SLOVENSKI STANDARD
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Polimerni materiali - Določevanje pepela - 1. del: Splošne metode (ISO/DIS 3451-1:2018)

Plastics - Determination of ash - Part 1: General methods (ISO/DIS 3451-1:2018)

Kunststoffe - Bestimmung der Asche - Teil 1: Allgemeine Grundlagen (ISO/DIS 3451-1:2018)

Plastiques - Détermination du taux de cendres - Partie 1: Méthodes générales (ISO/DIS 3451-1:2018)

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Plastics — Determination of ash —

Part 1: General methods

Plastiques — Détermination du taux de cendres —

Partie 1: Méthodes générales

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This fifth edition cancels and replaces the fourth edition (ISO 3451-1:2008), which has been technically revised by adding an automated instrument method (Method D).

A list of all parts in the ISO 3451- series can be found on the ISO website.

Plastics — Determination of ash —

Part 1:
General methods

SAFETY PRECAUTIONS — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This document 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 poly(alkylene terephthalate) materials, unplasticized cellulose acetate, polyamides and poly(vinyl chloride) plastics, including some specific filled, glass-fibre-reinforced and flame-retarded materials, are specified in ISO 3451-2, ISO 3451-3, ISO 3451-4 and ISO 3451-5.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

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

— direct calcination, i. e. by burning the organic matter and heating the residue at high temperature until constant mass is reached, which may be carried out by two different procedures:

— burning with e.g. a Bunsen burner and one or more calcining steps in a muffle furnace (method A);

— burning and calcining as one single step in a muffle furnace (method A — rapid ashing), if it can be demonstrated that the rapid ashing yields the same results as method A;

— 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.

— automated instrument method (method D).

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 7.2).

NOTE The mass of the ash may vary with the temperature of calcination. For example, higher temperatures such as 850 °C will convert calcium carbonate and other carbonates to their oxides and thus give lower values for the ash.

5 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.

5.1 Ammonium carbonate, anhydrous.

5.2 Ammonium nitrate, approximately 10 % (by mass) solution.

5.3 Sulfuric acid, concentrated 98 %, $\rho = 1{,}84$ g/cm^3.

WARNING — Highly corrosive. Handle with suitable skin and eye protection in a fume cupboard. Reacts exothermically with water.

5.4 Sulfuric acid, 50 % (by volume) solution.

WARNING — Handle with care. Prepare by slowly adding the concentrated acid to water.

6 Apparatus

Some of the listed items may not be necessary for each method.

6.1 Crucible, made of silica, porcelain, ceramics, fibres , quartz, glass or platinum, thereof, inert to the material tested and suitable for the temperatures used. The use of a crucible lid/watch-glass may be beneficial for samples producing a fine particulate ash.

6.2 Gas burner, or other appropriate heat source.

6.3 Muffle furnace, powered by electric resistance heated or by microwave heating, capable of being maintained at 600 °C ± 25 °C, 750 °C ± 50 °C, 850 °C ± 50 °C or 950 °C ± 50 °C, as appropriate.

6.4 Analytical balance, accurate to 0,1 mg.

6.5 For **automated instruments**, the following requirements apply:

— fully automatic analysis instrument with integrated drying oven, muffle furnace, analytical balance and a sample turntable;

— loss of weight during the drying and ashing process is preferably continuously monitored during the ashing process;

— the built-in analytical balance can weigh each sample in turn, and recorded by the software;

— instrument should have a method to correct the mass difference caused by buoyancy, thus the weighing can be took in different temperature, samples do not need to be cooled down during test;

— having a furnace that operates at the prescribed temperature, and capability to maintain a constant temperature constant to within ±5°C;

— having a holder for one or more crucibles, e.g. by using a turntable, or an automatic way to introduce and extract the crucibles consecutively into and from the furnace;

— temperature and sample mass ranges in accordance with the experimental requirements;

— recording device capable of automatically recording the sample mass versus temperature and time;

— measurement of temperature signals with an accuracy of ±2% or better;

— measurement of mass with an accuracy of ±0,1 mg or better.

NOTE Automatic ash determination equipment, in line with the specifications prescribed in this standard, is produced by several equipment manufacturers.

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

6.7 **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.

6.8 **Weighing bottle.**

6.9 **Fume cupboard** or other suitable means of ventilation.

7 Procedure

7.1 Test portion

Take a quantity of the test sample sufficient to yield 5 mg to 500 mg of ash (see Table 1). In the case of reinforced materials, take a test portion of according to Table 1. If the likely quantity of ash is unknown, carry out a preliminary ash determination. Depending on the approximate ash content, choose the size of the test portion to be used from Table 1, the mass portion may have direct effect on result.

Table 1 — Mass of test portion

Approximate ash %	**Test portion** g	**Mass of ash obtained** mg
≤ 0,01	≥ 200	approximately 5 to 10
> 0,01 to 0,05	100	10 to 50
> 0,05 to 0,1	50	25 to 50

Table 1 *(continued)*

Approximate ash %	Test portion g	Mass of ash obtained mg
> 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 to 25	2	200 to 500
> 25	1	> 250

For plastics yielding very low ash, it is necessary to use larger 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 (6.1) in convenient amounts for a succession of burnings until the whole of the test portion has been burnt.

7.2 Test conditions

Calcination shall be continued to constant mass as defined in 7.3.6.

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 that allow the attainment of constant mass preferably in less than 3 h. A higher temperature or the use of sulfation generally shortens the duration of the calcination.

Whichever method — A, B, C or D — 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 or other suitable means for ventilation for the ashing procedure, if not required otherwise by the furnace being used (e.g. for furnaces with an integrated exhaust system that shall not be operated in a fume cupboard).

For method A, if it can be conclusively demonstrated for a particular sample type that direct ashing in a muffle furnace without preheating/igniting the sample over a Bunsen flame or equivalent gives the same result, then this version of method A (rapid ashing) is permitted. The use of this rapid ashing method shall be included in the test report.

NOTE The precision and reproducibility of the ashing procedure depends on the temperature at which the burning and/or calcination process in the furnace (ohmic resistance heating or microwave heating) is performed.

Efficient ventilation of the furnace is equally important, especially if the furnace is used for burning and calcination (rapid ashing method).

To ensure a constant quality of the ashing procedure, the lab shall verify the operation of the furnace at regular intervals. This verification shall include

— Calibration of the furnace temperature using a reference traceable to a national standard according to a validated calibration procedure;

— Check of the efficiency of the furnace ventilation.

NOTE Especially for plastics containing fillers that decompose under elevated temperatures, e.g. like Calcium carbonate, a precise and constant temperature in the furnace is important to ensure precise and reproducible results.