Designation: D4745 – 14

Standard Classification System and Basis for Specification for Filled Polytetrafluoroethlyene (PTFE) Molding and Extrusion Materials Using ASTM Methods[1]

This standard is issued under the fixed designation D4745; 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 standard classification system and basis for specification covers polytetrafluoroethylene (PTFE) filled molding compounds made with virgin PTFE resins defined in Specification D4894, as Types II and III.

NOTE 1—This system can be used as a model for other PTFE compounds having particulate fillers that can survive the sintering temperatures of PTFE as can those listed in this standard. This system is restricted to virgin PTFE base resin for technical reasons. Recycled or reprocessed material cannot be processed successfully.

NOTE 2—The properties measured on commercially fabricated parts may differ from the listed values for samples prepared by the procedures given in this specification, depending on part geometry and processing parameters.

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

1.3 The following statement applies to the test method portion, Section 11, of this specification:*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 Note 5 *for a specific warning statement.*

NOTE 3—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D1708 Test Method for Tensile Properties of Plastics by Use of Microtensile Specimens

D3892 Practice for Packaging/Packing of Plastics

D4894 Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System[3]

3. Terminology

3.1 *Definitions*—The terminology given in Terminology D883 is applicable to this specification unless otherwise specified.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bulk density, n*—the mass in kilograms per cubic metre of resin compound measured under the conditions of the test.

3.2.2 *filled compound, n*—blend of PTFE resin as the matrix and particulate fillers, generally glass, other inorganic, metallic, or polymeric materials that withstand the sintering temperature of PTFE (327 to 380°C).

3.2.3 *free-flow resins (pelletized), n*—generally made by treatment of finely divided resins to produce free-flowing agglomerates.

3.2.4 *lot, n*—one production run.

3.2.5 *pigmented compound, n*—a compound in which a pigment is added for colorant purposes only.

3.2.6 *standard flow resins (nonpelletized), n*—finely divided resin with an average particle size less than 100 µm.

3.3 *Abbreviations*—Abbreviations are in accordance with Terminology D1600. PTFE is the acronym for polytetrafluoroethylene.

4. Classification

4.1 This specification covers the following two types of PTFE compounds:

4.1.1 *Type I*—Standard flow resins (nonpelletized) material, for general-purpose compression molding.

[1] This specification is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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[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 ASTM International Headquarters, 100 Barr Harbor Drive, West Conshohocken, PA 19428

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

TABLE 1 TFE Compounds, Type I, Standard Flow (Nonpelletized)

Grade		Raw Resin Bulk Density, min, g/L	Molded Parts (Molded and Sintered)				
			Specific Gravity, min	Specific Gravity, max	Tensile Strength		Elongation, min, %
					min, MPa	min, psi	
1	15 % glass fiber	400	2.150	2.250	19.6	2840	220
2	25 % glass fiber	425	2.150	2.250	15.7	2270	180
3	35 % glass fiber	350	2.200	2.300	10.3	1500	150
4	5 % glass fiber and 5 % MoS_2	300	2.150	2.300	13.8	2000	200
5	15 % glass fiber and 5 % MoS_2	375	2.150	2.300	13.8	2000	150
6	10 % graphite	350	2.100	2.220	17.9	2600	120
7	15 % graphite	300	2.100	2.200	13.8	2000	100
8	25 % carbon and graphite	350	1.950	2.150	9.6	1400	20
9	32 % carbon and graphite	250	1.900	2.100	6.9	1000	20
10	40 % bronze	500	2.950	3.350	16.5	2400	100
11	60 % bronze	650	3.850	4.154	12.4	1800	50
12	55 % bronze and 5 % MoS_2	700	3.500	4.000	10.3	1500	80
13	50 % stainless steel	500	3.200	3.600	15.2	2200	120
0	As specified by customer and supplier		As specified by customer and supplier.				

4.1.2 *Type II*—Free-flow resins (pelletized) material, for compression molding, automatic molding, or ram extrusion.

4.2 Grades of each type distinguished by the nature of the filler(s) are listed in Tables 1-3.

4.3 A one-line system is used to specify materials covered by this specification. The system uses predefined cells to refer to specific aspects of this specification, as the following illustrates:

Specification				
Standard Number Block	Type	Grade	Class	Special Notes
Example: Specification D4745 – 08	II	2		

4.3.1 For this example, the line callout would be Specification D4745 – 11, Type II, Grade 2, and would specify a free-flowing (pelletized) composition of polytetrafluoroethylene that has all of the properties listed for that type, and grade in the appropriate specified properties, tables, or both, in the specification identified. A comma is used as the separator between the standard number and the type. Separators are not needed between the type, grade, and class.[4] A provision for special notes is included so that other information can be provided when required. Precede special notes, when used, by a comma.

