International Standard ISO 3451/2

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Determination of ash — Part 2: Polyalkylene terephthalates

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

First edition — 1984-05-15

UDC 678.644 : 543.822 **Ref. No. ISO 3451/2-1984 (E)**

Descriptors : plastics, tests, determination, ashes.

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.

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:

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

Germany, F.R.

Printed in Switzerland

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.*

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	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