#) deal with paper and board for recycling and are issued from interlaboratory tests performed in September 2019 by 9 different laboratories according to the procedure specified in this document.

Table B.5 — Repeatability for determination of dry matter content in paper and board for recycling

Sample	Mean value %	Number of laboratories	Repeatability standard deviation s_r %	Repeatability limit r %	Coefficient of variation $C_{V,R}$ %
Corrugated board for recycling	86,4	8 ^a	0,30	0,80	0,35
Magazines for recycling	95,1	8 ^a	0,10	0,30	0,11

^a : One outlier for repeatability value exceeding critical values.