Designation: D 3307 – 00

An American National Standard

Standard Specification for Perfluoroalkoxy (PFA)-Fluorocarbon Resin Molding and Extrusion Materials[1]

This standard is issued under the fixed designation D 3307; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers melt processable molding and extrusion materials of PFA-perfluoro(alkoxy alkane) fluorocarbon resin. The materials are copolymers of TFE-fluorocarbon resins containing perfluoroalkoxy side chains.

1.2 This specification does not cover recycled plastics.[2]

1.3 The tests involved are intended to provide information for identifying the materials covered. It is not the function of this specification to provide engineering data for design purposes. Specimens prepared by injection molding or extrusion could yield test results that may vary from the values in this specification.

1.4 The values stated in SI units[3] are to be regarded as the standard. The values given in parentheses are for information only.

1.5 The following precautionary caveat pertains only to the test methods portions, Sections 8 and 9 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.*

NOTE 1—This specification, ISO 12086–1(1995), and ISO 12086–2(1995) differ in approach or detail. Data obtained using either may not be technically equivalent.

2. Referenced Documents

2.1 *ASTM Standards:*

D 150 Test Methods for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials[4]

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[5]

D 638 Test Method for Tensile Properties of Plastics[5]

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[5]

D 883 Terminology Relating to Plastics[5]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[5]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[5]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[5]

D 3892 Practice for Packaging/Packing of Plastics[6]

D 4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry[7]

D 5033 Guide for the Development of Standards Relating to the Proper Use of Recycled Plastics[7]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[8]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[8]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[8]

2.2 *ISO Standards:*

ISO 12086–1 Plastics–Fluoropolymer Dispersions and Moulding and Extrusion Materials–Part 1[9]

ISO 12086–2 Plastics–Fluoropolymer Dispersions and Moulding and Extrusion Materials–Part 2[9]

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology D 883.

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

4. Classification

4.1 This specification covers six types of PFA-fluorocarbon resins supplied in pellet form for molding and extrusion.

4.2 A one-line system may be used to specify materials covered by this specification. The system uses predefined cells

[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 (Section D20.15.12).

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[2] As defined in Guide D 5033.

[3] As defined in IEEE/ASTM SI 10.

[4] *Annual Book of ASTM Standards*, Vol 10.01.

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

[6] *Annual Book of ASTM Standards*, Vol 08.02.

[7] *Annual Book of ASTM Standards*, Vol 08.03.

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

[9] Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

to refer to specific aspects of this specification, as illustrated as follows:

Specification				
Standard Number Block	Type	Grade	Class	Special Notes
Example: Specification D 3307 – 98,	I			

In this standard, the only specifications are type; no grade or class is required. A comma is used as the separator between the standard number and the type. Separators are not needed between the type, grade, and class.[10] Provision for special notes is included so that other information can be provided when required. An example would be in Specification D 3295 where dimensions and tolerances are specified for each AWG size within type and class. When special notes are used, they should be preceded by a comma.

5. General Requirements

5.1 The materials shall be of uniform composition and so prepared as to conform to the requirements of this specification.

5.2 The materials described in this specification shall be free of foreign matter to such a contamination level as may be agreed upon between the purchaser and the seller.

6. Detail Requirements

6.1 The materials covered by this specification shall conform to the requirements prescribed in Table 1 and Table 2 when tested by the procedures specified herein. Table 2 lists those tests requiring a specimen molded as described in 9.1.

7. Sampling

7.1 Sampling shall be statistically adequate to satisfy the requirements of 10.4.

8. Number of Tests

8.1 One set of test specimens as prescribed in Section 9 shall be considered sufficient for testing each sample. The average result of the specimens tested shall conform to the requirements of this specification.

9. Test Methods

9.1 *Test Specimens*:

9.1.1 Prepare a molded sheet 1.5 ± 0.25-mm (0.060± 0.010-in.) thick. Use a picture-frame-type chase having a suitable blanked-out section and thickness to produce the desired sheet. Use clean aluminum foil, 0.13 to 0.18 mm (0.005 to 0.007 in.) thick, in contact with the resin. A high temperature mold release agent may be sprayed on the aluminum foil to help prevent the foil from sticking to the sheet. Use steel molding plates at least 1.0 mm (0.040 in.) thick and of an area adequate to cover the chase.

