INTERNATIONAL STANDARD

ISO 638-2

First edition
2021-05

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

Part 2: Suspensions of cellulosic nanomaterials

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

Partie 2: Suspensions de nanomatériaux cellulosiques

Reference number
ISO 638-2:2021(E)

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Published in Switzerland

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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 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 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 638-2, together with ISO 638-1, 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 water 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 water content of cellulosic nanomaterial suspensions.

ISO 638-1[1] is dedicated to the determination of the dry matter content or moisture content in paper, board, pulp and cellulosic nanomaterials in solid form, which all may be produced from virgin and/or recycled materials.

ISO 287[2] is 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] is used in laboratory procedures or is referred to in other International Standards in which the stock concentration of an aqueous pulp suspension requires determination.

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

Part 2: Suspensions of cellulosic nanomaterials

1 Scope

This document specifies an oven-drying method for the determination of the dry matter content in suspensions of cellulosic nanomaterials. The procedure is applicable to cellulosic nanomaterial suspensions which do not contain any appreciable quantities of materials other than water that are volatile at the temperature of 105 °C ± 2 °C. It is used, for example, in the case of cellulosic nanomaterial suspensions samples taken for chemical and physical tests in the laboratory, when a concurrent determination of dry matter content is required.

NOTE 1 ISO 638-1[1] specifies 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; ISO 287[2] specifies the determination of moisture content of a lot of paper and board; ISO 4119[3] specifies the determination of stock concentration of aqueous pulp suspensions; ISO 801 (all parts)[4] specifies the determination of 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.

2 Normative references

There are no normative references in this document.

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), or a material having internal structure or surface structure in the nanoscale, with the internal structure or surface structure composed predominantly of cellulose

Note 1 to entry: The terms nanocellulose (NC) and cellulose nanomaterial (CNM) are alternative terms for 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
<container> mass reached by a container after drying until the difference between two successive dryings and weighings does not exceed a specified mass fraction of the test specimen after drying

3.3
constant mass
<test specimen> mass reached by a *test specimen* (3.7) 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 a specified mass fraction of the test specimen after drying

3.4
dry matter content
ratio of the mass of a *test specimen* (3.7), after drying to *constant mass* (3.3) at a temperature of 105 °C ± 2 °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.

3.5
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
suspension
heterogeneous mixture of materials comprising a liquid and a finely dispersed solid material

Note 1 to entry: As the concentration of cellulosic nanomaterial increases, the suspension becomes more viscous from liquid to gel.

Note 2 to entry: In the case of cellulosic nanomaterials, a material of a mass fraction of 1 % to 5 % concentration is generally in gel form depending on the type of cellulosic nanomaterial. Above these concentrations, the material can be in solid form such as powder.

[SOURCE: ISO/TS 80004-6:2013, 2.13, modified — Notes 1 and 2 added.]

3.7
test specimen
portion of the sample on which the test is conducted

4 Principle

Test specimens taken from cellulosic nanomaterial samples in suspension form are weighed before and after drying to constant mass.

The dry matter content is calculated from the mass of the test specimen before and after drying.

5 Apparatus

5.1 Balance, which can be read to the nearest 0,1 mg.

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.

5.3 Drying oven, capable of maintaining the air temperature at 105 °C ± 2 °C, and suitably ventilated.

5.4 Desiccator.

6 Sampling

Care shall be taken that the procedure is appropriate for the material being sampled and to ensure that the test specimens are representative of the sample available. Filtration to concentrate dilute samples prior to sampling is not recommended as it can result in loss of cellulosic material. The sampling method used shall be reported according to c) in Clause 11 Test report.

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

7 Preparation of test specimens

Protect the test specimens from evaporation. Do not use bare hands to handle the test specimens. Handle the test specimens and weighing containers with clean, dry, rubber or polyethylene gloves or adapted tools (e.g. tweezers). For determination of dry matter content of cellulosic nanomaterial samples as received, place each test specimen as soon as obtained in a tared container and close it immediately.

