



Standard Test Method for Ash Content in Thermoplastics¹

This standard is issued under the fixed designation D 5630; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method was developed to determine the inorganic content of thermoplastics by destructive testing techniques. Ash levels of 0.01 % or above are covered by this test method.

1.2 These ashing procedures are used only to quantify the residual solids in the polymer and are not used to identify the individual chemical components of the ash qualitatively.

1.3 This test method is limited to those materials (including glass) that are stable to 900°C.

1.4 Fluorinated polymers or any polymers containing halogenated components have not been included in these procedures.

1.5 Two different techniques for determining the inorganic residue in plastics are listed as follows:

1.5.1 *Procedure A, Muffle-Furnace Technique*—Samples to be ashed may be in powder or pellet form. This technique uses large sample sizes. Samples are flamed over a burner prior to being ashed in a muffle furnace.

1.5.2 *Procedure B, Rapid-Ash Muffle-Furnace Technique*—Samples to be ashed may be in a powder or pellet form. This technique uses sample sizes of 2 to 8 g. Samples are ignited and ashed in a muffle furnace.

NOTE 1—This test method is similar to ISO 3451/1-1981(E).

1.6 The values stated in SI units are to be regarded as the standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Section 9 for specific precautionary statements.

2. Referenced Documents

2.1 ASTM Standards:

D 883 Terminology Relating to Plastics²

D 1600 Terminology for Abbreviated Terms Relating to Plastics²

E 691 Practice for Conducting an Interlaboratory Study to

Determine the Precision of a Test Method³

2.2

ISO Standard:

ISO 3451/1-1981(E) Plastics—Determination of Ash— Part 1, General Methods, 5.3 Method A—Direct Calcination⁴

3. Terminology

3.1 Definitions—For definitions of plastics terms, see Terminologies D 883 and D 1600. There are no terms in this test method that require new or other than dictionary definitions.

4. Summary of Test Method

4.1 This test method is based on a loss in weight of a thermoplastic sample that is heated and combusted to oxidize all organic matter.

5. Significance and Use

5.1 Inorganic residues from thermoplastics ashing may be antiblock, fillers, or catalyst residues. The quantitative amounts of each are important variables of the manufacturing process.

6. Interferences

6.1 A flame height of over 2.5 cm is likely to cause a loss of fine particles.

6.2 Large sample sizes (Note 4) could result in the evolution of pyrolysis products that could affect the ash recovery.

6.3 Furnace doors must be in the closed position during the ignition period to prevent too-rapid oxidation and combustion of the sample (Note 6).

6.4 Ensure that all crucibles are cooled to ambient temperature before weighing.

7. Apparatus

7.1 *Balance*—This shall be a laboratory balance having the capability to weigh the sample to the nearest 0.0001 g. The balance should be checked periodically to ensure accuracy.

7.2 *Crucibles*—Porcelain or quartz-fiber, of sufficient size.

NOTE 2—Coors porcelain crucibles, or CEM quartz-fiber crucibles No. 303040,⁵ or equivalents, may be used.

7.3 *Muffle Furnace*—Electric-resistance-heated or

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁵ Porcelain crucibles are available from catalogs such as Fisher. CEM quartz-fiber crucibles are available from CEM Corp., Matthews, NC.

microwave-heated furnace, capable of maintaining a temperature of $900 \pm 25^\circ\text{C}$.

NOTE 3—If an electric muffle furnace is used, it should be positioned in a well-ventilated hood. If a microwave furnace is used, it should be positioned adjacent to the hood and the exhaust tube vented into the hood to prevent breathing of the byproducts of the combustion. A microwave furnace should be capable of providing an air flow of $2.8 \text{ m}^3/\text{min}$ to be moved through the microwave cavity.

7.4 *Cooling Assembly*—A nichrome triangle is positioned over a polypropylene funnel connected to a dry compressed-air source, or equivalent. See Fig. 1, Procedure B. Calibrate the air flow with a wet test meter or bubblemeter to yield a flow rate of approximately 30 L/min.

7.5 *Burner*.

7.6 *Desiccator*.

8. Reagents and Materials

8.1 *Desiccant*—Materials suitable for use in the desiccator may be chosen from the following:

8.1.1 *Anhydrous Calcium Sulfate*.

8.1.2 *Silica Gel*.

9. Safety Precautions

9.1 Always wear safety glasses when working in the laboratory.

9.2 Exercise all normal safety precautions when working with open flames and high temperatures. Use insulated gloves and long crucible tongs when taking samples out of a muffle furnace or putting them into it.