5. Ordering Information

5.1 The filled compounds of PTFE are ordered using the type, (see 4.1) and the grade (see reference Tables 1 and 2), or they are ordered using the designation of the supplier.

6. Requirements

6.1 The PTFE compounds covered by this specification shall be uniform (filler and resin particles evenly distributed) and shall contain no foreign material.

6.2 The PTFE compounds shall conform to the requirements prescribed in Tables 1-3 when tested by the procedures specified herein. Table 1 and Table 3 list requirements for Type I. Table 2 and Table 3 reference requirements for Type II.

6.3 PTFE compounds containing high temperature polymer fillers have the potential to be varicolored or mottled in appearance. This appearance has no effect on physical properties and shall not be cause for rejection.

7. Sampling

7.1 Sampling shall be statistically adequate to satisfy the requirements of 12.4.

8. Number of Tests

8.1 Routine lot inspection tests shall consist of those carried out to determine the requirements specified in Tables 1-3 depending on type.

8.2 The requirements specified in Tables 1-3, as they apply, are sufficient to establish conformity of a material to this specification. When the number of test specimens is not stated in the test method, single determinations may be made. If more than single determinations are made on specimens from separate portions of the same sample, the results shall be averaged. The single or average result shall conform to the requirements prescribed in this specification.

9. Test Specimens

9.1 Test specimens shall be cut from a billet molded in accordance with the following procedure. An acceptable alternate procedure for molding a test plaque is described in Specification D4894, subsection 9.1.1.

9.1.1 *Safety Warning*—At normal processing temperatures, PTFE liberates harmful vapors. Provide adequate ventilation in areas where PTFE compounds are exposed to elevated temperatures. Avoid contaminating smoking materials with PTFE compounds.

9.2 *Test Preforms:*

[4] See the *ASTM Form and Style Manual*, available from ASTM Headquarters.

TABLE 2 TFE Compounds, Type II, Free-Flow (Pelletized)

Grade		Raw Resin Bulk Density, min, g/L	Molded Parts (Molded and Sintered)				
			Specific Gravity, min	Specific Gravity, max	Tensile Strength		Elongation, min, %
					min, MPa	min, psi	
1	15 % glass fiber	625	2.150	2.25	13.8	2000	200
2	25 % glass fiber	625	2.150	2.250	12.4	1800	150
3	35 % glass fiber	650	2.150	2.250	8.3	1200	100
4	5 % glass fiber and 5 % MoS_2	575	2.150	2.300	17.2	2500	170
5	15 % glass fiber and 5 % MoS_2	600	2.150	2.300	13.8	1800	120
6	10 % graphite	600	2.070	2.190	13.8	2000	150
7	15 % graphite	550	2.100	2.200	10.3	1500	60
8	25 % carbon and graphite	500	1.950	2.100	8.3	1200	20
9	32 % carbon and graphite	400	1.900	2.200	6.9	1000	20
10	40 % bronze	750	2.950	3.250	13.8	2000	85
11	60 % bronze	900	3.800	4.000	10.3	1500	20
12	55 % bronze and 5 % MoS_2	900	3.500	4.000	6.9	1000	20
13	50 % stainless steel	850	3.200	3.600	13.8	2000	100
0	As specified by customer and supplier		As specified by customer and supplier.				

TABLE 3 Required Filler Content

	Mass, %	Tolerance, ±, %
1	0 to 3	1
2	4 to 25	2
3	26 to 60	3
4	61 to 75	5

9.2.1 *Test Billet*

9.2.1.1 Prior to molding, screen the material through a 2.0-mm hand sieve, if necessary.

9.2.1.2 Preform solid test billets in a mold (see Fig. 1) having a cross-sectional area not greater than 25.8 cm^2 ≤ (4 $in.^2$) and of sufficient height to contain the sample. End plug clearance shall be sufficient to ensure escape of entrapped air during pressing. A mold length of 305 to 380 mm (12 to 15 in.) produces a billet approximately 50 to 75 mm (2 to 3 in.) long. The billet length shall not exceed 75 mm (3 in.).

