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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.

6.3 Cellulose nanomaterial sampling

For cellulose nanomaterials, care should be taken that the procedure is appropriate for the material being sampled. There is no standard procedure for sampling cellulose nanocrystals, either in aqueous suspensions or dried forms, or for sampling dilute (wet) cellulose nanofibrils. When the original cellulose nanomaterial sample is in a wet form or a free aqueous suspension (dilute or concentrated), it shall be dried using an appropriate method such as heating, freeze-drying, or spray-drying, to give the sample a solid form such as flakes, powder or other solid, which shall be mixed to homogeneity. The test specimen shall be obtained from this pre-dried sample. In a similar manner, obtain a moisture content or dry matter content specimen from the sample. Filtration to concentrate dilute samples prior to drying is not recommended as it may result in loss of dissolved material which gives ash upon heating to 900 °C.

Chemically modified pulps are often prepared through TEMPO-mediated oxidation, carboxymethylation or phosphorylation prior to the production of cellulose nanofibrils (CNF). Chemically modified pulps, CNF made from chemically modified pulps, as well as cellulose nanocrystals (CNC) extracted from pulps by sulfuric acid hydrolysis or oxidative procedures, may contain various cations associated with the ionic carboxylate or phosphate groups introduced at the surface during production. When these pulps or cellulose nanomaterials are in the acidic form (i.e. contain only protons as the cations), they will have very low ash content. The ash content will be greater in pulps and cellulose nanomaterials containing metal cations, such as sodium or calcium, or organic cations such as alkyl ammonium cations.

NOTE TEMPO stands for 2,2,6,6-tetramethylpiperidine-1-oxyl.

The mass of the test specimen shall be at least 1 g on a dry basis. For cellulose nanomaterials — particularly those in the form of a dilute suspension, and/or those of very low ash content such as the acidic forms of cellulose nanomaterials — it is often not feasible to collect sufficient test specimen to yield at least 10 mg of ash on ignition. A residue less than 10 mg is still acceptable and in conformity with the requirements of this document.

NOTE Less than 10 mg ash could lead to reduced precision.

If the material is very low-density and/or flyaway (e.g. freeze-dried cellulose nanocrystals), it may be compacted (e.g. by compressing it manually in the crucible or using equipment which will not impart extraneous mineral content to the sample) in a manner to increase the bulk density such that sufficient material will fit in the crucibles used. However, this reduces the speed of incineration.

7 Procedure

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.

7.1 General

Carry out the procedure in at least duplicate. Allow wet test specimens and moisture content, or dry matter content specimens to air-dry, and condition other test specimens under dust-free conditions in the ambient laboratory air until they reach equilibrium moisture.

7.2 Measurement of moisture or dry matter content

Determine the moisture or dry matter content on the relevant test specimen (air-dry) using the procedure described in ISO 287 or ISO 638, as relevant. Do not use the moisture or dry matter content test specimen for incineration. Weigh the crucible containing the moisture or dry matter content test specimen at the same time as the crucible containing the test specimen (air-dry) used for incineration (see 7.3).

7.3 Incineration

Heat the crucible (5.1) without any sample for 30 min to 60 min in the muffle furnace (5.2) at 900 °C ± 25 °C. Allow it to cool to room temperature in a desiccator (5.4). Ash samples (particularly those containing CaO) can absorb moisture from typical $CaSO_4$ desiccant; more efficient desiccants such as molecular sieves are recommended.

Weigh the empty crucible to the nearest 0,1 mg. Add the test specimen and weigh again immediately to the nearest 0,1 mg. To prevent low-density materials such as freeze-dried cellulose nanocrystals from becoming flyaway during ignition, they may be compacted (the bulk density increased) as described in 6.3.

Place the crucible containing the test specimen in the furnace at room temperature and heat the crucible slowly, preferably in such a manner that the sample burns but does not burst into flames. Check that no material is lost in the form of flying particles.

NOTE The procedure for this step depends on the equipment available. Some muffle furnaces have a door that, when open, forms a horizontal platform in front of the entrance. This platform and similar devices can be used when burning off the organic material in the sample.

When the combustion is complete or nearly complete, so that only small amounts of carbon are visible, expose the crucible to the full heat (900 °C ± 25 °C) of the furnace for 1 h. Do not prolong the heating period and do not attempt to reach “constant mass”. Some constituents can lose mass slowly over a long period of time.

7.4 Measurement of residue (ash) mass

Remove the crucible from the furnace and allow it to attain room temperature in a desiccator (5.4). Weigh the crucible and contents as before to the nearest 0,1 mg.

8 Expression of results

For each crucible, calculate the ash content using Formula (1).

$$X = \frac{100 m_r}{m_s} \tag{1}$$

where

X is the ash content, as a percentage of the mass of the test specimen on an oven-dry basis;

m_r is the mass of the residue (the mass of the crucible with the residue, minus that of the empty crucible) in grams (g);

m_s is the mass of the test specimen, on an oven-dry basis, in grams (g). This is determined from the average of the replicate moisture content or dry matter content determinations.

