
Plastics — Determination of ash —

**Part 1:
General methods**

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

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Contents

	Page
Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Reagents (for methods B and C only)	2
6 Apparatus	2
7 Procedure	3
7.1 Test portion.....	3
7.2 Test conditions.....	4
7.3 Method A — Direct calcination.....	5
7.4 Method B — Calcination following sulfuric acid treatment after burning.....	5
7.5 Method C — Calcination following sulfuric acid treatment before burning.....	6
7.6 Method D —Automated instrument method.....	6
8 Number of tests	7
9 Expression of results	7
10 Precision	7
11 Test report	8
Bibliography	9

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

This fifth edition cancels and replaces the fourth edition (ISO 3451-1:2008), which has been technically revised by adding an automated instrument method (Method D).

A list of all parts in the ISO 3451 series can be found on the ISO website.

Plastics — Determination of ash —

Part 1: General methods

SAFETY PRECAUTIONS — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document specifies general methods, with suitable test conditions, for the determination of the ash of a range of plastics. The particular conditions chosen can be included in the specifications for the plastic material in question.

Particular conditions applicable to poly(alkylene terephthalate) materials, unplasticized cellulose acetate, polyamides and poly(vinyl chloride) plastics, including some specific filled, glass-fibre-reinforced and flame-retarded materials, are specified in ISO 3451-2, ISO 3451-3, ISO 3451-4 and ISO 3451-5.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

It is possible to determine the ash of an organic material by four main methods (methods A, B, C and D).

- a) Direct calcination, i.e. by burning the organic matter and heating the residue at high temperature until constant mass is reached, which may be carried out by two different procedures:
 - burning with, for example, a Bunsen burner and one or more calcining steps in a muffle furnace (method A);
 - burning and calcining as one single step in a muffle furnace (method A — rapid ashing), if it can be demonstrated that the rapid ashing yields the same results as method A.

- b) Calcination after sulfation, which may be carried out by two different procedures:
- with sulfuric acid treatment after burning, i.e. by burning the organic matter, transforming the inorganic residue into sulfates with concentrated sulfuric acid and heating the residue at high temperature until constant mass is reached. This is the common method of obtaining “sulfated ash” (method B);
 - with sulfuric acid treatment before burning, i.e. by heating the organic matter together with concentrated sulfuric acid up to temperatures where fuming and subsequent burning of the organic matter occur, and finally heating the residue at high temperature until constant mass is reached (method C). This procedure may be used if volatile metal halides are liable to evaporate during burning of the organic matter. It is not applicable to silicones or fluorine-containing polymers.
- c) Automated instrument method (method D).

In each case, the final step of the procedure is calcination at a pre-defined temperature (see 7.2).

NOTE The mass of the ash can vary with the temperature of calcination. For example, higher temperatures such as 850 °C convert calcium carbonate and other carbonates to their oxides and thus give lower values for the ash.

5 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.

5.1 **Ammonium carbonate**, anhydrous.

5.2 **Ammonium nitrate**, approximately 10 % (by mass) solution.

5.3 **Sulfuric acid**, concentrated 98 %, $\rho = 1,84 \text{ g/cm}^3$.

WARNING — Highly corrosive. Handle with suitable skin and eye protection in a fume cupboard.
Reacts exothermically with water.

5.4 **Sulfuric acid**, 50 % (by volume) solution.

WARNING — Handle with care. Prepare by slowly adding the concentrated acid to water.

6 Apparatus

some of the listed items may not be necessary for each method.

6.1 **Crucible**, made of silica, porcelain, ceramics, fibres, quartz, glass or platinum, thereof, inert to the material tested and suitable for the temperatures used. The use of a crucible lid/watch-glass may be beneficial for samples producing a fine particulate ash.

6.2 **Gas burner**, or other appropriate heat source.

6.3 **Muffle furnace**, powered by electric resistance heating or by microwave heating, capable of being maintained at $(600 \pm 25) \text{ }^\circ\text{C}$, $(750 \pm 50) \text{ }^\circ\text{C}$, $(850 \pm 50) \text{ }^\circ\text{C}$ or $(950 \pm 50) \text{ }^\circ\text{C}$, as appropriate.

6.4 **Analytical balance**, accurate to 0,1 mg.