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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Paper and board — Determination of ash

Papiers et cartons — Détermination des cendres

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2144 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

This third edition cancels and replaces the second edition (ISO 2144 : 1983), subclause 5.3 and clause 8 of which have been revised to amend the temperature of ignition.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Paper and board — Determination of ash

1 Scope and field of application

This International Standard specifies a procedure for the determination of ash of paper and board.

For the purposes of this method, papers and boards can be divided as follows.

1.1 Ashless papers and boards (see term 6.4 in ISO 4046), where the raw materials used in their manufacture have been specially selected and treated to reduce the mineral content to a minimum, or the papers and boards themselves have been so treated.

1.2 Other papers and boards to which no loading has been added.

1.3 Papers and boards loaded during manufacture. The determination of ash, carried out as specified, will usually give all the information required. If further data are needed about the nature of the loading material used, a chemical examination of the ash itself is necessary. (See the annex.).

1.4 Papers and boards coated during or after manufacture. The base paper or board may contain loading, and an ash determination by this method will give a result representing the total ash from all the mineral matter in and on the paper or board. For most instances of routine examination the result so obtained will suffice, but occasions may arise when the ash from the coating and that from the loading in the paper or board require separate determination. In such instances it is necessary to ascertain the nature of the adhesive used in order that a suitable method may be employed to remove the coating from the base paper or board so that separate determinations can be carried out.

2 References

ISO 186, *Paper and board — Sampling to determine average quality.*

ISO 187, *Paper and board — Conditioning of samples.*

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

ISO 4046, *Paper, board, pulp and related terms — Vocabulary.*

3 Definition

ash: The amount of residue of a material left after incineration determined and expressed according to the procedure specified in this International Standard.

4 Principle

Ignition of a test portion placed in a crucible, in a muffle furnace, until combustion is complete and constant mass is attained.

5 Apparatus

5.1 Crucibles, complete with well-fitting lids, and made from materials such as platinum, fused alumina, porcelain or silica which do not change in mass under ignition conditions.

NOTE — As platinum reacts at high temperatures with barium carbonate and zinc compounds, the use of platinum crucibles is to be avoided when it is suspected that these materials are present in the sample.

5.2 Balance, accurate to 0,1 mg.

5.3 Electric muffle furnace capable of being controlled at 900 ± 25 °C. A gas-fired muffle or a gas burner may be used provided it is capable of giving a similar temperature.

5.4 Desiccator.

6 Sampling

Carry out the sampling in accordance with the procedure given in ISO 186.

7 Preparation of test piece

The test piece shall consist of a number of small portions, of total mass not less than 1 g, or sufficient to give an ash of not less than 10 mg, taken from various parts of the sample in such a manner as to be thoroughly representative of it.

In the case of ashless paper or board where the mineral content is extremely low, take sufficient paper or board to give an ash of not less than 2 mg.

It is not necessary to condition the test piece, unless the result is required on an air-dry basis, in which case carry out conditioning in accordance with ISO 187.

Since the result is normally required in terms of the moisture-free condition, however, it is recommended that a test for moisture content be carried out independently according to ISO 287, on a separate portion of the sample weighed out at the same time. The result shall be used to convert the ash content determined on the air-dry sample to the moisture-free (oven-dry) basis. Alternatively, the determination may be carried out on the oven-dried paper or board by drying the test piece to constant mass at $105 \pm 2^\circ\text{C}$ and weighing to the nearest 0,1 mg; this may be done with sufficient accuracy for the purpose in the ignited and weighed crucible used for ashing the paper or board (if the lid is sufficiently well-fitting).

8 Procedure

Ignite the crucible and lid (5.1) cool in the desiccator and weigh to the nearest 0,1 mg; then weigh the test piece to the nearest 0,1 mg in the crucible. Ignite the crucible and contents, taking care to cover the crucible during the early stages of ignition to avoid loss of small particles. Start the ignition at a low temperature and raise it gradually to $900 \pm 25^\circ\text{C}$ as the volatile matter is driven off. With an electric muffle furnace (5.3) this can be accomplished by placing the crucible at the entrance for a few minutes until all volatile matter has been evolved, after which the crucible lid may be carefully removed and put to one side, and the door closed. An ample access of air is necessary because, for example, where barium sulphate is present this could easily be reduced to the sulphite in the presence of carbonaceous matter.

The temperature shall be raised at such a rate that the test piece does not burst into flame, which causes draught and loss of material.

Allow combustion to continue to completion, a condition indicated by the absence of black particles in the ash. Great care is necessary at all stages to protect the crucible and its contents from draughts of air which may cause serious losses. Stirring is not recommended at all, but in the case of large and heavy ashes, it may be necessary to turn the ash fragments over gently with a platinum wire to ensure that complete combustion has taken place. In such cases, extreme caution is necessary to avoid loss of ash.

When combustion is complete, replace the crucible lid and remove the crucible to the desiccator (5.4) and cool for 45 min when porcelain or quartz ware is used, or for 15 min when using platinum; then weigh the crucible and contents to the nearest 0,1 mg and repeat the process of ignition and weighing until the mass is constant, i.e. until two consecutive weighings do not differ by more than 0,5 mg.

NOTES

1 In the case of ashless papers or boards, it may be advisable to ignite successive small portions at low temperature in the same covered crucible in order to reduce the bulk and finally to ignite the whole to constant mass. A small platinum crucible shall always be used in order to minimize any effect that slight errors in the mass of the crucible would contribute to the final result.

2 It may be desirable in certain instances to work at a lower temperature, for example when the paper or board contains an appreciable quantity of calcium carbonate. In such circumstances a temperature of $575 \pm 25^\circ\text{C}$ is recommended.

9 Expression of results

The ash, expressed as a percentage by mass, is given by the formula

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the empty crucible and cover;

m_2 is the mass, in grams, of the crucible, cover and ash.

Take as the result the arithmetic mean of at least two determinations provided that they do not differ by more than 5 % of the mean.

Report the percentage of ash to three significant figures, except in the case of ashless paper, for which two significant figures are sufficient.

10 Test report

The test report shall include the following particulars:

- all the indications necessary for complete identification of the sample;
- reference to this International Standard;
- the ash;
- the temperature used for incineration;
- whether "oven-dry" or "air-dry" (conditioned) basis;
- any unusual features observed in the course of the test;
- any operations not specified in this International Standard or in the International Standards to which reference is made, or regarded as optional, which might have affected the results.

Annex

Special case: Papers and boards loaded during manufacture

(This annex forms part of the standard.)

The method for determination of ash specified in this International Standard will give the ash of any sheet of paper or board, but the significance of the results will depend primarily on the use that is to be made of the information obtained.

When the paper or board contains loading and mineral coating materials which undergo only a negligible change in mass on ignition (for example, titanium dioxide), the ash may be taken as an approximate measure of the amount of mineral matter

present in the paper or board. With most materials, however, there will be a loss, sometimes substantial. For example, oven-dry china clay will lose 11 % to 14 % and calcium carbonate approximately 44 %, while with some materials the loss will vary owing to their indefinite composition. The significance of the ash in terms of the loading in the paper or board can, therefore, only be ascertained by means of a factor when a single material of known chemical composition and behaviour on ignition has been used.

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