



Designation: D3159 – 20

Standard Specification for Modified ETFE Fluoropolymer Molding and Extrusion Materials¹

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

1. Scope*

1.1 This specification covers molding and extrusion materials of modified ETFE fluoropolymer. The ETFE resin is a copolymer of ethylene and tetrafluoroethylene. Modified ETFE resin contains no more than 5 mole % of other fluoromonomers.

1.2 The values stated in SI units as detailed in **IEEE/ASTM SI-10** are to be regarded as standard. The values given in parentheses are for information only.

NOTE 1—Although this specification and ISO 20568-1 and ISO 20568-2 differ in approach or detail, data obtained using either are technically equivalent.

1.3 The following safety hazards caveat pertains only 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 Recycled material is not appropriate for this specification because performance requirements cannot be met with recycled material. Therefore, this specification is for virgin material only.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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. Referenced Documents

2.1 ASTM Standards:²

- D150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation
- D618 Practice for Conditioning Plastics for Testing
- D638 Test Method for Tensile Properties of Plastics
- D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D883 Terminology Relating to Plastics
- D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
- D1600 Terminology for Abbreviated Terms Relating to Plastics
- D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
- D3892 Practice for Packaging/Packing of Plastics
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- IEEE/ASTM SI-10 Use of the International System of Units (SI): The Modern Metric System

2.2 ISO Standards:

- ISO 20568-1 Plastics—Fluoropolymer Dispersions and Moulding and Extrusion Materials—Part 1
- ISO 20568-2 Plastics—Fluoropolymer Dispersions and Moulding and Extrusion Materials—Part 2

3. Terminology

3.1 Definitions:

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

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

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.

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

TABLE 1 Specific Gravity, Melting Point, and Flow Rate Requirements

| Type Grade | I | | | II | | | III | | IV | V |
|---------------------------|------|-------------------|------|------|------|------|------|------|------|------|
| | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 1 | 1 |
| Specific gravity, min: | 1.69 | 1.69 | 1.69 | 1.75 | 1.75 | 1.75 | 1.83 | 1.83 | 1.74 | 1.74 |
| Specific gravity, max: | 1.76 | 1.76 | 1.76 | 1.84 | 1.84 | 1.84 | 1.88 | 1.88 | 1.78 | 1.80 |
| Melting point, °C, min: | 255 | 255 | 250 | 220 | 220 | 220 | 218 | 218 | 220 | 250 |
| Melting point, °C, max: | 280 | 280 | 280 | 259 | 259 | 259 | 230 | 230 | 240 | 260 |
| Flow rate, g/10 min, min: | 2.0 | 8.0 | 25 | 2.0 | 10.1 | 25 | 9.0 | 25.0 | 10 | 15 |
| Flow rate, g/10 min, max: | 16.0 | 28.0 ^A | 45 | 10.0 | 19.0 | 40 | 18.0 | 35.0 | 40 | 30 |

^A Measured with 1.588-mm (0.0625-in.) orifice; other values measured with 2.095-mm (0.0825-in.) orifice.

3.1.3 Abbreviated Terms:

3.1.4 General—The abbreviated terms given in Terminology D1600 are applicable to this specification.

4. Classification

4.1 This specification covers five types of modified ETFE fluoropolymer supplied in pellet form classified according to their specific gravity. The resins of each type are divided into one to three grades according to their melt flow rate.

4.2 A one-line system shall be used to specify materials covered by this specification. The system uses predefined cells to refer to specific aspects of this specification, as illustrated as follows:

| Specification | | | | |
|-----------------|------|-------|---------|-------|
| Standard Number | Type | Grade | Special | Notes |
| Block | : | : | : | : |
| — | — | — | — | — |

Example: Specification D3159 – 95, I 2

For this example, the line callout shall be, Specification D3159 – 95,I2 and shall specify a modified ETFE fluoropolymer that has all of the properties listed for that type and grade in Tables 1 and 2. A comma is used as the separator between the standard number and the type. Separators are not needed between the type and grade.³

5. General Requirements

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

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

6. Performance Requirements

6.1 The average test result of the lot shall conform to the requirements prescribed in Tables 1 and 2 when tested by the procedures specified herein. Table 2 lists those tests requiring a specimen molded as described in Section 8.

7. Sampling

7.1 The materials shall be sampled in accordance with an adequate statistical sampling program.

8. Specimen Preparation

8.1 Prepare a molded sheet 1.5 ± 0.3 mm (0.06 ± 0.01 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 sprayed on the aluminum foil helps 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.