9.2.1.3 Assemble the mold. Add the resin to the mold. Insert the top plug and apply hand pressure, making certain that the pusher is centered in the mold. Place the mold in a hydraulic press and remove the support ring or spacers. Increase the loading smoothly to the final preforming pressure in 3 to 5 min. Use 20.7 MPa (3000 psi) for compounds containing up to 4 % by weight filler. Use 34.5 MPa (5000 psi) for compounds containing 5 to 25 % by weight and 68.9 MPa (10 000 psi) for compounds containing 26 % or more filler. Hold under maximum pressure for 2 to 5 min. Open the press, remove the top pusher from the mold, and force the preform vertically out of the mold, using a continuous, smooth movement.

9.2.1.4 Place the preform in a sintering oven and sinter in accordance with the procedure in Table 4.

9.2.1.5 *Sectioning Test Billet:*

(1) Divide the test billet by removing a 1.6 mm (1⁄16 in.) minimum from one end of the test billet prior to preparation of the test specimens.

(2) Prepare five test specimens, 1 ± 0.25 mm (0.040 ± 0.010 in.) in thickness for the determination of tensile properties and cut a piece of suitable thickness for specific gravity measurements. This piece will be approximately cubical in shape, weighing at least 10 g. All surfaces must be smooth. Take care to avoid wedge-shape cuts.

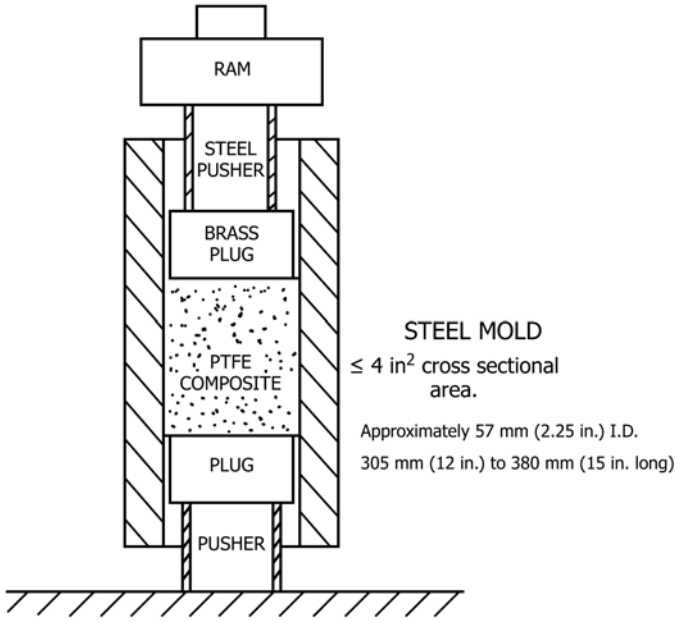


FIG. 1 Preforming of PTFE Composite Test Billet

TABLE 4 Sintering Procedures for Test Billets

Initial temperature, °C (±)[A]	Ambient
Rate of heating, °C/h (°F/h)	60 ± 5 (108 ± 9)
Hold temperature, °C (°F)	370 ± 6 (698 ± 10)
Hold time, min	240 ± 5
Rate of cooling, °C/h (°F/h)	60 ± 5 (108 ± 9)
Final temperature, °C (°F)[A]	95 ± 6 (203 ± 10)

[A] Oven can be opened safely at these temperatures.

9.2.2 *Specific Gravity (alternative molding)*

9.2.2.1 Prior to molding, screen the material through a 2.0-mm hand sieve, if necessary.

9.2.2.2 A cylindrical preforming die, 28.6 mm (1-⅛ in.) internal diameter by at least 76.2 mm (3 in.) deep or the test specimen as agreed to in X1.3 is used. End plug clearances shall be sufficient to ensure escape of air during pressing.

Molding pressures shall be followed as listed in 9.2.1.3 and sintered in accordance with Table 4.

9.2.3 Alternate mold size and molding pressure as agreed to by customer and supplier. Sinter in accordance with Table 4.

10. Test Conditions

10.1 Tests shall be conducted at 23 ± 2°C (73.4 ± 4°F). Since the resin does not absorb water, the maintenance of constant humidity during testing is not required.

11. Test Methods

11.1 *Filler Content:*

11.1.1 *Scope*—This burn-out procedure for filler content shall be used for determining the amount of a filler in a compound. The entire procedure shall be carried out in an inert atmosphere. Reaction with oxygen produces volatile products that will cause incorrect results. This procedure is not suitable for use with fillers that are not thermally stable at the maximum temperature of the method.