Check that there is a reasonable agreement between the replicate measurements. For test specimens with ash contents above 0,1 %, reasonable agreement exists if the deviation of results from the mean of parallel determinations does not exceed 10 % of the mean value. If this is not the case, repeat the entire procedure with new test specimens, preferably of larger mass.

Calculate the mean ash content. Express the mean to the nearest 0,1 % for samples with ash content above 1 % and to the nearest 0,01 % or smaller for samples with ash content below 1 %. If the mean ash content is below 0,1 %, it may be reported as "below 0,1 %", or the individual results of replicate determinations may be reported.

NOTE Precision values (repeatability and reproducibility) are given in Annex A.

9 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 2144:2019;

b) date and place of testing;

c) complete identification of the samples tested;

d) the result, expressed as indicated in Clause 8;

e) any departure from the procedure described in this document or any other circumstances which could have affected the result.

Annex A
(informative)

Precision

A.1 General — Pulp and paper

In an interlaboratory study conducted by the Collaborative Testing Services, Inc. (CTS), 12 laboratories determined the ash content on ignition of different pulp and paper samples as instructed in this document. Six samples were analysed in duplicate by each of the laboratories. After having rejected a few obvious outliers, the data in Table A.1 and Table A.2 were calculated. All data are expressed as percentage residue.

The calculations have been made according to ISO/TR 24498[2].

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 might not be valid for different materials or different test conditions.

NOTE Repeatability and reproducibility limits were calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where 2,77 = 1,96 $\sqrt{2}$.

A.2 Repeatability — Pulp and paper

Table A.1 — Repeatability for determination of ash content of pulp and paper

Sample	Mean value %	Standard deviation S_r (%)	Coefficient of variation $C_{V,r}$ (%)	Repeatability limit r (%)
Kraft softwood pulp	0,1	0,01	10	0,028
Kraft hardwood pulp	0,5	<0,01	2,0	0,028
Uncoated paper with $CaCO_3$	8,3	0,08	0,96	0,22
Uncoated paper without $CaCO_3$	8,8	0,05	0,57	0,14
Coated paper with $CaCO_3$	21,2	0,15	0,71	0,42
Coated paper without $CaCO_3$	28,2	0,15	0,53	0,42

A.3 Reproducibility — Pulp and paper

Table A.2 — Reproducibility for determination of ash content of pulp and paper

Sample	Mean value %	Standard deviation S_R (%)	Coefficient of variation $C_{V,R}$ (%)	Reproducibility limit R (%)
Kraft softwood pulp[a]	0,1	0,07	70	0,19
Kraft hardwood pulp	0,5	0,11	22	0,30
Uncoated paper with $CaCO_3$	8,3	0,46	5,5	1,3
Uncoated paper without $CaCO_3$	8,8	0,10	1,1	0,23

[a] The value for the Kraft softwood pulp (0,1 %) is below the scope (lower limit) of this document (0,2 %).

Table A.2 *(continued)*

Sample	Mean value %	Standard deviation S_R (%)	Coefficient of variation $C_{V,R}$ (%)	Reproducibility limit R (%)
Coated paper with $CaCO_3$	21,2	0,26	1,2	0,72
Coated paper without $CaCO_3$	28,2	0,24	0,85	0,66
[a] The value for the Kraft softwood pulp (0,1 %) is below the scope (lower limit) of this document (0,2 %).				

A.4 General — Cellulose nanomaterials

The precision data are based on typical cellulose nanocrystal and cellulose nanofibril samples.

A total of 16 laboratories participated in an interlaboratory study to determine the ash content as specified in this document. Four samples were analysed in triplicate by each of the laboratories. The data from one or two laboratories, depending on the sample, were considered as outliers and excluded from the study, as indicated. The precision data are shown in Tables A.3 and A.4.

The calculations have been made according to ISO/TR 24498[2].

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 might not be valid for different materials or different test conditions.

NOTE 1 Repeatability and reproducibility limits were calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where 2,77 = 1,96 √2.

NOTE 2 For sample CNF 1, three of the laboratories measured significantly higher ash content than the rest, possibly due to incomplete ashing.

A.5 Repeatability — Cellulose nanomaterials

Table A.3 — Repeatability for determination of ash content of cellulose nanomaterials

Sample	Number of laboratories	Mean value %	Standard deviation S_r (%)	Coefficient of variation $C_{V,r}$ (%)	Repeatability limit r (%)
CNC 1[a,b]	15	1,7	0,12	7,1	0,34
CNC 2[a,b]	14	1,8	0,033	1,8	0,091
CNF 1	16	0,17	0,016	9,4	0,045
CNF 2	14	0,80	0,023	2,9	0,064
[a] These two samples are from the same batch, but prepared separately.					
[b] In the case of one laboratory for each of these two samples, the k statistic — a measure of the consistency of the data — was higher than the critical k value. However, the data were included in the calculations since the mean value was well within the range of values for the other laboratories.					