

Transformed

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 2144

PAPER AND BOARD

DETERMINATION OF ASH

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BRIEF HISTORY

The ISO Recommendation R 2144, *Paper and board – Determination of ash*, was drawn up by Technical Committee ISO/TC 6, *Paper, board and pulps*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question led to the adoption of Draft ISO Recommendation No. 2144, which was circulated to all the ISO Member Bodies for enquiry in October 1970.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Korea, Rep. of	Sweden
Austria	Netherlands	Switzerland
Czechoslovakia	New Zealand	Thailand
Germany	Norway	Turkey
India	Poland	U.A.R.
Iran	Portugal	United Kingdom
Ireland	Romania	U.S.A.
Israel	South Africa, Rep. of	U.S.S.R.
Japan	Spain	

The following Member Bodies opposed the approval of the Draft :

Belgium
France

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

PAPER AND BOARD

DETERMINATION OF ASH

1. SCOPE

This ISO Recommendation describes the apparatus and procedure for the determination of ash of paper and board.

2. DEFINITION

Ash content. The amount of residue of a material left after incineration determined and expressed according to the procedure given in this ISO Recommendation.

3. APPARATUS

- 3.1 *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 should be avoided when it is suspected that these materials are present in the sample.

- 3.2 *Balance*, with overall accuracy of 0.1 mg.

- 3.3 *Electric muffle furnace*, having an operating temperature of 900 ± 25 °C. A gas-fired muffle or a gas burner may be used provided it is capable of giving a similar temperature.

- 3.4 *Desiccator*.

4. SAMPLING

Sampling should be carried out in accordance with the procedure given in ISO Recommendation R 186, *Method of sampling paper and board for testing*. *

5. PREPARATION OF TEST PIECE

The test piece should 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; they should be 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, sufficient paper or board should be taken 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 conditioning should be carried out in accordance with ISO Recommendation R 187, *Method for the conditioning of paper and board test samples*.

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 Recommendation R 287, *Method for the determination of moisture content of paper (oven-drying method)*, on a separate portion of the sample weighed out at the same time. The result should 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 between 103 and 105 °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).

6. PROCEDURE

Ignite the crucible and lid, 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 ± 25 °C* as the volatile matter is driven off. With an electric muffle furnace 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.

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 and cool for 45 minutes when porcelain or quartz ware is used, or for 15 minutes 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**.

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 should 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 paper or board contains an appreciable quantity of calcium carbonate. In such circumstances a temperature of 575 ± 25 °C is recommended.

7. EXPRESSION OF RESULTS

The results of at least two determinations should be consistent to within 5 % of their mean. The ash content should be reported as a percentage of the mass of oven-dry paper calculated from the mean of these results.

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

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

** Two consecutive weighings should not differ by more than 0.5 mg.

8. TEST REPORT

The test report should include the following information :

- (a) a reference to this ISO Recommendation;
- (b) the ash content;
- (c) the temperature used for incineration;
- (d) whether "oven-dry" or "air-dry" (conditioned) basis;
- (e) any operating details not specified in this ISO Recommendation, or optional;
- (f) any other circumstances that may have affected the results.

9. SIGNIFICANCE OF RESULTS

The method for determination of ash described in this ISO Recommendation will give the ash content 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 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.

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

- 9.1 **Ashless papers and boards** (see term No.153 in ISO Recommendation R 231, *Paper vocabulary – Third series of terms*), 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.
 - 9.2 **Other papers and boards to which no loading has been added.**
 - 9.3 **Papers and boards loaded during manufacture.** The determination of ash, carried out as described, 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.
 - 9.4 **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.
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