INTERNATIONAL STANDARD

ISO 3451-5

Second edition
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Plastics — Determination of ash —

Part 5: Poly(vinyl chloride)

Plastiques — Détermination du taux de cendres —

Partie 5: Poly(chlorure de vinyle)

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 3451 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3451-5 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 3451-5:1989), 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 5: Poly(vinyl chloride)

WARNING — The use of this part of ISO 3451 may involve hazardous chemicals, materials, operations or equipment. This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish proper safety and health practices and determine the application of regulatory limitations prior to use.

Poly(vinyl chloride) evolves hydrogen chloride on thermal decomposition and precautions should be taken to avoid inhalation of these or other fumes.

1 Scope

This part of ISO 3451 specifies three methods for the determination of the ash of poly(vinyl chloride). The general procedures given in ISO 3451-1 are followed. For ash, method A is used. For sulfated ash, methods B and C are used. All three methods are applicable to resins, compounds and finished products. Methods B and C are applicable when lead-containing compounds are present.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 3451. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this part of ISO 3451 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Principle

3.1 Method A (direct calcination)

The organic matter in a test portion is burnt off and the residue is heated at 950 °C until constant mass is reached.

3.2 Method B (calcination, with sulfuric acid treatment after combustion)

The organic matter in a test portion is burnt off, the residue is converted into sulfates using concentrated sulfuric acid and, finally, the residue is heated at 950 °C until constant mass is reached.

3.3 Method C (calcination, with sulfuric acid treatment before combustion)

The organic matter in a test portion is burnt off after adding concentrated sulfuric acid and the residue is heated at 950 °C until constant mass is reached. This method is recommended over method B because of the better reproducibility of the results.

Method B or method C should be used if lead-containing compounds are present.

4 Reagents (for methods B and C only)

4.1 **Sulfuric acid**, density 1,84 g/ml, analytical grade.

4.2 **Acetic acid**, 100 %, analytical grade.

WARNING — Care should be taken in handling sulfuric acid and acetic acid.

5 Apparatus

Apparatus as specified in ISO 3451-1, and in particular:

5.1 **Crucible of silica**, **platinum** or **porcelain with lid**, diameter of upper part 45 mm to 75 mm, height equal to the diameter. The size shall be such that the crucible is not more than half-filled by the test portion.

5.2 **Bunsen burner**, with silica triangle and tripod, or other suitable heating device.

5.3 **Muffle furnace** or **microwave furnace**, capable of being controlled thermostatically at 950 °C ± 50 °C.

5.4 **Pipettes**, of appropriate capacity (for methods B and C only).

WARNING — In 7.3.4 and 7.4.3, it is required to use a pipette for the addition of concentrated sulfuric acid. The acid must be introduced into the pipette using a suitable device (e.g. a rubber-bulb pipette filler) and must never be sucked up by mouth.

5.5 **Desiccator**, containing an effective drying agent that does not react chemically with the ash components.

NOTE In some cases, the affinity of ash for water can be greater than that of drying agents commonly used.

5.6 **Analytical balance**, accurate to 0,1 mg.

5.7 **Weighing bottles**.

6 Safety precautions

6.1 Always wear safety glasses when working in the laboratory.

6.2 Exercise all normal safety precautions when working with an open flame or at high temperatures. Use insulated gloves and long crucible tongs when putting samples in the muffle furnace or taking them out of it.

6.3 Carry out the heating of the sample in a fume cupboard, and for calcination use an appropriately vented muffle furnace.

6.4 Read carefully and follow strictly the instructions in the warnings at the beginning of the text and in clause 4 and subclauses 5.4 and 7.4.3.

7 Procedure

7.1 Test portion

The recommended test portion size is given in Table 1.

Table 1 — Mass of test portion

Sample	Test portion g
Resin	5
Dry blends or pellets, products with a filler content $>$ 10 %	2
Dry blends or pellets, products with no filler or with a filler content $\leqslant$ 10 %	5

7.2 Method A (determination of unsulfated ash)

7.2.1 Heat the clean crucible and lid (5.1) in the muffle furnace (5.3) at 950 °C ± 50 °C for 10 min and cool in the desiccator (5.5) to room temperature. Weigh the crucible with lid to the nearest 0,1 mg.

7.2.2 Introduce into the crucible a suitable quantity of sample (see Table 1) (finished product samples should preferably be cut into small pieces). Weigh the crucible with lid and test portion to the nearest 0,1 mg and calculate the mass of the test portion (m_0).

