BS ISO 1762:2015



BSI Standards Publication

Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C



BS ISO 1762:2015 BRITISH STANDARD

National foreword

This British Standard is the UK implementation of ISO 1762:2015. It supersedes BS ISO 1762:2001 which is withdrawn.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 525 °C

Papier, carton et pâtes — Détermination du résidu (cendres) après incinération à 525 °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).

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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 third edition cancels and replaces the second edition (ISO 1762:2001), which has been technically revised.

Paper, board and pulps — Determination of residue (ash) on ignition at 525 °C

1 Scope

This International Standard describes the determination of the residue (ash) on ignition of paper, board and pulp at 525 °C. It is applicable to all types of paper, board, and pulp samples. The ash may consist of

- a) mineral matter in the pulp and various residues from chemicals used in its manufacture,
- b) metallic matter from piping and machinery, and
- c) fillers, pigments, coatings, or residues from various additives.

In samples containing calcium carbonate, there is practically no decomposition of carbonate by ashing at 525 °C. Other fillers and pigments such as clay and titanium dioxide are also unaffected by ashing at 525 °C. Accordingly, the residue on ignition as determined by this International Standard provides a good estimate of the total inorganic matter in the sample, provided that the sample does not contain other minerals which decompose at or below this temperature. For example, magnesium carbonate and calcium sulfate may, at least partly, decompose at temperatures below 525 °C.

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 ash on ignition

mass of the residue remaining after a test specimen of paper, board, or pulp is ignited in a furnace at $525 \,^{\circ}\text{C} \pm 25 \,^{\circ}\text{C}$ as specified in this International Standard

4 Principle

A test specimen is weighed in a heat-resistant crucible and ignited in a muffle furnace at 525 °C \pm 25 °C. The moisture content of a separate test specimen is also measured. The percentage ash is then determined, on a moisture-free basis, from the weight of residue after ignition and the moisture content of the sample.

5 Apparatus

- **5.1 Heat-resistant crucibles**, made of platinum, porcelain, or silica, with a capacity of 50 ml to 100 ml.
- **5.2 Muffle furnace**, capable of maintaining a temperature of $525 \,^{\circ}\text{C} \pm 25 \,^{\circ}\text{C}$. It is recommended to place the furnace in a hood or to provide means for evacuating smoke and fumes.
- **5.3 Analytical balance**, accurate to 0,1 mg.
- 5.4 Desiccator.

6 Sampling and preparation of test specimen

If the analysis is being done to evaluate a lot, obtain a representative sample of paper, board, or pulp as described in ISO 186 or ISO 7213, as relevant. Sufficient sample must be collected to allow for at least duplicate determinations and for determination of moisture content. Take a test specimen consisting of small pieces, not larger than $1\ cm^2$, taken from various parts of the sample in such a manner as to be thoroughly representative of it. The total mass shall be at least 1 g moisture free and shall yield a weight of ash not less than 10 mg and preferably over 20 mg (See the Note in Clause 7). In a similar manner, obtain a moisture specimen from the sample.

7 Procedure

Carry out the procedure in duplicate.

Air-dry the test and moisture specimens in the laboratory atmosphere until they reach equilibrium moisture.

Determine the moisture content on the moisture specimen as described in ISO 287 or ISO 638, as relevant. Weigh this specimen at the same time as the specimen used for ash determination.

Heat the empty crucible (5.1) for 30 min to 60 min in the muffle furnace (5.2) at 525 °C ± 25 °C. Cool it to room temperature in a desiccator.

Weigh the empty crucible to the nearest 0,1 mg. Add the test specimen and immediately weigh again.

Place the crucible containing the test specimen in the furnace at room temperature and gradually raise the temperature to $525\,^{\circ}$ C (about $200\,^{\circ}$ C/h) in order to burn the sample without it bursting into flames, and to ensure that no material is lost in the form of flying particles.

Maintain the ignition temperature of 525 °C for at least 2 h in the case of pulp and board samples and at least 3 h for paper samples. The specimen shall be completely charred as indicated by the absence of black particles.

Remove the crucible from the furnace, and cool it to room temperature in a desiccator. Weigh the crucible and content to the nearest 0.1~mg.

NOTE If the sample has a very low residue on ignition (for example in the case of so-called ashless grades), it may be necessary to use several portions of the sample which are then ignited consecutively in the same crucible, in order to obtain a total residue of at least 10 mg.

8 Calculation

Calculate the percentage residue on ignition as shown in Formula (1):

$$X = \frac{100 \, m_{\rm r}}{m_{\rm s}} \tag{1}$$

where

- *X* is the residue on ignition, as a percentage of the mass of the oven-dry test specimen;
- $m_{\rm r}$ is the mass, in grams, of the residue (mass of crucible with residue, minus that of the empty crucible);
- $m_{\rm S}$ is the mass, in grams, of the test specimen, on an oven-dry basis. This is determined from the average of the duplicate moisture content determinations.

Report the mean of duplicate determinations 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 %.

9 Test report

The test report shall include the following information:

- a) a reference to this International Standard, i.e. ISO 1762:2015;
- b) date and place of testing;
- c) complete identification of the sample 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 may have affected the result.

Annex A (informative)

Precision

A.1 General

The precision data are based on typical pulp, paper, and paperboard samples.

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

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 $\sqrt{2}$.

A.2 Repeatability

In a study performed by one laboratory, the residue on ignition of a wide range of samples, including pulp, newsprint, uncoated paper, coated paper, and paperboard was determined as specified in this International Standard. Mean values and coefficients of variations for each type of sample are shown in <u>Table A.1</u>.

Coefficient of Repeatability Number of Mean value Standard deviation Sample variation limit determinationsa S_r (%) 0% CoV_r (%) r (%) Chemical and 6 0.71 0,010 1,4 0,028 mechanical pulp 3 Newsprint 3,50 0,010 0,29 0,028 Uncoated print-5 29,4 0,029 0,10 0,080 ing paper Coated printing 13 37.3 0.090 0.24 0.25 paper Paperboard 3.06 0.080 2.6 0.22 Samples from different batches were used for each type of material.

Table A.1 — Repeatability for determination of residue on ignition

A.3 Reproducibility

Five samples, representing different types of paper and paperboard, were analysed by 15 laboratories, as specified in this International Standard.

Mean values and coefficients of variations between laboratories are shown in Table A.2.

 ${\bf Table~A.2-Reproducibility~for~determination~of~residue~on~ignition}$

Sample	Mean value %	Standard deviation S_R (%)	Coefficient of variation $CoV_{R}\left(\%\right)$	Reproducibility limit R (%)		
Copy papera	9,33	0,18	1,95	0,50		
Coated paper 1a	32,0	0,77	2,41	2,13		
Coated paper 2a	25,6	0,51	1,99	1,41		
Paperboard 1	1,43	0,028	1,96	0,078		
Paperboard 2a	0,55	0,022	4,02	0,061		
^a For each of these four samples, the results are based on those of 14 laboratories.						

Bibliography

[1] TAPPI T211: Ash in wood, pulp, paper and paperboard: combustion at 525 $^{\circ}$ C



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