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## Plastics — Determination of ash — Part 4: Polyamides

*Plastiques — Détermination du taux de cendres —  
Partie 4: Polyamides*

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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 document 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](http://www.iso.org/directives)).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 3451-4:1998), which has been technically revised.

The main changes are as follows:

- automatic device method (method D) has been added according to align with the revised ISO 3451-1:2019.

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

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](http://www.iso.org/members.html).

# Plastics — Determination of ash —

## Part 4: Polyamides

**WARNING** — This document can involve hazardous chemicals, materials and operations. This document does not purport to address the safety problems associated with its use. It is the responsibility of the user to establish proper safety and health practices and determine any applicable regulatory limitations prior to use.

### 1 Scope

This document specifies methods for determination of the ash of polyamides, both filled and unfilled. It follows the general procedures given in ISO 3451-1.

### 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 3451-1:2019, *Plastics — Determination of ash — Part 1: General methods*

### 3 Terms and definitions

No terms and definitions are listed in this document.

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

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

### 4 Principle

#### 4.1 General

For unfilled materials, method A and method D or method C of ISO 3451-1:2019 is used.

For filled and glass-fibre reinforced materials, method A and method D of ISO 3451-1:2019 is used.

For glass-fibre filled materials containing flame retardant, antimony trioxide, and/or other, volatilizable, additives such as pigment zinc sulfide, a modification is incorporated to remove these as volatile bromine component(s).

#### 4.2 Unfilled materials

Direct calcination by burning the organic matter and treating the residue at a high temperature until constant mass is reached (ISO 3451-1:2019, method A and method D).

If the material contains metal halides and/or metals (especially in the presence of halogenated material) which are liable to evaporate during burning of the organic matter, calcination after sulfation can

be applied (ISO 3451-1:2019, method C). This procedure is carried out by heating the organic matter together with concentrated sulfuric acid up to a temperature where fuming and subsequent burning of the organic matter occurs, and finally treating the residue at a high temperature until constant mass is reached.

### 4.3 Filled and glass-fibre reinforced materials

Direct calcination, by burning the organic matter and treating the residue at a high temperature until constant mass is reached (ISO 3451-1: 2019, method A and method D) (see [4.4.2](#)).

### 4.4 Flame-retardant materials reinforced with glass fibre

**4.4.1** Calcination by burning the organic matter in the presence of decabromobiphenyl (DBB) and finally treating the residue at a high temperature until constant mass is reached (ISO 3451-1:2019, method A and method D).

**4.4.2** Some additives, for instance zinc sulfide, are also completely volatilized as bromides by burning the organic matter in contact with decabromobiphenyl (DBB). Information with respect to the additive(s) present in the material and the potential to evaporate during burning in contact with DBB should be requested from the supplier or obtained by testing on the pure chemical.

**4.4.3** Flame retardants with a high bromine content, for instance ethylene bis(tetrabromophthalimide) or brominated polystyrene may also be used. The applicability and amount of chemical to be used can be determined by carrying out ash determinations in accordance with [7.5](#) with increasing amounts of flame retardant until a constant result is obtained. The chemical should be applied as a powder.

## 5 Reagents (method C or method A in the presence of DBB)

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

**5.1 Ammonium carbonate**, anhydrous.

**5.2 Ammonium nitrate**, approximately a mass fraction of 10 % solution.

**5.3 Sulfuric acid**,  $\rho$  1,84 g/ml.

**5.4 Decabromobiphenyl (DBB)**, powder, technical grade or higher purity.

**WARNING** — The use of decabromobiphenyl can result in the formation of dioxins. Temperatures in the order of 600 °C to 850 °C are generally known as ideal temperatures for the formation of dioxins.

## 6 Apparatus

Apparatus specified in ISO 3451-1:2019, Clause 4 and in particular:

**6.1 Crucibles of silica, porcelain or platinum**, inert to the material tested, and typically of diameter (upper part) 50 mm to 60 mm and height equal to the diameter (see [4.4.3](#)).

**6.2 Muffle furnace**, powered by electric resistance heating or by microwave heating, capable of being maintained at  $(600 \pm 25)$  °C,  $(850 \pm 50)$  °C or at a minimum temperature of 850 °C.

**6.3 Fume cupboard.**