AMERICAN SOCIETY FOR TESTING AND MATERIALS 1916 Race St. Philadelphia, Pa 19103 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM If not listed in the current combined Index, will appear in the next edition. An American National Standard

# Standard Specification for Polytetrafluoroethylene (PTFE) Molding and Extrusion Materials<sup>1</sup>

This standard is issued under the fixed designation D 1457; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense to replace L-P-403c. Consult the DoD Index of Specifications and Standards for the specific year which has been adopted by the Department of Defense.

#### 1. Scope

- 1.1 This specification covers molding and extrusion resins of polytetrafluoroethylene (PTFE) that have never been melted after preforming or molding and are normally processed by methods similar to those used in powder metallurgy or ceramics, or by ram extrusion or extrusion with a volatile aid. These PTFE resins are homopolymers of tetrafluoroethylene or copolymers containing not more than 1 % by weight of other fluoromonomers. The usual methods of processing thermoplastics generally are not applicable to these materials because of their viscoelastic properties at processing temperatures. The materials included herein do not include mixtures of PTFE resin with additives such as colorants, fillers, plasticizers, any fabricated articles, or reprocessed or reground resin. The methods and properties included are those required to identify the various types of resins. Additional procedures are provided in Appendix X1 for further characterization of the resins.
- 1.2 The values stated in SI units as detailed in Practice E 380 are to be regarded as the standard, and the practices of E 380 are incorporated herein.
- 1.3 The following precautionary caveat pertains only to the test methods portion, Section 13, of this specification: This standard does not purport to address all of the safety problems, 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.

#### 2. Referenced Documents

- 2.1 ASTM Standards:
- D618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing<sup>2</sup>
- D 638 Test Method for Tensile Properties of Plastics<sup>2</sup>
- D 792 Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement<sup>2</sup>
- D 883 Terminology Relating to Plastics<sup>2,3</sup>
- D 1505 Test Method for Density of Plastics by the Density-Gradient Technique<sup>2</sup>

- D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials<sup>4</sup>
- D 1898 Practice for Sampling of Plastics<sup>4</sup>
- D 3297 Practice for Molding and Matching Tolerances for PTFE Resin Parts<sup>5</sup>
- D 3892 Practice for Packaging/Packing of Plastics<sup>5</sup>
- D 4052 Test Method for Density and Relative Density of Liquids By Digital Density Meter<sup>6</sup>
- D4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry<sup>5</sup>
- E 11 Specification for Wire-Cloth Sieves for Testing Purposes<sup>7</sup>
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods<sup>8</sup>
- E 380 Practice for Use of the International System of Units (SI)<sup>8</sup>
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>8</sup>
- 2.2 Military Standard:9
- MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

## 3. Terminology

- 3.1 Definitions:
- 3.1.1 General—The terminology given in Terminology D 883 is applicable to this specification.
  - 3.2 Descriptions of Terms Specific to This Standard:
- 3.2.1 bulk density, (n)—the weight in grams of a volume of 1000 mL of resin measured under the conditions of the test.
- 3.2.2 extended specific gravity, (n)—the specific gravity of a specimen of PTFE material molded as described in this specification and sintered for an extended period of time, compared to the sintering time for the measurement of SSG using the appropriate sintering schedule given in this specification.
- 3.2.3 preforming, (n)—compacting powdered PTFE material under pressure in a mold to produce a solid object, called a preform, that is capable of being handled. Molding and

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 08.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 08.04.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 08.02.

<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 08.03.

<sup>&</sup>lt;sup>6</sup> Annual Book of ASTM Standards, Vol 05.03.

<sup>&</sup>lt;sup>7</sup> Annual Book of ASTM Standards, Vols 14.02, 04.01, and 04.02.

<sup>8</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>&</sup>lt;sup>9</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

compaction are terms used interchangeably with preforming for PTFE.