Prepare at least duplicate test specimens for each sample.

Use enough test specimen to ensure that a minimum of 20 mg of solid material will remain after drying. For example, the requirement leads to a test specimen mass more than 4 g for a 0,5 wt% sample, 2 g for a 1 wt% sample, 1 g for a 2 wt% sample and 0,4 g for a 5 wt% sample.

For dilute samples, it may be necessary to use several test portions of the sample which are then dried consecutively with the previously dried test portion(s) in the same container, in order to obtain a total residue of at least 20 mg. They can be placed in large diameter containers to speed drying.

When necessary to avoid prolonged heating of heat-sensitive samples which can decompose (e.g. acidic cellulose nanocrystals) or large volumes of suspension, the test specimen can be freeze-dried prior to oven-drying. Place the container with the test specimen in an appropriate freezer until the test specimen is completely frozen, then place the container/test specimen in a lyophilizer according to the manufacturer's instructions until the test specimen is completely dry. Remove from the lyophilizer and proceed to the next step.

8 Procedure

The following steps shall be applied to the prepared test specimens.

8.1 Carry out all weighings according to the balance requirement (5.1).

8.2 Repeatedly dry a container (5.2) in an oven (5.3) at 105 °C ± 2 °C and cool down in a desiccator (5.4) so that the difference between two consecutive dryings and weighings of the container does not exceed 1 % of the mass of test specimen after drying that is to be obtained in 8.13. Let the constant mass be the container mass.

NOTE 1 When the nominal value of dry matter content of the sample is given, it can be convenient to use the nominal value to estimate the required constant mass variation of the container for subsequent analyses.

NOTE 2 It can be convenient to use a quickly determined dry matter content to estimate the required constant mass variation of the container for subsequent analyses.

8.3 After placing a test specimen of appropriate mass in the container, weigh the closed container with the test specimen.

8.4 Calculate the test specimen mass in the closed container and let it be the test specimen mass before drying.

8.5 When necessary, pre-treat the container with the test specimen, e.g. freeze-drying as described in Clause 7. Open the container and place it with the test specimen and its lid in the drying oven.

8.6 Heat the container and test specimen to dryness at 105 °C ± 2 °C. Dryness can be verified visually when establishing an appropriate initial drying period. For aqueous test specimens, the initial drying time will vary with the volume to be evaporated.

8.7 After drying, fit the lid on to the container and allow the test specimen to cool in the desiccator (5.4).

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

8.9 Weigh the closed container with the test specimen.

8.10 Calculate the test specimen mass in the closed container.

8.11 The test specimen is considered to have reached constant mass when the difference between two successive weighings after drying at 105 °C ± 2 °C does not exceed 1 % of the test specimen mass after this period of drying.

8.12 The drying period between two successive weighings shall be at least one-half of the minimum initial drying time. During these periods do not put any new test specimens in the oven. The initial drying period, even for highly moist samples, should not be more than 16 h.

Care shall be taken to ensure that heat-sensitive test specimens do not char (blacken) during drying. Such test specimens should not be heated for longer than necessary following the initial weighing after "visual" dryness is achieved. Freeze-drying as described in Clause 7 may also be used to avoid any decomposition by prolonged heating.

8.13 Repeat 8.5 to 8.12 until constant mass is reached. When constant mass is reached, let it be the test specimen mass after drying.

8.14 If the mass variation of the container obtained in 8.2 is larger than 1 % of the test specimen mass after drying, use a smaller container and/or a larger test specimen, and repeat 8.2 to 8.13.

8.15 Repeat 8.2 to 8.14 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.

8.16 The results of the parallel determinations of dry matter content should not deviate from their mean by more than 2 % of the mean value. Otherwise, it is recommended to review the whole procedure to avoid unknown uncertainties, to use test specimens of a larger mass or a balance of better accuracy and to repeat 8.2 to 8.15.