9.1.2 Lay down and smoothly cover one plate with a sheet of aluminum foil. Place the mold chase on top of this assembly. Place within the mold chase sufficient molding material to produce the required sheet in such manner that the polymer charge is a mound in the middle of the chase. Place a second sheet of aluminum foil on top of the granules and add the top mold plate. Place the assembly in a compression molding press having platens that have been heated to 380 ± 5°C (716 ± 10°F).

9.1.3 Bring the press platens to incipient contact with the mold assembly. Hold for 2 to 4 min without pressure. Apply approximately 1 MPa (145 psi) and hold for 1 to 1.5 min. Then apply 2 to 4 MPa (290 to 580 psi) and hold for 1 to 1.5 min. Maintain the press at 380 ± 5°C (716 ± 10°F) during these steps. Remove the assembly from the press and place between two 20 ± 7-mm (0.75 ± 0.25-in.) steel plates whose temperature is less than 40°C (104°F).

9.1.4 When the sheet is cool enough to touch (about 50 to 60°C (122 to 140°F)), remove the aluminum foil from the sheet. (If the sheet is allowed to cool to room temperature, the aluminum foil cannot be pulled free.)

9.2 *Conditioning*:

9.2.1 For tests of specific gravity, tensile properties, and electrical properties, condition the molded test specimens in accordance with Procedure A of Practice D 618 for a period of at least 4 h prior to test. The other tests require no conditioning.

9.2.2 Conduct tests at the Standard Laboratory Temperature of 23 ± 2°C (73.4 ± 3.6°F) for determination of specific gravity, tensile properties, and electrical properties only. Since the resin does not absorb water, the maintenance of constant humidity during testing is not necessary. Conduct tests for melt flow rate and melting endotherm under ordinary laboratory conditions.

9.3 *Melt Flow Rate*—Determine the melt flow rate in

[10] See *ASTM Form and Style Manual.*

TABLE 1 Detail Requirements for Test on Molding and Extrusion Materials

	Type I	Type II	Type III	Type IV	Type V	Type VI
Melt flow,[A] g/10min:						
min	>7	1	>3	>10	1	>3
max	18	3	7	30	3	10
Melting endotherm peak,[B] min, °C	300	300	300	285	285	285

[A] See 9.3 of this specification.
[B] See 9.4 of this specification.

TABLE 2 Detailed Requirements for Molded Specimens

	Type I	Type II	Type III	Type IV	Type V	Type VI
Specific gravity, 23°C (73.4°F):[A]						
min	2.12	2.12	2.12	2.12	2.12	2.12
max	2.17	2.17	2.17	2.17	2.17	2.17
Tensile strength, min, 23°C (73.4°F):[B]						
MPa	20.68	25	20.68	22.75	25.51	22.75
psi	3000	3625	3000	3300	3700	3300
Elongation, 23°C (73.4°F), min, %[B]	275	300	275	275	260	275
Dielectric constant, max:[C]						
10^2 Hz	2.2	2.2	2.2	2.2	2.2	2.2
10^6 Hz	2.2	2.2	2.2	2.2	2.2	2.2
Dissipation factor, max:[C]						
10^2	0.0003	0.0003	0.0003	0.0003	0.0003	0.0003
10^6	0.0005	0.0005	0.0005	0.0005	0.0005	0.0005

[A] See 9.5 of this specification.
[B] See 9.6 of this specification.
[C] See 9.7 of this specification.

accordance with Test Method D 1238, Test Method A or B, with a temperature of 372 ± 1°C and using a total load, including piston, of 5000 g. The same requirements apply for the use of corrosion-resistant alloy for the barrel lining, orifice, and piston tip.

