INTERNATIONAL STANDARD

ISO 3451-1

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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, ρ = 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