
International Standard



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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

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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 3451/4 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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Plastics — Determination of ash — Part 4: Polyamides

1 Scope and field of application

This part of ISO 3451 specifies three methods for the determination of the ash of polyamides. The general procedures in ISO 3451/1 are followed. For unfilled materials, method C of ISO 3451/1 is used. For filled and glass-fibre reinforced materials, method A of ISO 3451/1 is used. For flame-retardant materials reinforced with glass-fibre, a modification is incorporated to remove any antimony trioxide present.

2 Reference

ISO 3451/1, *Determination of ash — Part 1: General methods.*

3 Principle

3.1 Unfilled materials

Calcination with sulfuric acid treatment before burning (method C of ISO 3451/1).

3.2 Filled and glass-fibre reinforced materials

Direct calcination, i.e. by burning the organic matter and treating the residue at high temperature until constant mass is reached (method A of ISO 3451/1).

3.3 Flame-retardant materials reinforced with glass-fibre

Calcination by burning the organic matter, cooling and treating the residue with an excess of hydrochloric acid solution, then heating gently at first until evolution of fumes ceases and finally treating the residue at high temperature until constant mass is reached.

4 Reagents

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

4.1 Ammonium carbonate, anhydrous.

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

4.3 Sulfuric acid, $\rho = 1,84$ g/ml, approximately 98 % (m/m) solution.

4.4 Hydrochloric acid, 32 % (m/m) hydrogen chloride solution.

5 Apparatus

Apparatus specified in ISO 3451/1, and in particular:

5.1 Crucibles of silica or platinum, diameter (upper part) 50 to 60 mm, height equal to the diameter, inert to the material tested.

5.2 Muffle furnace, capable of being controlled thermostatically at 600 ± 25 °C or 750 ± 50 °C.

6 Procedure

The sample shall be in the form of small pieces, granules or powder. Dry the samples of filled or reinforced materials before test, for example by heating at 100 °C *in vacuo* until constant mass is reached.

Take a quantity of the test sample sufficient to yield 5 to 50 mg of ash (in the case of materials reinforced with glass-fibre, take 10 g).

If the approximate ash is unknown, carry out a preliminary ash determination. According to the approximate ash, choose the size of test portion to be used from the table on the following page.

6.1 Unfilled materials

Follow the procedure in ISO 3451/1, method C, applying a calcination temperature of 750 ± 50 °C.

6.2 Filled and glass-fibre reinforced materials

Follow the procedure in ISO 3451/1, method A, applying a calcination temperature of 750 ± 50 °C. If at that temperature glass fibres present become molten and thus prevent further calcination of the polymer, lower the temperature of calcination to 600 ± 25 °C.