

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

This standard is issued under the fixed designation D4894; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This specification covers granular resins and test methods for polytetrafluoroethylene (PTFE) that have never been preformed or molded and are normally processed by methods similar to those used in powder metallurgy or ceramics, or by special extrusion processes. These PTFE resins are homopolymers of tetrafluoroethylene, or, in some cases, modified homopolymers containing not more than one percent 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 or plasticizers; nor do they include reprocessed or reground resin or any fabricated articles. The methods and properties included are those required to identify the various types of resins. Additional procedures are provided in the Appendix for further characterization of the resins.

1.2 The values stated in SI units as detailed in IEEE/ASTM SI-10 are to be regarded as the standard, and the practices of IEEE/ASTM SI-10 are incorporated herein.

1.3 The following precautionary caveat pertains only to the Specimen Preparation section, Section 9, and the Test Methods section, Section 10, of this specification: *This specification 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 Notes 3 and 9 for specific cautionary statements.

Note 1—Information in this specification is technically equivalent to related information in ISO 12086-1 and ISO 12086-2.

## 2. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- D618 Practice for Conditioning Plastics for Testing
- D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D883 Terminology Relating to Plastics
- D1708 Test Method for Tensile Properties of Plastics by Use of Microtensile Specimens
- D1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials
- D3295 Specification for PTFE Tubing, Miniature Beading and Spiral Cut Tubing
- D3892 Practice for Packaging/Packing of Plastics
- D4441 Specification for Aqueous Dispersions of Polytetrafluoroethylene
- D4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry
- D4745 Classification System and Basis for Specification for Filled Polytetrafluoroethlyene (PTFE) Molding and Extrusion Materials Using ASTM Methods
- D4895 Specification for Polytetrafluoroethylene (PTFE) Resin Produced From Dispersion
- E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System
- 2.2 ISO Standards:<sup>3</sup>
- ISO 12086-1 Plastics—Fluoropolymer Dispersions and Moulding and Extrusion Materials—Part 1: Designation

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.



TABLE 1 Detail Requirements for Tests on Resins<sup>A</sup>

Туре	Grade	Bulk Density, g/L	Particle Size, Average Diameter, µm	Water Content, max, %	Melting Peak Temperature	
					Initial °C	Second °C
I	1	700 ± 100	500 ± 150	0.04	А	327 ± 10
	2	675 ± 50	375 ± 75	0.04	Α	327 ± 10
11			<100	0.04	Α	327 ± 10
111	1	400 ± 125	<100	0.04	Α	327 ± 10
	2	850 ± 100	$500 \pm 150$	0.04	Α	327 ± 10
IV	1	650 ± 150	550 ± 225	0.04	А	327 ± 10
	2	>800		0.04	A	327 ± 10
	3	580 ± 80	200 ± 75	0.04	Α	327 ± 10
V		635 ± 100	$500 \pm 250$	0.04	327 ± 10	327 ± 10
VI		650 ± 150	800 ± 250	0.04	Α	327 ± 10

 $^{A}$  >5°C above the second melting peak temperature.

## System and Basis for Specification ISO 12086-2 Test Methods for Fluoropolymers

#### 3. Terminology

3.1 *Definitions*:

3.1.1 The terminology given in Terminology D883 is applicable to this specification.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 *bulk density*—the mass (in grams) per litre of resin measured under the conditions of the test.

3.2.2 *extended specific gravity (ESG)*—the specific gravity of a specimen of PTFE material molded as described in this specification and sintered (g.v.) for an extended period of time, compared to the sintering time for the measurement of standard specific gravity (SSG), using the appropriate sintering schedule given in this specification.

3.2.3 *lot, n*—one production run or a uniform blend of two or more production runs.

3.2.4 *preforming*—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 compaction are terms used interchangeably with preforming for PTFE.

3.2.5 *reground resin*—that produced by grinding PTFE material that has been preformed but has never been sintered.

3.2.6 *reprocessed resin*—that produced by grinding PTFE material that has been both preformed and sintered.

3.2.7 *sintering*—as it applies to PTFE, a thermal treatment during which the PTFE is melted and recrystallized by cooling with coalescence occurring during the treatment.

3.2.8 *skiving*—a machining operation during which a continuous film of PTFE material is peeled from the lateral surface of a cylindrical sintered molding.

3.2.9 *standard specific gravity (SSG)*—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.

3.2.10 *thermal instability index (TII)*—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 six types of PTFE generally used for compression molding or ram extrusion, or both:

4.1.1 *Type I*—Resin used for general-purpose molding and ram extrusion.

4.1.2 *Type II*—Finely divided resin with an average particle size less than 100 micrometres.

4.1.3 *Type III*—Modified resins, either finely divided or free-flowing, typically used in applications requiring improved resistance to creep and stress-relaxation in end-use.

4.1.4 *Type IV*—Free-flowing resins. Generally made by treatment of finely divided resin to produce free-flowing agglomerates.

4.1.5 *Type V*—Presintered. Resin that has been treated thermally at or above the melting point of the resin at atmospheric pressure without having been previously preformed.