9.4 *Melting Endotherm Peak*:

9.4.1 Determine the melting endotherm peak with a different thermal analyzer. Place a 20-mg specimen in a 4-mm tube with a thermocouple. Premelt the specimen at 325°C, and push the thermocouple into intimate contact with the melt. Cool the sample in air. Run the differential thermal analysis at 10°C/min with glass beads as a reference to a maximum temperature of 350°C. Extend straight lines down tangent to both sides of the melting endotherm, and take the temperature at which the lines intersect (peak minimum) as the melting endotherm peak.

9.4.2 *Precision*—The single instrument precision of the differential thermal analysis applied to this material is± 1.2°C (2S) as defined in Practice E 177.

9.4.3 Other thermal techniques capable of measuring the melting endotherm giving equivalent results may be used, such as Test Method D 4591.

9.5 *Specific Gravity*—Cut two specimens from the compression molded sheet and test in accordance with Test Method D 792 or Test Method D 1505. If the latter is used, the tube should have a linear gradient over the range from about 2.10 to 2.19.

9.6 *Tensile Properties*—Cut five specimens with the microtensile die shown in Fig. 1, which is exactly the same as Fig. 1 of Test Method D 1708. The die shall be of the steel-rule type of curvature of 5 ± 0.5-mm (0.20 ± 0.02-in.) type. Determine the tensile properties in accordance with the procedures described in Test Method D 638, except that the specimens used shall be as detailed above, the initial jaw separation shall be 22 ± 0.13 mm (0.866 ± 0.005 in.), and the speed of testing shall be 50 mm/min (2 in./min). Clamp the specimens with essentially equal lengths in each jaw. Determine the elongation from the chart, expressing it as a percentage of the initial jaw separation. Details appear in the Tensile Properties section of Specification D 2116.

9.7 *Dielectric Constant and Dissipation Factor*—Determine dielectric constant and dissipation factor on three specimens, each 101.6 mm (4 in.) in diameter in accordance with Test Methods D 150. Testing shall be at 10^2 Hz and 10^6 Hz.

10. Inspection and Certification

10.1 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.

10.2 *Lot*—Acceptance shall be the basis on which acceptance or rejection of the lot is made. The lot acceptance inspection shall consist of melting endotherm peak temperature and melt flow rate.

10.3 Periodic check inspection with reference to a specification based on this classification system shall consist of the test for all requirements of the material in accordance with this specification.

10.4 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 the requirements of this specification (line callout).

10.5 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.

11. Packaging and Package Marking

11.1 All packing, packaging, and marking provisions of Practice D 3892 shall apply to this specification.

12. Precision and Bias [11]

12.1 A round robin was conducted in 1985–1986 in accordance with Practice E 691, involving seven materials tested by six laboratories (see Table 3). For each material, the sheeting from which the test specimens were to be cut was obtained from one source. Using a steel rule die, one set of test specimens for each laboratory was cut by one of the laboratories. Sheeting and a duplicate die were furnished each participating laboratory and used to cut a second set of test specimens. Each test result was the average of five individual determinations. Each laboratory obtained four test results on each material, two test results each on the specimens furnished and two on the specimens cut by the laboratory doing the testing.

12.1.1 The properties used in the analysis are tensile strength and elongation at break. The stress-strain curves of the fluorocarbon polymers (but not the fluoropolymers: modified ETFE and poly(vinylidene fluoride), PVDF) are similar in shape. Data on ETFE and PVDF, therefore, were excluded from the analysis used for this precision and bias statement but are available for use in precision and bias statements and are to be included in the research report at ASTM. Based on advice from experts in statistical analysis of round-robin data, and since use of fillers is excluded in the applicable standards, information from the testing on glass-fiber filled PTFE also is not included in Table 3. In addition, the experts advised that information from the samples cut in one laboratory and tested by all the laboratories should not be included in Table 3. The data are available in the report.

NOTE 2—**Caution:** The following explanations of I_r and I_R (12.3-12.3.3) are intended only to present a meaningful way of considering the approximate precision of this specification. The data in Table 3 should not be applied rigorously to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories.

12.2 Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 12.3-12.3.3 would then be valid for such data.

12.3 *Concept of I_r and I_R*—If CV_r and CV_R have been calculated from a large enough body of data, and for test results that were averages from testing five specimens:

[11] Supporting data are available from ASTM Headquarters. Request RR:D20-1067.