Designation: D5630 – 06

StandardTest Method for Ash Content in Plastics[1]

This standard is issued under the fixed designation D5630; 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 plastics by destructive ashing procedures. 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 can not be 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. Test Method D2584 is recommended for unknown samples, and in instances where fusion of the inorganic portions may be of concern.

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

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

1.5.1 *Procedure A, Muffle-Furnace Technique*— For 5 – 50 gram samples. Samples are flamed over a burner prior to being ashed in a muffle furnace.

1.5.2 *Procedure B, Rapid-Ash Muffle-Furnace Technique*— For 5 – 50 gram samples. Samples are ignited and ashed in a muffle furnace.

NOTE 1—For more efficient ashing, the plastic sample should be in the form of powder or pellet.

NOTE 2—Procedure B 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:*[2]

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D2584 Test Method for Ignition Loss of Cured Reinforced Resins

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*

ISO 3451/1-1981(E) Plastics—Determination of Ash— Part 1, General Methods, 5.3 Method A—Direct Calcination[3]

3. Terminology

3.1 *Definitions*—For definitions of plastics terms, see Terminologies D883 and D1600. 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 plastic sample when combusted to oxidize all organic matter.

5. Significance and Use

5.1 Inorganic residues from plastics ashing may be antiblock, fillers, reinforcements, catalyst residues, colorants, etc. 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 5) could result in the evolution of pyrolysis products that could affect the ash recovery.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

Current edition approved Sept. 1, 2006. Published October 2006. Originally approved in 1994. Last previous edition approved in 2001 as D5630 - 01. DOI: 10.1520/D5630-06.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

*A Summary of Changes section appears at the end of this standard

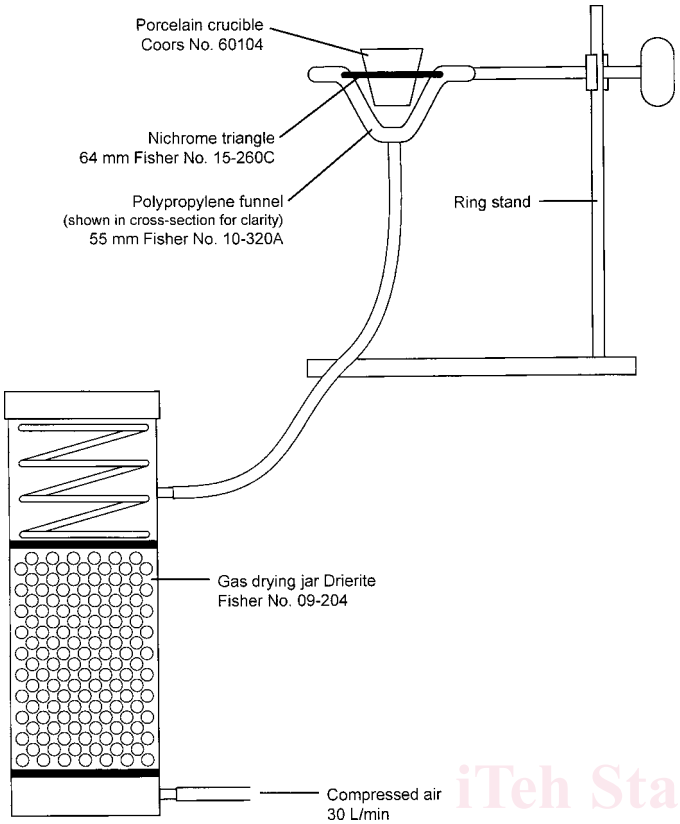

FIG. 1 Cooling Assembly

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 7).

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

7. Apparatus

7.1 *Balance*—A 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 3—Coors porcelain crucibles, or CEM quartz-fiber crucibles No. 303040,[4] or equivalents, may be used.

7.3 *Muffle Furnace*—Electric-resistance-heated or microwave-heated furnace, capable of maintaining a temperature of 900 ± 25°C.

NOTE 4—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 within or 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 m^3/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 transferring crucibles.

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.

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.

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 A1.1.

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 A1.1 into the preweighed crucible. Record the resulting value as W_2.

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