BS ISO 2144:2015

BSI Standards Publication

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

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National foreword

This British Standard is the UK implementation of ISO 2144:2015. It supersedes BS ISO 2144:1997 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PAI/11, Methods of test for paper, board and pulps.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

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

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Foreword

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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 WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 6, *Paper, board and pulps*.

This fifth edition cancels and replaces the fourth edition (ISO 2144:1997), which has been technically revised.

Introduction

The magnitude of the residue on ignition 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. This value varies from one pigment to another and also between different batches of the same pigment. For China clay, the residue on ignition at 900 °C varies from 89 % to 86 % and for calcium carbonate, it is about 56 %. If lower ignition temperatures are used, the corresponding figures will increase, but there is no guarantee that they will become exactly 100 % at any temperature.

For pulps and other materials without any added minerals, the residue on ignition is a measure of the amount of unwanted mineral constituents such as silica, silicates, particles of minerals, etc. Some soluble inorganic constituents such as sodium chloride will escape the determination, whereas sulfates will normally be retained.

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.

NOTE Determination of residue on ignition at 525 °C of pulps is described in ISO 1762[1].

INTERNATIONAL STANDARD ISO 2144:2015(E)

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

1 Scope

This International Standard describes the determination of the residue on ignition of pulps, papers, and boards. The International Standard is applicable to all types of pulp, paper, and board. The lower limit of the determination is about 0,2 %.

NOTE The procedure (Clause 7) requires that at least 10 mg of residue is weighed. The limit stated above corresponds to a 5 g sample. If the sample size is increased, this limit can be lowered.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.

3.1
residue on ignition
mass of the residue left after incineration of a test specimen of pulp, paper, and board in a furnace at 900 °C ± 25 °C by the procedure specified in this International Standard

Note 1 to entry: This property has been referred to as “ash content”, for example, in earlier editions of this International Standard.

4 Principle

The test specimen is weighed in a heat-resistant dish and incinerated at 900 °C ± 25 °C in a muffle furnace. The mass of the residue is determined by weighing the dish after the incineration of the test specimen.

5 Apparatus

Ordinary laboratory equipment, including the following:

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

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

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

The furnace is preferably placed in a hood or it is provided with means for evacuating smoke and fumes.

5.3 Analytical balance, accurate to 0,1 mg.

6 Sampling and preparation of test specimen

If possible, take the test specimen as described in ISO 186 or ISO 7213 as relevant.

7 Procedure

Carry out the procedure in duplicate. Record all weighings to the nearest 0,1 mg. Allow wet samples to dry under dust-free conditions in the laboratory air.

Determine the moisture content on a separate test specimen (air-dry) by the procedure described in ISO 287 or ISO 638 as relevant. Weigh this test specimen at the same time as the test specimen (air-dry) used for incineration.

The portions to be incinerated shall consist of a number of small pieces, of size no larger than 1 cm^2, of a total mass of not less than 1 g, or sufficient to give a residue on ignition of not less than 10 mg taken from various parts of the sample in such a manner as to be thoroughly representative of it.

If the sample has a very low residue on ignition (for example, in the case of so-called ash-less qualities), take a test specimen portion of sufficient mass to yield at least 2 mg of residue. In these cases, it could be necessary to divide the test specimen into two or several smaller portions which are incinerated consecutively in the same dish.

Heat the dish (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.

Weigh the empty dish. Add the appropriate amount of sample and weigh again immediately.

Heat the dish 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 1 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 dish to the full heat (900 °C ± 25 °C) of the furnace for 1 h.

NOTE 2 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.

Remove the dish from the furnace and allow it to attain room temperature in a desiccator. Weigh the dish as before.

8 Expression of results

For each dish, calculate the residue on ignition from the expression

$$X = \frac{100\,a}{m} \tag{1}$$

where

X is the residue on ignition, as a percentage on oven-dry basis;

a is the mass of the residue (the mass of the dish with the residue minus that of the empty dish) in grams;

m is the mass of the test specimen, on oven-dry basis, in grams

Check that there is a reasonable agreement between the two duplicates and report the mean to the nearest 0,1 % for samples with residue on ignition above 1 % and to the nearest 0,01 % for samples with residue below 1 %.

NOTE Normally, a reasonable agreement is considered to exist if the range of results from parallel determinations is less than 5 % of the mean.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard, i.e. ISO 2144:2015;

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 International Standard or any other circumstances which could have affected the result.

Annex A
(informative)

Precision

A.1 General

In an interlaboratory study conducted by the Collaborative Testing Services, Inc. (CTS), 12 laboratories determined the residue on ignition (ash) as instructed in this International Standard. 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 are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where 2,77 = 1,96 √2.

A.2 Repeatability

Table A.1

Sample	Mean value %	Standard deviation S_r (%)	Coefficient of variation CoV_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 C_aCO_3	8,3	0,08	0,96	0,22
Uncoated paper without C_aCO_3	8,8	0,05	0,57	0,14
Coated paper without C_aCO_3	21,2	0,15	0,71	0,42
Coated paper with C_aCO_3	28,2	0,15	0,53	0,42

A.3 Reproducibility

Table A.2

Sample	Mean value %	Standard deviation S_R (%)	Coefficient of variation CoV_R (%)	Repeatability 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 C_aCO_3	8,3	0,46	5,5	1,3
Uncoated paper without C_aCO_3	8,8	0,10	1,1	0,23
Coated paper without C_aCO_3	21,2	0,26	1,2	0,72
Coated paper with C_aCO_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 International Standard (0,2%).

Bibliography

[1] ISO 1762, *Pulps — Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C*

[2] ISO/TR 24498, *Paper, board and pulps — Estimation of uncertainty for test methods*

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