8.2 Lay a sheet of aluminum foil down to smoothly cover one plate. 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 300 ± 5°C (572 ± 10°F).

8.3 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 300 ± 5°C (572 ± 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).

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

9. Conditioning

9.1 For tests of specific gravity, tensile properties, and electrical properties, condition the molded test specimen in accordance with Practice D618, Condition 4/23/50. The other tests require no conditioning.

³ See the ASTM Form and Style Manual, available from ASTM Headquarters.

TABLE 2 Detail Requirements for Molded Test Specimens for All Resins in This Specification

| Type Grade | I | | | II | | | III | | IV | V |
|---|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 1 | 1 |
| Tensile strength, min, psi: | 5500 | 4400 | 5500 | 4500 | 4500 | 4000 | 4000 | 4000 | 5000 | 4000 |
| Elongation, min, %: | 275 | 200 | 300 | 300 | 300 | 350 | 350 | 350 | 350 | 350 |
| Dielectric constant, 10 ³ Hz, max: | 2.6 | 2.6 | 2.6 | 2.6 | 2.6 | 2.6 | 2.6 | 2.6 | 2.6 | 2.6 |
| 10 ⁶ Hz, max: | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 |
| Dissipation factor, 10 ³ Hz, max: | 0.0008 | 0.0008 | 0.0008 | 0.0008 | 0.0008 | 0.0008 | 0.0008 | 0.0008 | 0.0008 | 0.0008 |
| 10 ⁶ Hz, max: | 0.009 | 0.009 | 0.009 | 0.009 | 0.009 | 0.009 | 0.009 | 0.009 | 0.009 | 0.009 |

9.2 Conduct tests at the standard laboratory temperature of $23 \pm 2^\circ\text{C}$ ($73.4 \pm 3.6^\circ\text{F}$) 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 flow rate and melting endotherm under ordinary laboratory conditions.

10. Packaging and Marking

10.1 *Packaging*—The material shall be packaged in standard commercial containers so constructed as to ensure acceptance by common or other carriers for safe transportation to the point of delivery unless otherwise specified in the contract or order.

10.2 *Marking*—Shipping containers shall be marked with the name of the material, type, and quantity therein.

10.3 All packing, packaging, and marking provisions of Practice **D3892** shall apply to this specification.

11. Test Methods

11.1 Melt Flow Rate:

11.1.1 *Principle of Test Method*—Determine the flow rate at $297 \pm 1^\circ\text{C}$ using Procedure A or B or Test Method **D1238**. Use the extrusion plastometer described in Test Method **D1238** and modified in accordance with 11.1.2. The sample shall be pellets or pieces of approximately the same suitable size cut from molded or extruded forms. Strips about 6 mm wide by 76 mm long (0.25 by 3 in.) shall be conveniently handled. See Table 2 or 3 of Test Method **D1238** for the amount of charge that shall be adjusted accordingly with the melt density of ETFE being used. Usually the charge amount is 5 to 15 g. Measure the flow rate using a load of 49 N for Types I, II, and III. Collect four successive cuts for the manual method.

11.1.2 *Apparatus*—The apparatus shall consist of an extrusion plastometer, described in Test Method **D1238**, modified by use of corrosion-resistant⁴ alloy for the barrel lining, orifice, piston tip, and orifice securing device.⁵ The orifice diameter is

⁴ “Stellite” Grade No. 19, Haynes-Stellite Co., Kokomo, IN, and “Duranickel” No. 301, International Nickel Co., 67 Wall St., New York, NY 10005, have been found resistant to fluorocarbon resins. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁵ Suitable instruments are available commercially.

2.095 by 8.000 mm long (0.0825 by 0.315 in.) for all types except 1.588 by 6.070 mm (0.0625 by 0.239 in.) for Type I, Grade 2.

11.1.3 Procedure:

11.1.3.1 *Temperature Calibration*—The specified melt temperature of $297 \pm 1^\circ\text{C}$ is the temperature measured in the melt 13 mm (0.5 in.) above the orifice. This temperature shall be obtained by controlling the temperature measured in the thermometer well at approximately 305°C . Prior to making a test, set the plastometer temperature as follows: With the orifice in place, insert a standardized thermocouple (**Note 2**) through the orifice from the bottom of the plastometer to a point 13 mm (0.5 in.) above the top of the orifice. Charge 5 g of resin granules to the plastometer, compact with the piston, and wait 10 ± 0.5 min for the melt temperature to reach equilibrium. Make the necessary adjustment in the temperature controller to bring the melt temperature to $297 \pm 1^\circ\text{C}$. Repeat this calibration procedure and record the temperature versus time at 1-min intervals for the first 10 min. The polymer shall reach the 297°C temperature within 5 min. With polymer in the plastometer for an elapsed time of 10 min for each point measured, determine the melt temperature at 6.4-mm (0.25-in.) intervals from 6.4 mm to 51 mm (2 in.) above the orifice. The entire temperature profile shall be within a range of 2°C . This precision is readily obtained by proper insulation of the sides, bottom, and top of the plastometer.

