Designation: D 5630 – 01

Standard Test Method for Ash Content in Plastics[1]

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 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 D 2584 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:*

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[2]

D 2584 Test Method for Ignition Loss of Cured Reinforced Resins[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

2.2 *ISO Standard:*

ISO 3451/1-1981(E) Plastics—Determination of Ash— Part 1, General Methods, 5.3 Method A—Direct Calcination[5]

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 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.

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.

---

[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 September 10, 2001. Published November 2001. Originally published as D 5630 – 94. Last previous edition D 5630 – 94.

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

[3] *Annual Book of ASTM Standards*, Vol 08.02

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

303040,[6] 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.

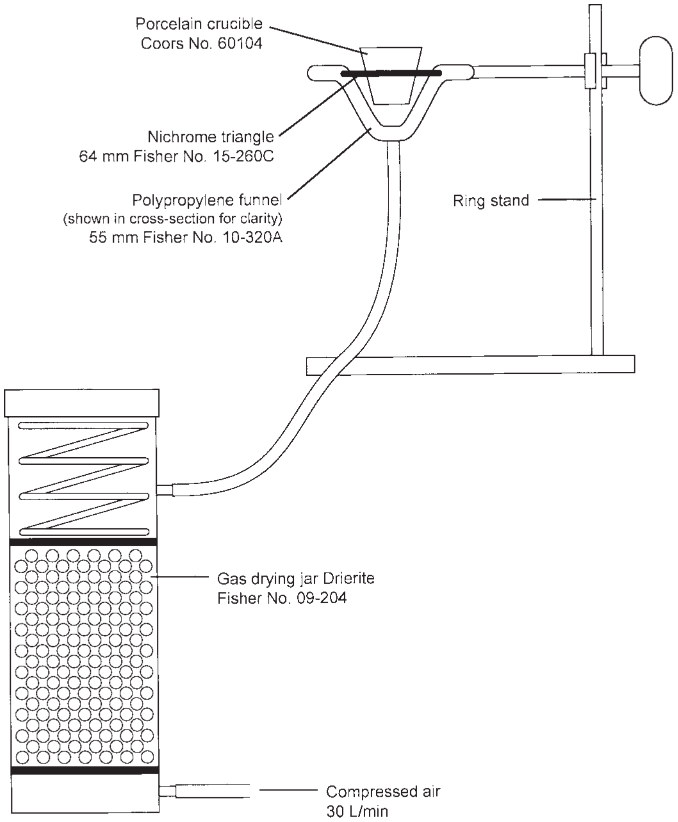

FIG. 1 Cooling Assembly

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.

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 plastics), 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 in the crucible.

13.3 *Muffle Furnace*—Using tongs, remove the crucible from the burner, and place it in the muffle furnace (electric or microwave) set at the desired temperature in accordance with Table A1.1.

13.4 After the prescribed duration in the muffle furnace (as specified in Table A1.1), using tongs, remove the crucible from the muffle furnace and place it in a desiccator until cool.

---

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

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 sample after 30 min and cool in a desiccator. Weigh the cooled crucible and record the weight. If the current weight varies more than 2 mg, continue ashing the sample at the recommended temperature until constant weight is reached.

13.7 Calculate the ash content of the sample in accordance with Equation 1.

14. Procedure B—Rapid-Ash Muffle Furnace Technique

14.1 *Sample Type*—Determine the type of polymer and anticipated amounts 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 A1.2.

14.2 *Weighing*—Weigh a clean, dry, porcelain crucible or a quartz-fiber crucible to the nearest 0.0001 g. Record the resulting value as W_1.

14.2.1 Weigh approximately the amount of material given in Table A1.2 into the preweighed crucible. Record the resulting value as W_2.

NOTE 5—Experience has shown that 2.0-g samples can be ignited consistently at 800°C without problems in a muffle furnace. Sample weights larger than 8 g are not recommended.

NOTE 6—The procedure is applicable to pelletized resins and compact products such as sheets and molded articles. Powdered materials may require modifications in the crucible size and ignition conditions due to their voluminous nature.

14.3 *Muffle Furnace*—Place the crucible containing the sample into a muffle furnace set at the desired temperature in accordance with Table A1.2 and close the muffle furnace door tightly.

NOTE 7—The door of the muffle furnace should be closed during the ignition period. If a microwave furnace is used, the ceramic door of the furnace should be maintained in a tightly closed position during the ignition period.

14.4 After the prescribed duration in the muffle furnace (as specified in Table A1.2), carefully remove the crucible from the muffle furnace and place it in a dessicator until cool.

NOTE 8—The residual ash should be free of any carbonaceous matter after the ignition period. Due to variations in furnace design and geometry, however, minor variations in ignition temperature or time, or both, may be required.

NOTE 9—If the cooling assembly shown in Fig. 1 is used to accelerate the cooling process for porcelain crucibles, position the crucible on the triangle of the cooling assembly and adjust the dry-air flow to approximately 30 L/min. Allow the crucible to cool to ambient temperature.

14.5 Weigh the cooled crucible, and record the resulting value of W_3.

14.6 After recording the data, place the crucible into the muffle furnace at the recommended temperature. Remove the crucible after 5 min, and cool in a desiccator. Weigh the cooled crucible and record the resulting value. If the current weight varies more than 2 mg, continue ashing the sample at the recommended temperature until constant weight is reached.