11.1.2 The equipment used is a tube furnace suitable for use at 680 ± 25°C (1250 ± 50°F). The furnace shall have a combustion tube and combustion boats made of materials suitable to withstand reactive decomposition gases. A system is needed to supply a constant purge of dry nitrogen, or suitable purge gas, through the furnace at a rate of 10 to 50 mL/min.

11.1.3 Scrub the pyrolysis gases with a base, such as a dilute solution of sodium hydroxide, and vent to the outside atmosphere.

NOTE 4—Vycor glass or ceramic is recommended for the combustion tube. Nickel or platinum is recommended for the combustion boats.

NOTE 5—Take care to avoid leakage of fumes into the work area. Take precautions to avoid the inhalation of, or exposure to gases from the pyrolysis since these gases may be hazardous.

11.1.4 Add approximately 2 g of the sample to each of the combustion boats, using standard quantitative laboratory weighing practices. Place boats containing the samples into the furnace. Heat and hold at 680 ± 25°C (1250 ± 50°F) until complete degradation of the PTFE has occurred. One hour at maximum temperature is typically sufficient. Calculate the percentage of filler as the net weight of the residue multiplied by 100, divided by the net weight of the original sample.

11.2 *Filler Content (Alternate Method):*

11.2.1 *Equipment*—Thermogravimetric analyzer, capable of weighing a sample as heat is applied. The analyzer shall be programmable to heat at a predetermined rate to a maximum temperature of at least 680 ± 25°C (1250 ± 50°F). A constant supply of nitrogen, or suitable purge gas, is needed to flow through the furnace. Follow the manufacturer's instructions for recommended flow rates. The crucible shall be constructed of aluminum oxide or platinum.

11.2.2 The pyrolysis gases shall be scrubbed in accordance with 11.1.3.

11.2.3 Add 50 ± 10 mg to a crucible. Place crucible and sample in furnace. Ensure purge gas is flowing through the system at the recommended rate. Program the analyzer to heat at 20°C/min ± 1°C (36°F/min ± 1.8°F) to 680 ± 25°C (1250 ± 50°F). The analyzer shall be capable of calculating the residue left after the test cycle is complete. Follow the manufacturer's instructions.

11.3 *Bulk Density:*

11.3.1 Perform the test in accordance with instructions titled *bulk density* in Specification D4894, subsection 10.2.

11.4 *Tensile Properties:*

11.4.1 *Procedure*—Using the microtensile cutting die shown in Fig. 2, cut five tensile specimens from the slices prepared as in 9.2.1.5. Determine the tensile properties in accordance with the procedures described in Test Method D1708, except the initial jaw separation shall be 22.2 ± 0.13 mm (0.875 ± 0.005 in.), and the speed of testing shall be 50 mm (2 in.)/min. Clamp the specimens with essentially equal lengths in each jaw. Determine elongation expressed as a percentage of the initial jaw separation.

11.5 *Specific Gravity:*

11.5.1 Determine the specific gravity of specimens taken from the test preforms described in 9.2.

11.5.2 Make specific gravity determinations in accordance with the procedures described in Test Methods D792. Add wetting agent to the water as appropriate to reduce surface tension and ensure complete wetting of the specimen.

12. Inspection and Certification

12.1 Inspection and certification of the material supplied with reference to a specification based on this classification system shall be for conformance to the requirements specified herein.

12.2 Lot-acceptance inspection shall be on the basis on which acceptance or rejection of the lot is made. The lot-acceptance inspection shall consist of: manufacturers' test data for specific gravity, tensile strength, and elongation.

12.3 Periodic check inspection with reference to a specification based upon this classification system shall consist of the tests for all requirements of the material under the specification. Inspection frequency shall be adequate to ensure the material is certifiable in accordance with 12.4.

