SLOVENSKI STANDARD
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Polimerni materiali - Določ evanje pepela - 2. del: Polialkilenteraftalatni materiali

Plastics -- Determination of ash -- Part 2: Poly(alkylene terephthalate) materials

Plastiques -- Détermination du taux de cendres -- Partie 2: Matières poly(téréphtalate d'alkylène)

Ta slovenski standard je istoveten z: **ISO 3451-2:1998**

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INTERNATIONAL STANDARD

ISO 3451-2

Second edition
1998-12-15

Plastics — Determination of ash —

Part 2:
Poly(alkylene terephthalate) materials

Plastiques — Détermination du taux de cendres —

Partie 2: Matières poly(téréphtalate d'alkylène)

Reference number
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Contents

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3451-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 3451-2:1984), which has been technically revised.

ISO 3451 consists of the following parts, under the general title *Plastics — Determination of ash*:

— *Part 1: General methods*

— *Part 2: Poly(alkylene terephthalate) materials*

— *Part 3: Unplasticized cellulose acetate*

— *Part 4: Polyamides*

— *Part 5: Poly(vinyl chloride)*

Plastics — Determination of ash —

Part 2:
Poly(alkylene terephthalate) materials

WARNING — This part of ISO 3451 may involve hazardous chemicals, materials and operations. This part of ISO 3451 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 the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 3451 specifies methods for determination of the ash of poly(ethylene terephthalate), poly(butylene terephthalate) and copolymers of the two, both filled and unfilled. The general procedures given in ISO 3451-1 are followed. For unfilled materials method A or method C of ISO 3451-1:1997 is used. For filled and glass-fibre reinforced materials method A of ISO 3451-1:1997 is used.

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

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 3451. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 3451 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3451-1:1997, *Plastics — Determination of ash — Part 1: General methods.*

3 Principle

3.1 Unfilled materials

Direct calcination by burning the organic matter and treating the residue at a high temperature until constant mass is reached (ISO 3451-1:1997, method A).

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 3451-1:1997, 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.

3.2 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 3451-1:1997, method A) (see note 1 in 3.3).

3.3 Flame-retardant materials reinforced with glass fibre

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 3451-1:1997, method A).

NOTE 1 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.

NOTE 2 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 6.4 with increasing amounts of flame retardant until a constant result is obtained. The chemical should be applied as a powder.

4 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.

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

WARNING — The use of decabromobiphenyl may 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.

5 Apparatus

Apparatus specified in clause 4 of ISO 3451-1:1997, and in particular:

5.1 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 note 2 in 3.3).

5.2 Muffle furnace, capable of being maintained at (600 ± 25) °C, (850 ± 50) °C or at a minimum temperature of 850 °C.

5.3 Fume cupboard.

6 Procedure

The material shall be in the form of small pieces of 1 cm × 0,5 cm × 0,2 cm or smaller, granules or powder. Filled or reinforced materials shall be dried before calcination, i.e. by heating at 100 °C until constant mass is reached.

6.1 Test portion

Take a quantity of the test sample sufficient to yield 5 mg to 200 mg of ash (see table 1). In the case of reinforced materials, take a test portion of 2 g or more (see table 1). If the likely quantity of ash is unknown, carry out a preliminary ash determination. According to the approximate ash content, choose the size of the test portion to be used from table 1.

Table 1 — Mass of test portion

Approximate ash %	Test portion g	Mass of ash obtained mg
⩽ 0,01	⩾ 200	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 to 1	10	20 to 100
> 1 to 10	5	50 to 500
> 10	2	200

6.2 Unfilled materials

Follow the procedure described in ISO 3451-1:1997, method A, applying a calcination temperature of (850 ± 50) °C.

If the material contains metal halides or metals in the presence of halogenated material, liable to evaporate during the calcination procedure, or in those cases where “sulfated ash” is required, method C shall be applied (ISO 3451-1:1997, method C).

6.3 Filled and glass-fibre reinforced materials

Follow the procedure described in ISO 3451-1:1997, method A, applying a calcination temperature of (850 ± 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 and repeat the procedure with a fresh test portion.

6.4 Materials reinforced with glass fibre containing flame-retardant antimony trioxide and/or other volatilizable additives

The sample shall be ground or cut to pieces of 1 cm × 0,5 cm × 0,2 cm or smaller. Proceed as in 5.3.1 and 5.3.2 of ISO 3451-1:1997, method A.

Add to the sample a quantity of DBB (4.4) equal to half the mass of the sample in grams and mix well in the crucible. Place the crucible in the muffle furnace, applying a calcination temperature of at least 850 °C. The muffle furnace shall be placed in a fume cupboard. Continue as directed in ISO 3451-1:1997, from 5.3.4 of method A.

Directly placing the crucible into the muffle furnace is preferred. If direct calcination leads to large differences between repeated tests, for instance due to loss of ash-containing material, gently heat the crucible over a quiet flame until formation of fumes ceases. Make sure that the volatile components are properly drawn off by a fume cupboard. Place the crucible in the muffle furnace and apply a calcination temperature of at least 850 °C. Continue as directed in ISO 3451-1:1997, from 5.3.4 of method A. Glass-fibre crucibles provided with two glass-fibre discs may also be used. The crucible shall be prepared by heating it in the muffle furnace at the test temperature and cooling it in a desiccator until constant mass is reached. Introduce the sample between the glass-fibre discs into the crucible. Place the crucible in the muffle furnace for 30 min. Allow the crucible to cool in a desiccator for 20 min.

For unground material, the residue in the crucible shall be calcinated a second time according to the procedure described in 6.4. The amount of DBB added to the residue shall be 1 g.