- 3.2.4 reground resin, (n)—that produced by grinding PTFE material that has been molded or compacted but has never been sintered.
- 3.2.5 reprocessed resin, (n)—that produced by grinding PTFE material that has been molded or compacted and sintered.
- 3.2.6 sintering, (n)—as it applies to PTFE, is a thermal treatment during which the PTFE is melted and recrystallized by cooling with coalescence occurring during the treatment.
- 3.2.7 skiving, (n)—a machining operation during which a continuous film of PTFE material is peeled from the lateral surface of a cylindrical sintered molding.
- 3.2.8 standard specific gravity (SSG), (n)—the specific gravity of a specimen of PTFE material molded as described in this specification and sintered using the appropriate sintering schedule given in this specification.
- 3.2.9 thermal instability index (TII), (n)—a measure of the decrease in molecular weight of PTFE material which has been heated for a prolonged period of time.

#### 4. Classification

- 4.1 This specification covers the following seven types of PTFE generally used for compression molding or extrusion, or both:
- 4.1.1 Type I—Granular resin used for general-purpose molding and extrusion.
- 4.1.2 Type III—Resin produced from a coagulated dispersion and normally used with a volatile processing aid. Type III resins are divided into seven grades by such characteristics as bulk density, agglomerate size, melting peak temperature, standard specific gravity, tensile properties, etc. Each grade is divided into four classes to indicate performance in the test for extrusion pressure as described in 13.8.
- 4.1.3 Type IV—Finely divided resin with an average particle size less than 100  $\mu$ m.
- 4.1.4 Type V—A modified granular resin, either finely divided or pelletized, typically used in applications requiring improved resistance to creep and stress relaxation in end use.
- 4.1.5 Type VI—Free-flowing resins. Generally made by treatment of finely divided resin to produce free-flowing agglomerates. Type VI resins are divided into three grades according to bulk density level.
- 4.1.6 Type VII—Presintered. Resin that has been treated thermally at or above the melting point of the resin at atmospheric pressure without having been previously molded or preformed.
- 4.1.7 Type VIII—Granular resin, not presintered for ram extrusion only.

Note 1—See Tables 1 and 2 for division of types by grades and classes. See footnotes to Table 1 for former classifications. Type II was deleted from this specification in an earlier edition.

### 5. General Requirements

- 5.1 The resin shall be uniform and shall contain no additives or foreign material.
- 5.2 The color of the material as shipped by the seller shall be white.

#### 6. Detail Requirements

6.1 The resins covered by this specification shall conform to the requirements prescribed in Tables 1 and 2, when tested by the procedures specified herein. Table 1 lists tests to be carried out on resins. Table 2 lists tests requiring a specimen molded as described in Section 10.

#### 7. Test Specimens

7.1 Test specimens shall be cut from disks or billets molded in accordance with the procedures given in Section 10.

#### 8. Sampling

- 8.1 Unless otherwise agreed upon between the seller and the purchaser, sample the resin in accordance with sections on the Random Sampling of Unstratified Materials, Sampling Stratified Materials, Scope of Specific Sampling Procedures, and Sampling Molded Powder in Practice D 1898. Adequate statistical sampling prior to packaging shall be considered an acceptable alternative.
- 8.2 A lot of resin shall consist of one continuous production run or a uniform blend of two or more production runs. The producer shall take (and test) sufficient within-lot samples to assure adequate in-process quality control and continuing conformance to the property requirements of this specification.

#### 9. Number of Tests

- 9.1 Unless otherwise agreed upon in writing by the purchaser and seller, routine lot inspection tests shall consist of those specified in Table 1 except melting peak temperatures. Measurement of standard specific gravity (see Table 2) is to be part of the routine lot inspection tests. Periodic tests shall consist of all the tests specified in Tables 1 and 2 and shall be made at least once per year.
- 9.2 The tests listed in Tables 1 and 2, 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 on separate portions of the same sample are made, the results shall be averaged. The single or average result shall conform to the requirements prescribed in this specification.