14.7 Calculate the ash content of the sample in accordance with Equation 1.

15. Calculation or Interpretation of Results

15.1 Calculate the ash, mass %, in the analysis sample as follows:

$$\textit{ash, mass}\ \% = \frac{(W_3 - W_1)}{(W_2 - W_1)} \times 100 \tag{1}$$

$$\textit{ppm ash, mass}\ \% = \frac{(W_3 - W_1)}{(W_2 - W_1)} \times 10^6 \tag{2}$$

where:

W_1 = mass of sample crucible, g,

W_2 = mass of sample crucible, g, plus mass of sample, g, and

W_3 = mass of sample crucible, g, plus ashed mass of sample, g.

16. Report

16.1 Report the ash content, mass %, to the second decimal (hundredth of a percent) if the value is equal to or greater than 1 %. Report the results to the nearest 10 ppm if the ash content mass percent is less than 1 % (see Equation 2).

17. Precision and Bias

17.1 *Precision*—The results from a collaborative study are given in Table 1. The data were analyzed in accordance with Practice E 691. A total of eight laboratories participated in this study of eight materials.

17.2 *Concept of r and R*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing two specimens, then:

17.2.1 *Repeatability Limit*, *r* (comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the *r* value for that material.

17.2.2 *Reproducibility Limit*, *R* (comparing two test results for the same material, obtained by different operators using different equipment in different laboratories)—The two test results should be judged not equivalent if they differ by more than the *R* value for that material.

17.2.3 Any judgment in accordance with 17.2.1 or 17.2.2 would have an approximate 95 % (0.95) probability of being correct.

TABLE 1 Precision Summary (Procedures A and B)[A]

Material/Filler	Mean,%	S_r	S_R	V_r	V_R	*r*	*R*
HDPE/antiblock	0.015	0.0038	0.0052	25.5	34.7	0.0107	0.0146
LDPE/antiblock	0.149	0.0047	0.0054	3.2	3.6	0.0132	0.0151
LDPE/antiblock	0.437	0.0047	0.0059	1.1	1.3	0.0131	0.0165
LDPE/antiblock	1.00	0.0090	0.0090	0.9	0.9	0.0253	0.0253
PET/SiO_2	3.18	0.0448	0.0448	1.4	1.4	0.1253	0.1253
PET/TiO_2	12.46	0.0461	0.0515	0.4	0.4	0.1292	0.1441
Nylon/glass	33.16	0.2715	0.2822	0.8	0.9	0.7602	0.7902
PET/TiO_2	44.81	0.3707	0.4000	0.8	0.9	1.0379	1.1201
Average				4.3	5.5		

[A] S_r is the within-laboratory repeatability and S_R is the between-laboratory reproducibility. *r* and *R* are the 95 % limits for a single sample for repeatability and reproducibility, respectively. *V*

17.3 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method, bias was not determined.

18. Keywords

18.1 antiblock; ash; mineral fillers; muffle furnace; plastics; polymer; residues; thermoplastics

ANNEX

(Mandatory Information)

A1. ASH ANALYSIS TABLES

TABLE A1.1 Procedure A

Polymer Type	Ash Content, %	Sample Size, g	Furnace Temperature, ° C	Duration, min
*	0.01–1	50	800	30
*	1–5	10	800	30
*	5–80	5	800	30
*	LDPE, LLDPE, MDPE, HDPE, PP, ionomer, EVA, EEA, thermoplastic elastomers, other olefinic polymers and copolymers, and thermosets			
**	0.01–1	50	900	30
**	1–5	10	900	30
**	5–60	5	900	30
**	PS, HIPS, ABS, SBS, SIBS, SEBS, other styrenic compounds, PC, PET, PBT, Nylon 6, Nylon 66, other polyamide resins and compounds			

TABLE A1.2 Procedure B

Polymer Type	Ash Content, %	Sample Size, g	Furnace Temperature, ° C	Duration, min
*	0.01–1	2–6	800	5
*	1–10	4	800	5
*	10 and above	2	800	5
*	LDPE, LLDPE, MDPE, HDPE, PP, ionomer, EVA, EEA, thermoplastic elastomers, other olefinic polymers and copolymers, and thermosets			
**	0.01–1 %	2–6	900	20
**	1–10 %	4	900	20
**	10 % and above	2	900	20
**	PS, HIPS, ABS, SBS, SIBS, SEBS, other styrenic compounds, and PC			
***	10 % and above	2	850	10
***	Nylon 6, Nylon 66, and other polyamide resins and compounds, PET, PBT, and other polyesters			

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 5630 – 01:

(*1*) Reference of Test Method D 2584 for use where fusion of residue is a concern, or sample is unknown.

(*2*) Converted from a test for thermoplastics to a test for plastics.

(*3*) The use of the cooling assembly described Section 7.3 is optional (Note 8).

(*4*) Appendix was changed to an Annex (mandatory requirement) as this section specifies the sample size, test temperature and test time.

(*5*) Procedure B test temperature for polyamides and polyesters changed from 900°C to 850°C based on results in a research report.

(*6*) Minor editorial changes for clarification.

(*7*) Addition of Summary of Changes section.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).