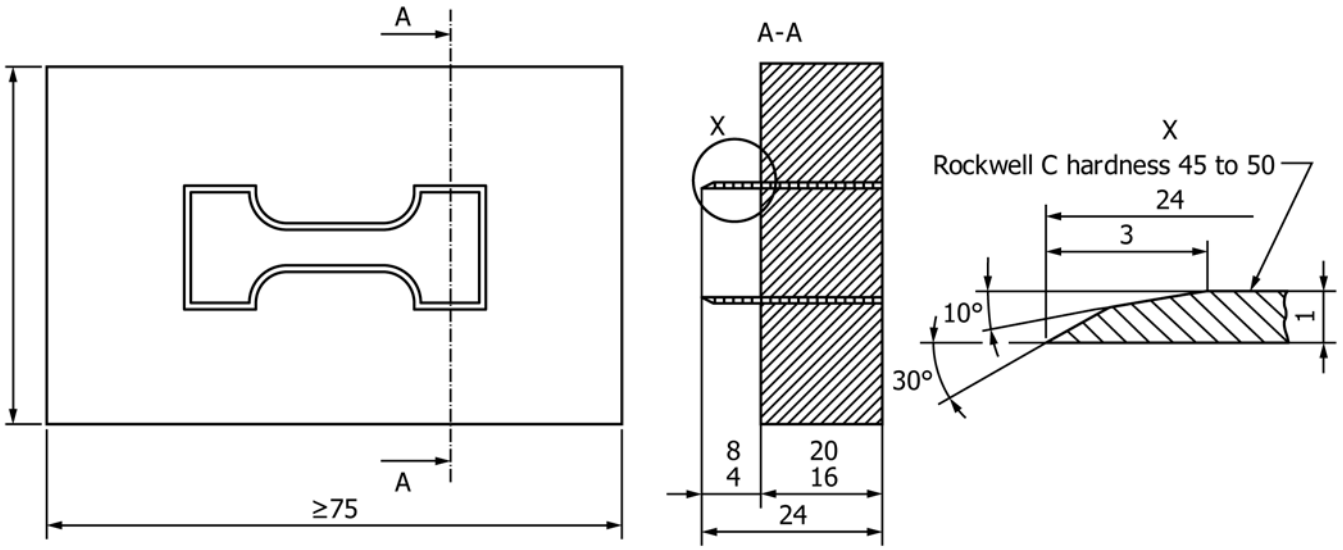
12.4 Certification shall be that the material was manufactured by a process in statistical control, sampled, tested, and inspected in accordance with this classification system, and that the average values for the lot meet requirements of the specification (line callout).

12.5 A report of test results shall be furnished when requested. The report shall consist of results of the lot-acceptance inspection for the shipment and the results of the most recent periodic-check inspection.

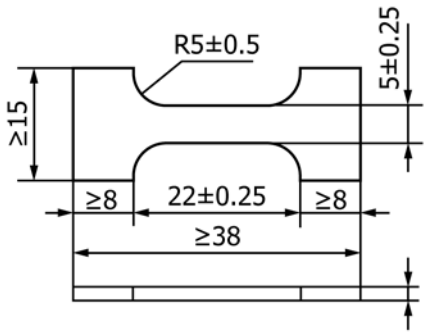
13. Packaging and Package Marking

13.1 Packaging and packing provisions shall be in accordance with Practice D3892.

13.2 *Marking*—Shipping containers shall be marked with the name of the material, and quantity contained therein, and appropriate cautionary information.



Steel-rule die
(inside dimensions for die are the same as test specimen)
Die to be sharpened on outside edge only (as shown in A-A)



Test specimen

NOTE 1—Dimensions in millimetres.

FIG. 2 Microtensile Die—ISO

14. Keywords

14.1 filled compounds; filled polytetrafluoroethylene; fluorocarbon polymers; fluoropolymer; fluoropolymer composites; polytetrafluoroethylene; PTFE

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL USEFUL TESTS

X1.1 Scope

X1.1.1 In addition to their use for specification purposes, the tests described in this specification have utility for characterizing PTFE compounds. Other useful properties of PTFE compounds are be measured by adding a few details to the specification tests. The purpose of this Appendix is to provide the details needed to determine these additional characteristics.

X1.2 Referenced Documents

X1.2.1 No additional documents to Section 2.

X1.3 Dimensional Changes During Molding (Shrinkage and Growth)

X1.3.1 Measure the inside diameter (ID) to ±0.0254 mm (0.001 in.) of the die used to make the preform in 9.2.1.2 or agreed upon between supplier and customer (mold size and preforming pressure). Measure the height of the preform if interested in growth. After the piece has been sintered and cooled to ambient temperature in accordance with Table 4, measure the diameter and height of the sintered piece.

X1.3.2 *Calculation:*

X1.3.2.1 Percent mold shrinkage = [(diameter of sintered piece/ID) - 1] × 100

X1.3.2.2 Percent preform shrinkage = [(diameter of sintered piece/diameter of preform) - 1] × 100

X1.3.2.3 Percent growth = [(height of sintered piece/height of preform) - 1] × 100

X1.3.2.4 Positive values reflect an increase in the dimension during sintering. Negative values reflect a decrease in the dimension during sintering.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4745 - 11a$^{\varepsilon 01}$) that may impact the use of this standard. (August 1, 2014)

(1) Changed title.

(2) Updated scope section.