#### 10. Sample Preparation

- 10.1 Test Disks:
- 10.1.1 The die shown in Fig. 1 shall be used for the molding of test disks (Note 2). Place flat aluminum disks, 0.08 to 0.38 mm (3 to 15 mils) thick and 76 mm (3 in.) in diameter, on both sides of the resin when molding Type III resins.
- Note 2: Caution—Although PTFE resin can be used continuously at temperatures of 260°C (500°F) or intermittently up to 327°C (621°F), it can evolve small quantities of gaseous products when heated above 204°C (400°F). Some of these gases are harmful. Consequently, exhaust ventilation must be used whenever the resins are heated above this temperature. Since a burning cigarette would exceed 204°C (400°F), those working with PTFE resins should ensure that tobacco is not contaminated.
- 10.1.2 Screen 14.5 g (for tensile properties) or 7.25 g (for electrical properties discussed in Appendix X1.7) of PTFE

TABLE 1 Detail Requirements for Tests on Resins

Type <sup>A</sup>	Grade	Class	Bulk Density, g/L	Particle Size Average Diameter, μm	Water Content, max, %	Melting Peak Temperature		Extrusion Pressure	
						Initial °C	Second °C	MPa	psi
l <sub>B</sub>	1		700 ± 100	500 ± 150	0.04	N	327 ± 10	NAJ	NAJ
	2		$675 \pm 50$	$375 \pm 75$	0.04	N	$327 \pm 10$	NA	NAJ
ju	10	Α	475 ± 100	500 ± 150	0.04	N	$327 \pm 10$	$9.7 \pm 4.2^{\kappa}$	1410 ± 610 <sup>K</sup>
		В	$475 \pm 100$	$500 \pm 150$	0.04	N	327 ± 10	24.1 ± 10.3 <sup>L</sup>	3500 ± 1500 <sup>L</sup>
=		С	475 ± 100	$500 \pm 150$	0.04	N	327 ± 10	$55.2 \pm 20.7^{M}$	8000 ± 3000 <sup>A</sup>
i.	20	Ā	$475 \pm 100$	$500 \pm 150$	0.04	N	327 ± 10	$9.7 \pm 4.2^{\kappa}$	$1410 \pm 610^{K}$
2	_	В	$475 \pm 100$	$500 \pm 150$	0.04	N	$327 \pm 10$	24.1 ± 10.3 <sup>L</sup>	3500 ± 15004
4		Ċ	$475 \pm 100$	500 ± 150	0.04	N	$327 \pm 10$	55.2 ± 20.7 <sup>M</sup>	8000 ± 3000^
P		Ď	$475 \pm 100$	500 ± 150	0.04	N	327 ± 10	32.5 ± 17.5 <sup>M</sup>	4710 ± 25404
ĮII	3 <i>€</i>	Ā	$475 \pm 100$	425 ± 150	0.04	N	$327 \pm 10$	$9.7 \pm 4.2^{K}$	1410 ± 610 <sup>K</sup>
	•	В	475 ± 100	425 ± 150	0.04	N	$327 \pm 10$	$24.1 \pm 10.3^{L}$	3500 ± 15004
		č	475 ± 100	425 ± 150	0.04	N	327 ± 10	55.2 ± 20.7 <sup>M</sup>	8000 ± 30004
ĺ	4	Ä	475 ± 100	650 ± 200	0.04	N	327 ± 10	$9.7 \pm 4.2^{\kappa}$	1410 ± 610K
	•	В	475 ± 100	650 ± 200	0.04	N	327 ± 10	24.1 ± 10.3 <sup>L</sup>	3500 ± 15004
		č	475 ± 100	650 ± 200	0.04	N	327 ± 10	$55.2 \pm 20.7$ M	8000 ± 3000
		Ď	475 ± 100	650 ± 200	0.04	N	327 ± 10	15.6 ± 8.0 <sup>K</sup>	2170 ± 1160
	5	В	600 ± 100	500 ± 150	0.04	N	327 ± 10	24.1 ± 10.3 <sup>L</sup>	3500 ± 15004
	6	Č	475 ± 100	450 ± 150	0.04	N	327 ± 10	34.5 ± 13.8 <sup>M</sup>	5000 ± 2000
	J	Ď	475 ± 100	500 ± 150	0.04	N	327 ± 10	32.5 ± 17.5 <sup>M</sup>	4710 ± 2540
	7	B	475 ± 100	500 ± 150	0.04	N	327 ± 10	25.9 ± 10.4 <sup>L</sup>	3750 ± 1500
	,		470 ± 100	000 1 100	0.04		021 E 10	2010 12 1011	0.00 2 .000
IV <sup>F</sup>				<100	0.04	N	327 ± 10	NAJ	NAJ
Λa	1		350 ± 75	<100	0.04	N	327 ± 10	NAJ	NA
	ż		850 ± 50	500 ± 50	0.04	335	327 ± 5	NAJ	NAJ
VI	1 <i>H</i>		650 ± 150	525 ± 200	0.04	N	327 ± 10	NAJ	NAJ
	21		>800		0.04	N	327 ± 10	NAJ	NA.
	3		$580 \pm 80$	200 ± 75	0.04	N	327 ± 10	NAJ	NAJ
VII			635 ± 100	500 ± 200	0.04	327 ± 10	327 ± 10	NAJ	NA
VIII			600 ± 100	900 ± 100	0.04	335	327 ± 5	NAJ	NAJ

