International Standard 3451/1

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Plastics — Determination of ash — Part 1 : General methods

Plastiques — Détermination du taux de cendres — Partie 1 : Méthodes générales

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

International Standard ISO 3451/1 was developed by Technical Committee ISO/TC 61, *Plastics*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 3451-1976), which had been approved by the member bodies of the following countries :

Austria	Ireland	Spain
Belgium	Israel	Sweden
Chile	Italy	Switzerland
Finland	Mexico	Thailand
France	Netherlands	Turkey
Germany, F. R.	New Zealand	United Kingdom
Hungary	Poland	USA
India	Portugal	USSR
Iran	Romania	Yugoslavia

No member body had expressed disapproval of the document.

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Plastics — Determination of ash — Part 1 : General methods

1 Scope and field of application

This part of ISO 3451 specifies general methods, with suitable test conditions, for the determination of the ash of a range of plastics (resins and compounds). The particular conditions chosen may be included in the specification for the plastic material in question.

NOTE — Particular conditions applicable to plastics containing glass fibre reinforcement and/or certain fillers will be specified in further parts of ISO 3451 pertaining to specific types of plastics.

2 Principle

It is possible to determine the ash of an organic material by three main methods :

a) Direct calcination, i.e. by burning the organic matter and treating the residue at high temperature until constant mass is reached. (Method A.)

b) Calcination after sulphation, which may be carried out by two different procedures :

— With sulphuric acid treatment after burning, i.e. by burning the organic matter, transforming the inorganic residue into sulphates with concentrated sulphuric acid and treating the residue at high temperature until constant mass is reached. This is the common method of obtaining "sulphated ash". (Method B.)

— With sulphuric acid treatment before burning, i.e. by heating the organic matter together with concentrated sulphuric acid up to temperatures where fuming and subsequent burning of the organic matter occur, and finally treating the residue at high temperature until constant mass is reached. This procedure may be applied if volatile metal halides are liable to evaporate during burning of the organic matter. It should never be applied to silicones or fluorine-containing polymers. (Method C.)

In each case the final step of the operation is a calcination at 600 °C, 750 °C, 850 °C, or 950 °C, until constant mass is reached (see 5.2).

3 Reagents (for methods B and C only)

During the analysis, use only reagents of analytical grade and only distilled water or water of equivalent purity.

3.1 Ammonium carbonate, anhydrous.

3.2 Ammonium nitrate, approximately 10 % (m/m) solution.

3.3 Sulphuric acid, ϱ 1,84 g/ml.

3.4 Sulphuric acid, 50 % (V/V) solution.

4 Apparatus

4.1 Crucible of silica, porcelain or platinum, inert to the material tested.

4.2 Gas burner, or other appropriate heat source.

4.3 Muffle furnace, capable of being controlled at 600 ± 25 °C, 750 ± 50 °C, 805 ± 50 °C, or 950 ± 50 °C as appropriate.

4.4 Analytical balance, accurate to 0,1 mg.

4.5 Pipettes, of suitable capacity (for methods B and C only).

4.6 Desiccator, containing an efficient desiccant which does not interact with the ash.

NOTE — In certain cases, the ash may have a greater affinity for water than some substances commonly used as desiccants.

4.7 Weighing bottle.

5 Procedure

5.1 Test portion

Take a quantity of the test sample sufficient to yield 5 to 50 mg of ash. If the approximate ash is unknown, carry out a preliminary ash determination.

NOTE — According to the result obtained, the recommended test portion is chosen from the following table.

Approximate ash	Test portion	Mass of ash obtained
%	g	mg
< 0,01	200 min.	5 to 50
> 0,01 to 0,05	100	10 to 50
> 0,05 to 0,1	50	25 to 50
> 0,1 to 0,2	25	25 to 50
> 0,2	10 max.	20 to 50

For plastics yielding very low ash, it is necessary to use large test portions. When it is impossible to burn the whole of the test portion at one time, weigh the required quantity in a suitable weighing bottle and introduce it into the crucible (4.1) in convenient amounts at a time for a succession of burnings until the whole of the test portion has been burnt.

5.2 Test conditions

Calcination shall be continued to constant mass as defined in 5.3.6, but the duration of the calcination in the muffle furnace (4.3) shall not exceed 3 h at the specified temperature.

The choice of the calcination temperature and the use of the sulphation method depend on the nature of the plastic and any additives it may contain. If a choice exists between different satisfactory conditions, choose those which allow the attainment of constant mass in less than 3 h. A higher temperature or the use of sulphation generally shortens the duration of the calcination.

Whichever method — A, B or C — is used, choose one of the following temperature ranges for the final step of calcination, unless other temperatures are requested for special technical or commercial reasons :

600 ± 25 °C, 750 ± 50 °C, 850 ± 50 °C, 950 ± 50 °C.

