

Designation: D3159 - 22

# Standard Specification for Modified ETFE Fluoropolymer Molding and Extrusion Materials<sup>1</sup>

This standard is issued under the fixed designation D3159; 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 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.

### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

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 Plas-

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.

<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 (Section D20.15.12).

Current edition approved March 15, 2022. Published March 2022. Originally approved in 1973. Last previous edition approved in 2020 as D3159 - 20. DOI: 10.1520/D3159-22.

<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.

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

Type Grade —	1			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
max:	1.76	1.76	1.76	1.84	1.84	1.84	1.89	1.89	1.78	1.80
Melting point,										
°C,										
min:	255	255	250	220	220	220	218	218	220	250
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
max:	16.0	28.0 <sup>A</sup>	45	10.0	19.0	40	18.0	35.0	40	30

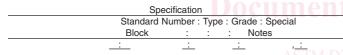
<sup>&</sup>lt;sup>A</sup> Measured with 1.588-mm (0.0625-in.) orifice; other values measured with 2.095-mm (0.0825-in.) orifice.

# 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:



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.<sup>3</sup>

# 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  $1.5 \pm 0.3$  mm  $(0.06 \pm 0.01$  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  $300 \pm 5^{\circ}\text{C}$  (572  $\pm$  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  $300 \pm 5^{\circ}\text{C}$  (572  $\pm$  10°F) during these steps. Remove the assembly from the press and place between two  $20 \pm 7$ -mm (0.75  $\pm$  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.

<sup>&</sup>lt;sup>3</sup> See the ASTM Form and Style Manual, available from ASTM Headquarters.

٧ Type Grade 2 2 2 1 3 1 3 1 1 5500 4400 5500 4500 4500 4000 4000 4000 5000 4000 Tensile strenath. min, psi: Elongation, 275 200 300 300 300 300 350 350 350 350 min, %: Dielectric constant. 103 Hz, max: 2.9 2.9 2.9 2.9 2.9 2.9 2.9 2.9 2.9 2.9 10<sup>6</sup> Hz, max: 2.9 2.9 2.9 2.9 2.9 2.9 2.9 2.9 2.9 2.9 Dissipation factor, 0.0012 0.0012 0.0012 0.0012 0.0012 0.0012 0.0012 0.0012 0.0012 0.0012 103 Hz, max: 106 Hz, max: 0.015 0.015 0.015 0.015 0.015 0.015 0.015 0.015 0.015 0.015

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

9.2 Conduct tests at the standard laboratory temperature of  $23 \pm 2^{\circ}\text{C}$  (73.4  $\pm$  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 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: a/catalog/standards/sist/4a68d87

11.1.1 Principle of Test Method—Determine the flow rate at 297  $\pm$  1°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-resistant<sup>4</sup> alloy for the barrel lining, orifice, piston tip, and orifice securing device.<sup>5</sup> The orifice diameter is

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°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  $10 \pm 0.5$  min for the melt temperature to reach equilibrium. Make the necessary adjustment in the temperature controller to bring the melt temperature to  $297 \pm 1$ °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.  $327.5^{\circ}$ C, potassium dichromate, m.p.  $398.0^{\circ}$ C, and zinc, m.p.  $419.4^{\circ}$ 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°C/min (18°F/min).

<sup>&</sup>lt;sup>4</sup> "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, <sup>1</sup> which you may attend.

<sup>&</sup>lt;sup>5</sup> Suitable instruments are available commercially.

- 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  $\pm 1.2$ °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  $5\pm0.5$ -mm ( $0.2\pm0.02$ -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  $22.0\pm0.13$  mm ( $0.875\pm0.005$  in.), and the speed of testing shall be  $50\pm5$  mm/min ( $2\pm0.2$  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.
- 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).
- 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<sup>3</sup> Hz and 10<sup>6</sup> Hz.

### 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

 $<sup>^6\,\</sup>mathrm{Supporting}$  data are available from ASTM Headquarters, Request RR:D20-1067.