^ Former Type I, Classes 1 through 5, Type II, Type IV, Classes 5, 6, and 9, and Type V, Class 2 have been deleted because they are no longer listed as commercial products.

- <sup>8</sup> Formerly Type I, Class 6.
- Formerly Type III, Class 1.
- <sup>D</sup> Formerly Type III. Class 2.
- Formerly Type III, Class 3.
- F Formerly Type IV, Classes 1 through 4.
- <sup>G</sup> Formerly Type V, Class 1.
- H Formerly Type IV, Class 7.
  Formerly Type IV, Class 8.
- Not applicable.
- K Tested at a reduction ratio of 100 to 1. (Reduction ratio is the ratio of the cross-sectional area of the preform to the cross-sectional area of the die.)
- La Tested at a reduction ratio of 400 to 1.
- M Tested at a reduction ratio of 1600 to 1.
- N>5° above second melting peak temperature.

resin through a No. 10 hand sieve into the mold. Adjust the lower plug height so that the resin can be leveled by drawing a straightedge in contact with the across the top of the mold cavity. Insert the mold in a suitable hydraulic press and apply pressure gradually (Note 3) until a total of 13.8 MPa (2000 psi) for Type III resin or 34.5 MPa (5000 psi) for all other types is attained. After this point has been reached, hold the pressure on the disk for 3 min. Remove the disk from the die. A wax pencil may be used to write sample identification on the disk at this time.

NOTE 3—As a guide, increasing the pressure at a rate of 3.45 MPa (500 psi)/min is suggested until the desired maximum is attained.

10.1.3 Place the sintering oven in a laboratory hood (or equip it with an adequate exhaust system) and sinter preforms according to Table 3, Procedure B for Types I, III, IV, and VI, and Procedure C for Type V.

NOTE 4—Although the rate of pressure application is not critical, the cooling cycle is most important and the conditions cited in this procedure must be followed very closely. If they are not followed, the

crystallinity of the disks and the resulting physical properties will be markedly changed. Therefore, the use of a programmed oven is recommended for the most precise sintering cycle control so that the hood window may be left down during the entire sintering procedure, the latter being an important safety consideration.

10.2 Test Specimens for Standard Specific Gravity and Thermal Instability Index:

10.2.1 A cylindrical preforming mold, 28.6 (11/8 in.) internal diameter by at least 76.2 mm (3 in.) deep, is used to prepare the preforms. Clearance should be sufficient to ensure escape of air during pressing. Place flat aluminum foil disks, normally 0.013 mm (0.005 in.) thick and 28.6 mm (11/8 in.) diameter, on both sides of the resin, when molding Type III resins. The test resin should be near ambient temperature prior to molding.

Note 5—For maximum precision, the weighing and preforming operations should be carried out in a constant-temperature room at  $23 \pm 1^{\circ}$ C (73.4  $\pm$  1.8°F). The method should not be run below 22°C (71.6°F) due to the crystalline transition which leads to possible cracks in sintered