NOTE 2—Suitable standards for thermocouple calibration are: lead, m.p. 327.5°C , potassium dichromate, m.p. 398.0°C , and zinc, m.p. 419.4°C .

11.1.4 *Precision and Bias*—Precision and bias for this test method are to be determined by round-robin testing.

11.2 *Specific Gravity*—Determine the specific gravity of a specimen approximately 25 by 38 mm (1 by 1.5 in.) blanked or cut from the molded plaque (see Section 8) in accordance with the procedures described in Test Methods **D792**. Add 2 drops of a wetting agent to the water in order to reduce the surface tension and ensure complete wetting of the specimen.

11.3 Melting Endotherm Peak Temperature:

11.3.1 Determine the endotherm melting peak with a differential thermal analyzer (DTA) or with a differential scanning calorimeter (DSC) operated in a DTA mode at $10^\circ\text{C}/\text{min}$ ($18^\circ\text{F}/\text{min}$).

11.3.1.1 If DTA equipment is used, refer to Test Method **D3418** except that the DTA shall be run at 10°C/min to 325°C (617°F).

11.3.1.2 With either the DTA or DSC, extend the straight lines tangent to both sides of the endotherm melting temperature. Take the temperature at which the lines intersect as the endotherm melting peak temperature.

11.3.2 *Precision*—The single instrument precision of the differential thermal analysis applied to this test method is $\pm 1.2^\circ\text{C}$ (2S) as defined in Practice **E177**.

11.4 *Tensile Properties:*

11.4.1 Cut five specimens with the microtensile die shown in **Fig. 1**. The die shall be of the steel-rule or solid metal type of curvature of $5 \pm 0.5\text{-mm}$ ($0.2 \pm 0.02\text{-in.}$) type. Determine the tensile properties in accordance with the procedures described in Test Method **D638** except that the specimens used shall be as detailed above, the initial jaw separation shall be $22.0 \pm 0.13\text{ mm}$ ($0.875 \pm 0.005\text{ in.}$), and the speed of testing shall be $50 \pm 5\text{ mm/min}$ ($2 \pm 0.2\text{ in./min}$). 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.

11.4.2 *Precision*—For tensile strength, the within-laboratory precision is $\pm 5\%$ of the average (2S), and the between-laboratory precision is $\pm 7\%$ of the average (2S). For elongation, the within-laboratory precision is $\pm 10\%$ (2S), and the between-laboratory precision is 12% (2S).⁶

11.4.3 *Bias*—No statement of bias can be prepared for this test method, since there is no absolute method for use as a comparison basis.

11.5 *Dielectric Constant and Dissipation Factor*—Determine dielectric constant and dissipation factor in accordance with Test Methods **D150**. Testing shall be done at 10^3 Hz and 10^6 Hz .

⁶ Supporting data are available from ASTM Headquarters, Request RR:D20-1067.

12. Number of Tests

12.1 One set of test specimens as prescribed in Section **11** shall be considered sufficient for testing each sample. The average result of the samples tested shall conform to the requirements of this specification.

13. Certification and Inspection

13.1 Inspection and certification of the material supplied with reference to this specification shall be for conformance to the requirements specified herein.

13.2 Lot-acceptance inspection shall be the basis on which acceptance or rejection of the lot is made. The lot-acceptance inspection shall consist of the following:

13.2.1 Melt flow rate, and

13.2.2 Melting point.

13.3 Periodic-check inspection with reference to a specification 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 **13.4**.

13.4 Certification shall be that the material was manufactured by a process in statistical control, sampled, tested and that the average values for the lot meet the requirements of the specification.

13.5 *Reports*—A report of the test results shall be furnished when requested. The report shall consist of results of the lot-acceptance inspection for the shipment.

14. Keywords

14.1 ETFE fluoropolymer; extrusion material; fluoropolymers; melt-processible fluoropolymer; molding material; recycled; tetrafluoroethylene copolymers; tetrafluoroethylene-ethylene copolymers