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Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 900 °C

Papiers, cartons, pâtes et nanomatériaux à base de cellulose — Détermination du résidu (cendres) après incinération à 900 °C

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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 www.iso.org/iso/foreword.html.

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

This sixth edition cancels and replaces the fifth edition (ISO 2144:2015), which has been technically revised. The main changes compared to the previous edition are as follows:

— The scope has been changed to cover also cellulose nanomaterials instead of only paper, board and pulps;

— A definition of cellulose nanomaterial, along with additional instructions for sampling, sample preparation, and incineration for cellulose nanomaterials have been incorporated;

— Additional instructions are given on how to express results when a sample has low ash content.

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

The magnitude of the residue (ash content) on ignition at a given temperature is related to, but not equal to, the content of mineral constituents in the sample. For coated and filled products, the amount of added mineral constituents can only be calculated from the result if the loss on ignition of the particular pigment used is known. For China clay, the residue on ignition at 900 °C varies from 89 % to 86 % and for calcium carbonate it is about 56 %.

The determination is mainly used as a screening test for checking the overall quality of a product, in many cases against specifications. The ignition procedure described can be used as a preliminary step when determining particular mineral constituents.

Determination of residue (ash content) on ignition at 525 °C of paper, board, pulps and cellulose nanomaterials is described in ISO 1762[1].

Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 900 °C

1 Scope

This document describes the determination of the residue (ash content) on ignition of paper, board, pulps and cellulose nanomaterials. This document is applicable to all types of paper, board, pulp and cellulose nanomaterial. This document provides measurement procedures to obtain a measurement precision of 0,01 % or better for residue (ash content) on ignition at 900 °C.

In the context of this document, the term "cellulose nanomaterial" refers specifically to cellulose nano-object (see 3.2 to 3.4). Owing to their nanoscale dimensions, these cellulose nano-objects can have intrinsic properties, behaviours or functionalities that are distinct from those associated with paper, board and pulps.

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 287, *Paper and board — Determination of moisture content of a lot — Oven-drying method*

ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

ISO 7213, *Pulps — Sampling for testing*

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
residue on ignition
ash content
ratio of the mass of the residue remaining after a test specimen of paper, board, pulp or *cellulose nanomaterial* (3.2) is ignited at 900 °C ± 25 °C to the oven-dry mass of the test specimen before ignition

Note 1 to entry: This property has been referred to as either "residue on ignition" or "ash content" in earlier editions of this document.

3.2
cellulose nanomaterial
material composed predominantly of cellulose, with any external dimension between approximately 1 nm and 100 nm, 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 and cellulosic nanomaterial are synonymous with cellulose nanomaterial.

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

Note 3 to entry: This generic term is inclusive of cellulose nano-object and cellulose nanostructured material.

Note 4 to entry: See also definitions of cellulose, nanoscale, cellulose nano-object and cellulose nanostructured material in ISO/TS 20477:2017.

[SOURCE: ISO/TS 20477:2017, 3.3.1, modified — "1 nm to 100 nm" changed to "1 nm and 100 nm"; abbreviations deleted from Note 1 to entry; Note 4 to entry added.]

3.3
nano-object
discrete piece of material with one, two or three external dimensions in the nanoscale

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: ISO/TS 80004-1:2015, 2.5]

3.4
cellulose nano-object
nano-object composed predominantly of cellulose

[SOURCE: ISO/TS 20477:2017, 5.2]

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 predominantly exhibited in this length range.

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

4 Principle

The test specimen is weighed in a heat-resistant crucible and incinerated at 900 °C ± 25 °C in a muffle furnace. The moisture or dry matter content of a separate test specimen is also measured. The percentage ash is then determined, on a dry (moisture-free) basis, from the mass of residue (ash) after ignition and the moisture or dry matter content of the sample.

Cellulosic and organic materials as well as carbonate in the sample are completely lost by ignition at 900 °C. For coated and filled products, the amount of added mineral constituents can only be calculated from the result if the loss on ignition of the particular pigment used is known. This value varies from one pigment to another and also between different batches of many pigments. If higher ignition temperatures are used, the corresponding loss on ignition will increase, but there is no guarantee that it will become exactly 100 % at any temperature. For pulps and other materials without any added minerals, the ash content is a measure of the amount of unwanted mineral constituents such as silica, silicates or particles of minerals. Some soluble inorganic constituents such as sodium chloride will escape the determination, whereas sulfates will normally be retained.

5 Apparatus

Ordinary laboratory equipment, including the following:

5.1 Crucibles of platinum, ceramics or silica, of capacity to accommodate about 10 g of sample (normally a capacity of 50 ml is sufficient).

The crucibles shall not lose or gain mass on ignition or react chemically with the sample or its ignition residue.

Larger-capacity crucibles may also be used for low-density materials to accommodate sufficient sample.

A lid of an appropriate material, placed slightly ajar to allow air for combustion, may also be used with the crucible to help prevent low density or flyaway material from escaping during the ash ignition process.

Platinum crucibles are recommended if a small amount of residue is expected.

5.2 Muffle furnace, capable of maintaining a temperature of 900 °C ± 25 °C.

It is recommended that the furnace be placed in a hood or that means are provided for evacuating smoke and fumes.

5.3 Analytical balance, with a scale division (readability) of 0,1 mg or better in order to obtain a measurement precision of 0,01 % or better.

5.4 Desiccator.

6 Sampling and preparation of test specimen

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.

6.1 Sample amount

Sufficient material shall be collected to allow for at least duplicate determinations and for determination of moisture or dry matter content.

6.2 Paper, board and pulp sampling

Guidance on obtaining representative samples found in ISO 186 for paper and board and in ISO 7213 for pulps delivered in bales or rolls shall be followed in cases where the analysis is being conducted to evaluate a lot. In this case or if the tests are made on another type of sample, take test specimens from various parts of the sample making sure they are thoroughly representative of the sample.

The test specimen taken for incineration shall consist of a number of small pieces no larger than 1 cm^2. In a similar manner, obtain a moisture content or dry matter content specimen from the sample.

The specimen for incineration shall have a total mass of not less than 1 g on an oven-dry basis, and sufficient to give a residue on ignition of not less than 10 mg.

If the material has a very low residue on ignition (for example, in the case of so-called ashless grades), take a test specimen of sufficient mass to yield at least 2 mg of residue. In these cases, it might be necessary to divide the test specimen into two or several smaller portions which are incinerated consecutively in the same crucible, in order to obtain a total residue of at least 10 mg.