~~2025-01~~

ISO/FDIS 3451-5:~~2025(E)~~

ISO/TC 61/SC 5/~~WG 11~~

Secretariat: DIN

Date: 2025-01-22

Plastics — Determination of ash —

Part 5: Poly(vinyl chloride)

Plastiques — Détermination du taux de cendres —

Partie 5: Poly(chlorure de vinyle)

FDIS stage

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Published in Switzerland

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Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 3451--5:2002), which has been technically revised.

The main changes are as follows:

— ——the test conditions have been adapted to relevant fillers;

— ——the thermobalance has been added as alternative method;

— ——the results of an interlaboratory comparison have been added to justify the changed test conditions for PVC compounds filled with chalk ($CaCO_3$).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Determination of ash —

Part 5: Poly(vinyl chloride)

WARNING — The use of this document may involve hazardous chemicals, materials, operations or equipment. This document does not purport to address the safety problems associated with its use. It is the responsibility of the user of this document 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 document 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 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 3451-1, *Plastics — Determination of ash — Part 1: General methods*

ISO 11358-1, *Plastics — Thermogravimetry (TG) of polymers — Part 1: General principles*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

— IEC Electropedia: available at ~~https://www.electropedia.org/~~https://www.electropedia.org/

4 Principle

4.1 Method A (direct calcination)

The organic matter in a test portion is burnt off and the residue is heated at a specific temperature given in ~~Table 1~~Table 1 until constant mass is reached. This method may be used with a muffle or microwave furnace ~~(6.3).~~(6.3).

Table 1 — Calcination temperatures for method A

Filler system	Temperature[c] °C
Unfilled	950 ± 50 °C
Glass fibres/Glass bubbles[a]	600 ± 25 °C
Talc powder[a] and other fillers except calcium carbonate	950 ± 50 °C
Calcium carbonate[b]	1 100 ± 50 °C
Mixtures with calcium carbonate[b]	1 100 ± 50 °C

[a] The test temperature shall allow for further investigations according to filler structure.

[b] The test temperature of 1 100 °C is required for formulations containing calcium carbonate only.

[c] The specified temperature tolerances apply to the muffle or microwave furnace. For the thermobalance, the applicable temperature tolerance is ±5 °C.

4.2 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.

4.3 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. They are not recommended to be used with a thermobalance.

5 Reagents (for methods B and C only)

5.1 **Sulfuric acid**, 100 %, density 1,84 g/ml, analytical grade.

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

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

6 Apparatus

Apparatus as specified in ISO 3451-1, and, in particular, the following.

6.1 **Crucible of aluminumaluminium oxide, silica, platinum** or **porcelain**, diameter of upper part 35 mm to 75 mm. The size shall be such that the crucible is not more than half-filled by the test portion. The crucibles shall be stable in the temperature range required for measurements.

Optionally, a suitable lid may be used to cover the crucible.

For use with a thermobalance, crucibles consistent with the type of thermobalance shall be used. AluminumAluminium oxide crucibles are normally used.

6.2 **Heating device**, such as Bunsen burner, with silica triangle and tripod, or other suitable heating device (not required for use with a thermobalance).