5.3 Method A — Direct calcination

5.3.1 Prepare the crucible (4.1) by heating it in the muffle furnace (4.3) at the test temperature until constant mass is reached. Allow to cool in the desiccator (4.6) to room temperature for at least 1 h and weigh on the analytical balance (4.4) to the nearest 0,1 mg.

5.3.2 Introduce into the tared weighing bottle (4.7) the test portion, predried as described in the corresponding material specification or with a known volatile matter content corresponding to 5 to 50 mg of ash. Weigh again to the nearest 0,1 mg or to 0,1 % of the mass of the test portion. If the crucible will accommodate the test portion corresponding to the 5 to 50 mg of ash, this quantity may be placed directly into the crucible and weighed in it. High bulk materials may be compressed into tablets which may then be broken up into fragments of appropriate size.

5.3.3 Introduce into the crucible enough of the material to be calcined so that it is not more than half full. Heat the crucible directly on the burner or other suitable heating device (4.2) to burn slowly. Burning shall not be too vigorous to avoid loss of ash particles. Cool and add another part of the test portion. Repeat the operation described until the whole test portion is consumed.

5.3.4 Introduce the crucible into the muffle furnace preheated to the prescribed temperature and calcine for 30 min.

5.3.5 Place the crucible in the desiccator, allow it to cool to room temperature for 1 h and weigh on the analytical balance (4.4) to the nearest 0,1 mg.

5.3.6 Calcine again under the same conditions until constant mass is reached, i.e. until the results of two consecutive weighings do not differ from each other by more than 0,5 mg.

5.4 Method B — Calcination following sulphuric acid treatment after burning

5.4.1 Proceed as specified in 5.3.1, 5.3.2 and 5.3.3.

5.4.2 After cooling, add the sulphuric acid solution (3.4) drop by drop with a pipette of suitable capacity (4.5) to moisten the residue completely and heat until fuming ceases, avoiding too vigorous boiling.

5.4.3 If traces of carbonaceous materials remain after cooling, add 1 to 5 drops of the ammonium nitrate solution (3.2) and heat until the evolution of white fumes ceases completely.

5.4.4 In order to reconvert metal oxides formed during the preceding steps into sulphates, add, after cooling, about 5 drops of the concentrated sulphuric acid (3.3) and heat until there is no further evolution of white fumes, avoiding vigorous boiling or the loss of ash by excessive fuming.

5.4.5 After cooling, add 1 to 2 g of the solid ammonium carbonate (3.1) and heat, avoiding loss of ash, until the fuming has ceased. Then place the crucible in the muffle furnace preheated to the indicated temperature and proceed as specified in 5.3.4, 5.3.5 and 5.3.6.

5.5 Method C — Calcination following sulphuric acid treatment before burning

5.5.1 Proceed as specified in 5.3.1 and 5.3.2.

5.5.2 Introduce into the crucible enough of the material to be calcined so that it is not more than half full. Add with a pipette (4.5) a sufficient amount of the concentrated sulphuric acid (3.3) to moisten the material completely. Cover the crucible with a watch-glass. Heat the crucible directly on the burner at low flame until the organic material begins to decompose.

Continue heating carefully, adjusting the watch-glass so as to allow the acid to be fumed off, making sure that no ash-containing material is lost. With plastics which have a tendency to lose ash-containing material, it is recommended that the crucible with its contents be placed into a holed asbestos board and heated with a low flame only until the organic matter smoulders rather than burns. If the initial charge into the crucible was insufficient to yield an acceptable mass of ash, allow the crucible to cool, add another part of the test portion and repeat the operation described before until the whole test portion is burned in the crucible. Remove the watch-glass, making sure that no solid particles are adhering to it.

NOTE — In cases where the sulphuric acid tends to creep over the lip of the crucible or where, despite precautions, some of the test portion tends to be lost from it by violent reaction (frequently in the case of PVC), the concentrated sulphuric acid may be replaced by a mixture of concentrated acetic and sulphuric acids. The use of these mixed acids should be agreed between the interested parties and reference made to it in the test report.

5.5.3 Proceed as specified in 5.4.3, 5.4.4 and 5.4.5.

6 Number of tests

The number of tests and the permissible scatter of results shall be stated in the particular standard for each material. If such information is not available, carry out two determinations and repeat the test as necessary until the results of two successive determinations do not differ from each other by more than 10 % of their mean.

7 Expression of results

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

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

where

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

m_1 is the mass, in grams, of ash obtained.

8 Test report

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the material tested;

c) reference to the method used (A, B or C), and to the acetic and sulphuric acid mixture, if used (see the note to 5.5.2);

d) calcining temperature used;

e) number and masses of test portions used;

f) ash results and scatter.