INTERNATIONAL STANDARD

ISO 3451-5

First edition
1989-09-15

Plastics — Determination of ash —

Part 5 :
Poly(vinyl chloride)

Plastiques — Détermination du taux de cendres —
Partie 5 : Poly(chlorure de vinyle)

Reference number
ISO 3451-5:1989(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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3451-5 was prepared by Technical Committee ISO/TC 61, *Plastics*.

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)*

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Plastics — Determination of ash —

# Part 5 :
Poly(vinyl chloride)

WARNING — Poly(vinyl chloride) evolves hydrogen chloride on thermal decomposition, and precautions should be taken to avoid inhalation of fumes.

1 Scope

This part of ISO 3451 specifies two methods for the determination of the ash of poly(vinyl chloride). The general procedures given in ISO 3451-1 are followed — method A (ash) or method B (sulfated ash). These methods may be used for resins, compositions and finished products.

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 3451-1:1981, *Plastics — Determination of ash — Part 1: General methods.*

3 Principle

Method A

Direct calcination, i.e. by burning the organic matter and treating the residue at 850 °C until constant mass is reached.

Method B

Calcination, with sulfuric acid treatment after combustion, i.e. by burning the substance and transforming the residue into sulfates using concentrated sulfuric acid and, finally, heating the residue at 850 °C until constant mass is reached. Should lead compounds be present, method B is recommended.

4 Reagents (for method B only)

4.1 Sulfuric acid, ρ 1,84 g/ml, of recognized analytical grade.

5 Apparatus

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

5.1 Crucible of silica or platinum, diameter of upper part 45 mm to 75 mm, height equal to the diameter. The size shall be sufficient so that the crucible is no 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, capable of being controlled thermostatically at 850 °C ± 50 °C.

5.4 Pipette, of appropriate capacity (for method B only).

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

NOTE 1 In some cases, the affinity of the ash for water may be greater than that of drying agents commonly used.

6 Procedure

6.1 Method A — Direct calcination (determination of ash)

6.1.1 Prepare the crucible (5.1) by heating in the muffle furnace (5.3) at 850 °C ± 50 °C until constant mass is reached. Allow to cool in the desiccator (5.5) to room temperature, but for at least 1 h, and weigh to the nearest 0,1 mg.

6.1.2 Introduce into the crucible 2 g to 5 g of the sample and reweigh. Heat the crucible directly on the heating device (5.2) so that the sample burns slowly and loss of ash is avoided. Continue this operation until no more smoke is evolved.

6.1.3 Place the crucible at the entrance of the muffle furnace maintained at 850 °C ± 50 °C (the temperature in the entrance zone is about 300 °C to 400 °C), then advance the crucible slowly into the furnace. Calcine slowly (to prevent loss of ash particles) for 30 min at 850 °C ± 50 °C.

6.1.4 Remove the crucible from the furnace. Place it in the desiccator, allow to cool to room temperature, but for at least 1 h, and weigh to the nearest 0,1 mg.

6.1.5 Calcine 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 duration of heating in the furnace shall not, however, exceed 3 h; if constant mass is not attained after this time, the mass after 3 h shall be used for calculating the test result.

6.2 Method B — Calcination, with sulfuric acid treatment after combustion (determination of sulfated ash)

6.2.1 Proceed as detailed in 6.1.1 and 6.1.2.

6.2.2 After allowing the crucible and contents to cool, add 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.

6.2.3 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.

6.2.4 Place the crucible at the entrance of the muffle furnace (5.3) maintained at 850 °C ± 50 °C and proceed as detailed in 6.1.3 , 6.1.4 and 6.1.5. The residue after calcination shall be white.

7 Number of determinations

Carry out two determinations. Calculate the arithmetic mean of the results. If the individual test results differ from each other by more than 10 % of their mean, repeat the procedure until two successive results do not differ from each other by more than 10 % of their mean.

8 Expression of results

The ash or sulfated ash content, 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, in grams, of the test portion;

m_1 is the mass, in grams, of ash obtained.

9 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.

10 Test report

The test report shall contain the following particulars:

a) a reference to this part of ISO 3451;

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

c) the method used, i.e.

method A — ash at 850 °C

method B — sulfated ash at 850 °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 is not attained after 3 h, report that fact (see 6.1.5).