7.2.3 Heat the crucible directly on the heating device (5.2) so that the test portion burns slowly and loss of ash is avoided. Continue this operation until no more smoke is evolved.

In the event of violent burning, the test portion may be added in successive portions.

7.2.4 Partially close the crucible with the lid so that the volatile matter evolved does not carry away particles of ash. Place the crucible at the entrance of the muffle furnace maintained at 950 °C ± 50 °C (the temperature in the entrance zone is about 300 °C to 400 °C), then advance the crucible slowly into the furnace. Calcinate for 30 min at 950 °C ± 50 °C.

It is recommended that the design of the lid be such that, when placed on the crucible, the lid fits well but does not close the crucible completely.

7.2.5 Remove the crucible with lid from the furnace, place it in the desiccator, allow to cool to room temperature and weigh to the nearest 0,1 mg (m_1).

7.2.6 Calcinate again, under the same conditions, until constant mass is reached; i.e. until the results of two consecutive weighings do not differ by more than 0,5 mg. The total duration of heating in the furnace shall however not exceed 3 h. If constant mass is not attained after this time, the mass after 3 h shall be used for calculating the test results.

7.3 Method B (determination of sulfated ash)

7.3.1 Proceed as detailed in 7.2.1.

7.3.2 Proceed as detailed in 7.2.2.

7.3.3 Proceed as detailed in 7.2.3.

7.3.4 After allowing the crucible and contents to cool, add concentrated sulfuric acid (4.1) dropwise by means of a pipette (5.4) of suitable capacity until the residue is soaked completely. Heat carefully on a suitable heating device (5.2) until the evolution of smoke ceases, taking care to avoid spattering of the contents of the crucible.

7.3.5 If, after allowing the crucible to cool, carbon is still evident, add 1 to 5 drops of sulfuric acid and reheat until evolution of white fumes has ceased.

7.3.6 Place the crucible at the entrance of the muffle furnace (5.3) maintained at 950 °C ± 50 °C and proceed as detailed in 7.2.4, 7.2.5 and 7.2.6. The residue after calcination shall be grey or white, but not black.

7.4 Method C (determination of sulfated ash)

7.4.1 Proceed as detailed in 7.2.1.

7.4.2 Proceed as detailed in 7.2.2.

7.4.3 Using a pipette, add drop by drop the minimum possible quantity of concentrated sulfuric acid (4.1) necessary to wet the test portion evenly. Close the crucible with the cover and heat on the heating device. Repeat this operation until cracking and carbonization are over.

In cases where the sulfuric acid tends to creep over the edge of the crucible or where, despite precautions, some of the test portion tends to be lost from the crucible by violent reaction, the concentrated sulfuric acid may be replaced by a mixture of concentrated acetic acid and concentrated sulfuric acid. The use of this mixed acid shall be agreed between the interested parties, however, and reference made to it in the test report.

WARNING — Carbonization is required before ignition since explosive combustion takes place if the crucible is put in the furnace immediately after addition of the sulfuric acid.

Care should be taken in preparing and handling the mixture of concentrated acetic acid and concentrated sulfuric acid.

7.4.4 Proceed as detailed in 7.2.4.

7.4.5 Proceed as detailed in 7.2.5.

7.4.6 Proceed as detailed in 7.2.6.

8 Number of determinations

Carry out two determinations. Calculate the arithmetic mean of the results. If the individual test results differ by more than 5 % of their mean, repeat the procedure until two successive results do not differ from each other by more than 5 % of their mean.

9 Expression of results

The unsulfated ash content (method A) or sulfated ash content (methods B and C), expressed in grams per 100 g of sample, is given by the formula

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass of the test portion, in grams;

m_1 is the mass of ash obtained, in grams.

10 Accuracy and precision

The accuracy and precision of these methods are not known as interlaboratory data are not available. Because of the wide range of formulations containing poly(vinyl chloride), it is not possible to give specific limits covering all of them.

11 Test report

The test report shall contain the following particulars:

a) a reference to this part of ISO 3451;

b) all details necessary for complete identification of the sample, including type, manufacturer's code number, source, trade name, etc.;

c) the method used (method A, B or C);

d) the mass of each of the two test portions used;

e) the individual results of the two determinations and the mean ash content;

f) if constant mass was not attained after a total time of 3 h, report that fact (see 7.2.6);

g) if a mixture of concentrated sulfuric acid and concentrated acetic acid was used for method C, report the ratio of the acids in the mixture (see 7.4.3);

h) the date of the test.