4.1.6 *Type VI*—Resin, not presintered, but for ram extrusion only.

Note 2—See Tables 1 and 2 for division of Types by Grades, and footnotes to Tables 1 and 2 (and Table X2.1 in Appendix X2.) for former classifications.

4.2 A line callout system is used to specify materials in this standard. The system uses predefined cells to refer to specific aspects of this specification, illustrated as follows:

Specification						
Standard Number Block	: Туре	: Type : Grade		:	Special notes	
:	:	:	:		:	
Example: ASTM D4894 – 04		2				

For this example, the line callout would be ASTM D4894 – 04, III2, and would specify a granular polytetrafluoroethylene that has all of the properties listed for that Type and Grade in the appropriate specified properties, Tables, or both, in this specification. In this case there is no Class item so the cell position for class is left blank. A comma is used as the separator between Standard Number and Type. Separators are



TABLE 2 Detail Requireme	nts for Tests	on Molded	Specimens
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Туре	Grade	Thermal Instability Index, max	Standard Specific Gravity		Tensile Strength, min		Elongation at break min %
			min	max	MPa	psi	
I	1	50	2.13	2.18	13.8	2000	140
	2	50	2.13	2.18	17.2	2500	200
П		50	2.13	2.19	27.6	4000	300
111	1	50	2.14	2.22	28.0	4060	450
	2	50	2.14	2.18	20.7	3000	300
IV	1	50	2.13	2.19	25.5	3700	275
	2	50	2.13	2.19	27.6	4000	300
	3	50	2.15	2.18	27.6	4000	200
V		NA <sup>A</sup>	NA <sup>A</sup>	NAA	NAA	NAA	NA <sup>A</sup>
VI <sup>B</sup>		NAA	NAA	NAA	NAA	NAA	NAA

<sup>A</sup> NA: Not Applicable by molding techniques included in this specification.

<sup>B</sup> Extrusions of this resin show different degrees of clarity from the others.

not needed between Type, Grade, and Class.<sup>4</sup> Provision for Special Notes is included so that other information will be provided when required. An example would be in Specification D3295 where dimensions and tolerances are specified for each AWG size within Type and Class. When Special Notes are used, they shall be preceded by a comma.

#### 5. Mechanical Properties

5.1 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 specimens molded as described in Section 9.

#### 6. Other Requirements

6.1 The resin shall be uniform and shall contain no additives or foreign material.

6.2 The color of the material as shipped by the seller shall be white.

### 7. Sampling

7.1 Sampling shall be statistically adequate to satisfy the requirements of 11.4

## 8. Number of Tests

8.1 Lot inspection shall include tests for bulk density, particle size and standard specific gravity. Periodic tests shall consist of all the tests specified in Tables 1 and 2 and shall be made at least one per year.

8.2 The tests listed in Tables 1 and 2, as they apply, are sufficient to establish conformity of a material to this specification. One set of tests specimens as prescribed in Section 7 shall be considered sufficient for testing each sample. The average of the results for the specimens tested shall conform to the requirements of this specification.

#### 9. Specimen Preparation

9.1 Test Disks:

9.1.1 Use the die shown in Fig. 1 for the molding of test disks. The test resin shall be near ambient temperature prior to molding (Note 5). Warning—PTFE 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, as they are during the sintering operations that are a part of this specification. Since the temperature of burning tobacco exceeds 204°C (400°F), those working with PTFE resins should ensure that tobacco is not contaminated.

9.1.2 Screen 14.5 g (for tensile properties) or 7.25 g (for electrical properties discussed in Appendix X1.7) of PTFE resin through a No. 10 hand sieve into the die. Adjust the lower plug height to allow the resin in the die can be leveled by drawing a straightedge in contact with the top of the die across the top of the die cavity. Insert the die in a suitable hydraulic press and apply pressure gradually (Note 3) until a total of 34.5 MPa (5000 psi) is attained. Hold this pressure for 3 min. Remove the disk 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 pressure is attained.

9.1.3 Sinter the preforms in accordance with Table 3 (Note 4).

9.1.3.1 Use Procedure B for Types I, II and IV and Procedure C for Type III.

Note 4—Although the rate of heating application is not critical, the cooling cycle is most important and the conditions cited in these procedures 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 will be left down during the entire sintering procedure, the latter being an important safety consideration.

9.2 Test Specimens for Standard Specific Gravity (SSG) and Extended Specific Gravity (ESG):

9.2.1 A cylindrical preforming die, 28.6 mm ( $1\frac{1}{8}$  in.) internal diameter by at least 76.2 mm (3 in.) deep, is used to prepare the preforms. End plug clearances shall be sufficient to ensure escape of air during pressing. The test resin shall be near ambient temperature prior to molding (Note 5).

<sup>&</sup>lt;sup>4</sup> See the *Form and Style for ASTM Standards* manual available from ASTM Headquarters.

Note 5—For maximum precision, the weighing and preforming operations shall be carried out at  $23 \pm 2^{\circ}$ C (73.4  $\pm 3.6^{\circ}$ F) (the "near ambient" temperature referred to herein). These operations shall not be preformed at