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# International Standard



# 3451 / 4

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

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

First edition — 1986-08-15

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UDC 678.675 : 543.822

Ref. No. ISO 3451/4-1986 (E)

Descriptors : plastics, polyamide, tests, combustion analysis, ash determination.

Price based on 2 pages

## 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  $600 \pm 25$  °C or  $750 \pm 50$  °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  $750 \pm 50$  °C.

### 6.2 Filled and glass-fibre reinforced materials

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

Approximate ash	Test portion	Mass of ash obtained
%	g	mg
< 0,01	200 min.	5 to 10
> 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

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

Proceed as in sub-clauses 5.3.1, 5.3.2 and 5.3.3 of ISO 3451/1, method A. Then cool the residue and treat with an excess of the hydrochloric acid solution (4.4), i.e. 0,5 ml per gram of test portion. Heat gently with a Bunsen burner until evolution of fumes ceases. Continue as specified in ISO 3451/1, method A, applying a calcination temperature of  $750 \pm 50$  °C, or  $600 \pm 25$  °C, as indicated in 6.2.

### 7 Number of tests

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.

### 8 Expression of results

The ash or sulfated 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.

### 9 Test report

The test report shall include the following particulars:

- reference to this part of ISO 3451;
- complete identification of the material tested;
- mass of test portion used;
- treatment, if any, of the test portions prior to the test;
- temperature used for calcination;
- individual results of the two determinations that do not differ from each other by more than 10 % of their mean (see clause 7) and the average for the ash or sulfated ash.

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