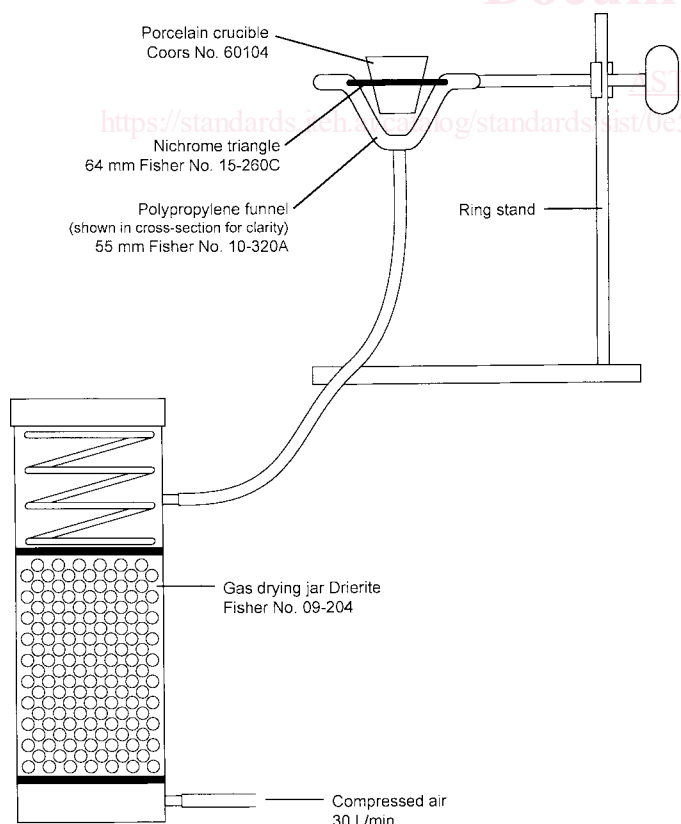


FIG. 1 Cooling Assembly

9.3 Always work with an appropriately vented muffle furnace or under a fume hood when ashing. Irritant fumes are given off by the polymer during ashing.

10. Sampling, Test Specimens, and Test Units

10.1 For hygroscopic materials such as nylons, etc., dry sample specimens to constant weight before testing. (See Table and Table)

11. Preparation and Apparatus

11.1 *Crucible Preparation*—Clean the porcelain crucibles with tap water and an abrasive detergent. Ensure that any baked-in residue is removed from the crucibles completely. Quartz-fiber crucibles are disposable items and do not require cleaning.

11.2 Fire (condition) the clean crucibles in a muffle furnace at the set operating temperature for 5 min to burn off any residue in the crucibles.

11.3 After firing (conditioning) the crucibles, place them in a desiccator to cool for 20 to 30 min (porcelain crucibles) or 2 min (quartz-fiber crucibles). Handle the crucibles with tongs only. Do not use gloves or cloth to hold the crucibles at any time.

12. Calibration and Standardization

12.1 Calibrate the analytical balances in accordance with the manufacturer's procedure.

13. Procedure A—Muffle Furnace Technique

13.1 *Sample Type*—Determine the type of polymer and anticipated amount of inorganics in the polymer to be ashed. Determine the ashing temperature, ashing time, and sample size required to perform the ash analysis in accordance with Table X1.1.

13.2 *Preashing Procedure*—Weigh a clean, dry porcelain crucible to the nearest 0.0001 g. Record the resulting value as W_1 .

13.2.1 Weigh approximately the amount of material in accordance with Table X1.1 into the preweighed crucible. Record the resulting value as W_2 .

13.2.2 Place the crucible on a burner. (The diameter of the flame equals 2.5 cm, and the height of the flame equals just enough to touch the wire gauze holding the crucible.) Ensure that the material does not overflow the crucible. When the flash point of the polymer is attained (which is approximately 600°C for most thermoplastics), the polymer begins to burn. To prevent the loss of fine particles, ensure that the flame intensity appearing from the crucible is low and that the flame height is not over 2.5 cm. Continue flaming until there are no visible flames appearing from the crucible.

13.3 *Muffle Furnace*—Remove the sample from the burner, and place it in the muffle furnace (electric or microwave) set at the desired temperature in accordance with Table X1.1.

13.4 After the prescribed duration in the muffle furnace (as specified in Table X1.1), remove the sample from the muffle furnace and place it in a desiccator until cool.

13.5 Weigh the cooled crucible and record the resulting value as W_3 .

13.6 After recording the data, place the crucible into the muffle furnace at the recommended temperature. Remove the