SLOVENSKI STANDARD
SIST EN ISO 3451-1:2000

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Polimerni materiali - Določ evanje pepela - 1. del: Splošne metode (ISO 3451-1:1997)

Plastics - Determination of ash - Part 1: General methods (ISO 3451-1:1997)

Kunststoffe - Bestimmung der Asche - Teil 1: Allgemeine Grundlagen (ISO 3451-1:1997)

Plastiques - Détermination du taux de cendres - Partie 1: Méthodes générales (ISO 3451-1:1997)

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Plastics - Determination of ash - Part 1: General methods (ISO 3451-1:1997)

Plastiques - Détermination du taux de cendres - Partie 1: Méthodes générales (ISO 3451-1:1997)

Kunststoffe - Bestimmung der Asche - Teil 1: Allgemeine Grundlagen (ISO 3451-1:1997)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by month of January 1998, and conflicting national standards shall be withdrawn at the latest by January 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 3451-1:1997 has been approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 3451-1

Third edition
1997-07-15

Plastics — Determination of ash —

Part 1:
General methods

Plastiques — Détermination du taux de cendres —

Partie 1: Méthodes générales

Reference number
ISO 3451-1:1997(E)

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 3451-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 3451-1:1981), which has been technically revised. The main change is the inclusion of a precision clause (clause 8).

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

— *Part 1: General methods*

— *Part 2: Polyalkylene terephthalates*

— *Part 3: Unplasticized cellulose acetate*

— *Part 4: Polyamides*

— *Part 5: Poly(vinyl chloride)*

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Plastics — Determination of ash —

Part 1:
General methods

1 Scope

This part of ISO 3451 specifies general methods, with suitable test conditions, for the determination of the ash of a range of plastics (resins and compounds). The particular conditions chosen may be included in the specifications for the plastic material in question.

Particular conditions applicable to plastics containing glass fibre reinforcement, fillers and/or certain additives are specified in further parts of ISO 3451 pertaining to specific types of plastics (see foreword).

2 Principle

It is possible to determine the ash of an organic material by three main methods:

a) Direct calcination, i.e. by burning the organic matter and heating the residue at high temperature until constant mass is reached (method A).

b) Calcination after sulfation, which may be carried out by two different procedures:

   — With sulfuric acid treatment after burning, i.e. by burning the organic matter, transforming the inorganic residue into sulfates with concentrated sulfuric acid and heating the residue at high temperature until constant mass is reached. This is the common method of obtaining "sulfated ash" (method B).

   — 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 heating the residue at high temperature until constant mass is reached (method C). This procedure may be used if volatile metal halides are liable to evaporate during burning of the organic matter. It is not applicable to silicones or fluorine-containing polymers.

In each case, the final step of the procedure is calcination at 600 °C, 750 °C, 850 °C or 950 °C until constant mass is reached (see 5.2).

3 Reagents (for methods B and C only)

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

3.1 Ammonium carbonate, anhydrous.

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

3.3 Sulfuric acid, $\rho = 1{,}84$ g/cm^3.

WARNING — Care should be taken in handling.

3.4 Sulfuric acid, 50 % (*V*/*V*) solution.

WARNING — Care should be taken in handling.

4 Apparatus

4.1 Crucible, made of silica, porcelain or platinum, inert to the material tested.

4.2 Gas burner, or other appropriate heat source.

4.3 Muffle furnace or **microwave furnace**, capable of being maintained at 600 °C ± 25 °C, 750 °C ± 50 °C, 850 °C ± 50 °C or 950 °C ± 50 °C as appropriate.

4.4 Analytical balance, accurate to 0,1 mg.

4.5 Pipettes, of suitable capacity (for methods B and C only).

4.6 Desiccator, containing an efficient desiccant which does not interact with the ash.

NOTE — In certain cases, the ash may have a greater affinity for water than some substances commonly used as desiccants.

4.7 Weighing bottle.

4.8 Fume cupboard.

5 Procedure

5.1 Test portion

Take a quantity of the test sample sufficient to yield 5 mg to 50 mg of ash. If the likely quantity of ash is unknown, carry out a preliminary determination.

Recommended test portion sizes are given in table 1.

Table 1 — Recommended size of test portion

Approximate ash (if known) %	Test portion g	Mass of ash obtained mg
≤ 0,01	200 min.	5 to 50
> 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

For plastics yielding very low ash, it is necessary to use large test portions. When it is impossible to burn the whole of the test portion at one time, weigh the required quantity in a suitable weighing bottle and introduce it into the crucible (4.1) in convenient amounts for a succession of burnings until the whole of the test portion has been burnt.

5.2 Test conditions

Calcination shall be continued to constant mass as defined in 5.3.6, but the duration of the calcination in the muffle furnace (4.3) shall not exceed 3 h at the specified temperature.

The choice of the calcination temperature and the use of the sulfation method depend on the nature of the plastic and any additives it may contain. If a choice exists between different satisfactory conditions, choose those which allow the attainment of constant mass in less than 3 h. A higher temperature or the use of sulfation generally shortens the duration of the calcination.

Whichever method — A, B or C — is used, choose one of the following temperature ranges for the final (calcination) step, unless other temperatures are requested for special technical or commercial reasons:

600 °C ± 25 °C, 750 °C ± 50 °C, 850 °C ± 50 °C, 950 °C ± 50 °C

Use a fume cupboard for the ashing procedure.

5.3 Method A — Direct calcination

5.3.1 Prepare the crucible (4.1) by heating it in the muffle furnace (4.3) at the test temperature until constant mass is reached. Allow to cool in the desiccator (4.6) to room temperature for 1 h, or until room temperature is reached, and weigh on the analytical balance (4.4) to the nearest 0,1 mg.

5.3.2 Introduce into the tared weighing bottle (4.7) a test portion, predried as described in the corresponding material specification or with a known volatile-matter content, corresponding to 5 mg to 50 mg of ash. Weigh again to the nearest 0,1 mg or to 0,1 % of the mass of the test portion. If the crucible will accommodate the test portion corresponding to 5 mg to 50 mg of ash, this quantity may be placed directly into the crucible and weighed in it. The procedure described below assumes that this will not be the case, however. High-bulk materials may be compressed into tablets which may then be broken up into fragments of appropriate size.

5.3.3 Introduce into the crucible enough of the test portion to half fill the crucible. Heat the crucible directly on the burner or other suitable heating device (4.2) to burn slowly. Burning shall not be too vigorous, to avoid loss of ash particles. Cool and add more of the test portion. Repeat the operations described above until the whole test portion has been burnt.

5.3.4 Introduce the crucible into the muffle furnace preheated to the prescribed temperature and calcine for 30 min.

5.3.5 Place the crucible in the desiccator, allow it to cool for 1 h, or until room temperature is reached, and weigh on the analytical balance (4.4) to the nearest 0,1 mg.

5.3.6 Calcine again under the same conditions until constant mass is reached, i.e. until the results of two consecutive weighings do not differ from each other by more than 0,5 mg.

5.4 Method B — Calcination following sulfuric acid treatment after burning

5.4.1 Proceed as specified in 5.3.1, 5.3.2 and 5.3.3.

5.4.2 After cooling, add sulfuric acid solution (3.4) drop by drop with a pipette of suitable capacity (4.5) to moisten the residue completely and heat until fuming ceases, avoiding too vigorous boiling.