
**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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 1762 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This second edition cancels and replaces the first edition (ISO 1762:1974). In this revision of the 1974 edition, the ignition temperature has been changed from 575 °C to 525 °C to minimize the decomposition of calcium carbonate.

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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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 186:—¹⁾, *Paper and board — Sampling to determine average quality*

ISO 287:1985, *Paper and board — Determination of moisture content — Oven-drying method*

ISO 638:1978, *Pulps — Determination of dry matter content*

ISO 7213:1981, *Pulps — Sampling for testing*

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1

residue on ignition ash on ignition

mass of the residue remaining after a sample of paper, board or pulp is ignited in a furnace at 525 °C ± 25 °C as specified in this International Standard

1) To be published. (Revision of ISO 186:1994)

4 Principle

A test specimen is weighed in a heat-resistant crucible and ignited in a muffle furnace at $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. 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 $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. 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 sample

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

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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 $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$. 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 $525\text{ }^{\circ}\text{C}$ (about $200\text{ }^{\circ}\text{C/h}$) 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 $525\text{ }^{\circ}\text{C}$ 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 follows:

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

where

- X is the residue on ignition, as a percentage of the mass of the oven-dry sample;
- m_r is the mass, in grams, of the residue (mass of crucible with residue, minus that of the empty crucible);
- m_s is the mass, in grams, of the sample, 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 Precision

9.1 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 Table 1.

Table 1

Sample	Number of determination ^a	Mean value %	Coefficient of variation
Chemical and mechanical pulp	6	0,71	1,4
Newsprint	3	3,50	0,29
Uncoated printing paper	5	29,4	0,10
Coated printing paper	13	37,3	0,24
Paperboard	3	3,06	2,6

^a Different samples were used for each type of material.

9.2 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 2.

Table 2

Sample	Mean value %	Coefficient of variation between laboratories
Copy paper ^a	9,33	1,95
Coated paper 1 ^a	32,0	2,41
Coated paper 2 ^a	25,6	1,99
Paperboard 1	1,43	1,96
Paperboard 2 ^a	0,55	4,02

^a For each of these four samples, the results are based on those of 14 laboratories.

10 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- 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.

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