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International Standard



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**Plastics — Determination of ash —
Part 2: Polyalkylene terephthalates**

Plastiques — Détermination du taux de cendres — Partie 2: Polyalkylène téréphtalates

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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 3451/2 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in July 1982.

It has been approved by the member bodies of the following countries:

Australia	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Sri Lanka
China	Japan	Sweden
Czechoslovakia	Korea, Rep. of	Switzerland
Egypt, Arab Rep. of	Netherlands	Tanzania
Finland	New Zealand	United Kingdom
France	Poland	USSR
Hungary	Romania	

The member body of the following country expressed disapproval of the document on technical grounds:

Germany, F.R.

Plastics — Determination of ash — Part 2: Polyalkylene terephthalates

1 Scope and field of application

This part of ISO 3451 specifies methods for the determination of the ash of polyethylene terephthalate, polybutylene terephthalate and copolymers of the two, both filled and unfilled. The general procedures given 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 glass-fibre filled materials, a modification is incorporated to remove any antimony trioxide present.

2 Reference

ISO 3451/1, *Determination of ash — Part 1: General methods*.
ISO 3451-2:1984
 http://www.iso.org/iso/catalogue/sist/455821ac-a4e7-48d6-9032-775975d5d8d6/iso-3451-2-1984

3 Principle

3.1 Unfilled materials

Calcination after sulfation, 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 treating the residue at high temperature until constant mass is reached.

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.

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 (4.4), 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 (for method C only)

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, ρ 1,84 g/ml.

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.

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

6 Procedure

6.1 Test portion

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 following table.

Approximate ash %	Test portion g	Mass of ash obtained 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

6.2 Unfilled materials

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

6.3 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.

6.4 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) (0,5 ml per gram of test portion). Heat gently with a Bunsen burner until evolution of fumes ceases. Continue as directed in ISO 3451/1, method A.

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.

Report the ash as the mean of two results that do not differ by more than 10 %.

9 Test report

The test report shall include the following particulars:

- a) reference to this International Standard;
- b) complete identification of the sample; including type, manufacturer's code number, source, trade name, etc.;
- c) temperature used for calcination;
- d) treatment of the sample prior to the test, if any;
- e) individual results of the two determinations and the average for the ash or sulfated ash obtained;
- f) mass of test portion used.

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