Designation: D 3222 – 99

Standard Specification for Unmodified Poly(Vinylidene Fluoride) (PVDF) Molding Extrusion and Coating Materials[1]

This standard is issued under the fixed designation D 3222; 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.

1. Scope *

1.1 This specification covers melt processable molding and extrusion materials, as well as coating materials of poly(vinylidene fluoride) fluoroplastic, commonly abbreviated PVDF (or PVF_2 in scientific literature). This specification covers thermoplastic resin materials supplied in pellet or powder form.

1.2 This specification applies only to the virgin homopolymer prepared from vinylidene fluoride, not copolymers, reinforced, filled grades or special grades with additives or treatments for modification of attributes.

1.3 The tests involved are intended to provide information for specification of unmodified PVDF homopolymer resins. It is not the purpose of this specification to provide engineering data for design purposes.

1.4 PVDF fluoroplastics melt between 156 and 180°C (312 and 356°F) and are thermally stable up to about 370°C (698°F).

NOTE 1—**Warning:** Evolution of corrosive and toxic hydrogen fluoride can occur under certain conditions.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

NOTE 2—PVDF exhibits polymorphism.[2] The type and extent of crystalline structure varies with the thermomechanical history of the sample. Specimens prepared by techniques different than prescribed in this specification could have properties that may vary from the values specified.

1.6 *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.* Specific precautionary statements are given in Note 1 and Section 10.

NOTE 3—There is no equivalent ISO standard for this specification. 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:*

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies[3]

D 150 Test Methods for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials[3]

D 256 Test Methods for Impact Resistance of Plastics and Electrical Insulating Materials[4]

D 257 Test Methods for D-C Resistance or Conductance of Insulating Materials[3]

D 542 Test Methods for Index of Refraction of Transparent Organic Plastics[4]

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[4]

D 638 Test Method for Tensile Properties of Plastics[4]

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[4]

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[4]

D 883 Terminology Relating to Plastics[4]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[4]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[4]

D 1898 Practice for Sampling of Plastics[4]

D 2863 Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-like Combustion of Plastics (Oxygen Index)[5]

D 3295 Specification for PTFE Tubing[5]

D 3418 Test Method for Transition Temperatures of Polymers by Thermal Analysis[5]

D 3835 Test Method for Determination of Properties of

[1] This specification is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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[2] Lovinger, A. J., "Poly(Vinylidene Fluoride)" *Developments in Crystalline Polymers*, Vol 1, Chapter 5, D. C. Bassett, Ed., Applied Science, London, 1982.

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

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

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

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

Polymeric Materials by Means of a Capillary Rheometer[6]
D 3892 Practice for Packaging/Packing of Plastics[5]
D 4895 Specification for Polytetrafluoroethylene (PTFE) Resins Produced From Dispersion[6]
E 380 Practice for Use of the International System of Units (SI)[7]

2.2 *IEC and ISO Standards:*

ISO 12086-1 Plastics—Fluoropolymer Dispersion and Moulding and Extrusion Materials—Part 1: Designation and Basis for Specification[8]
ISO 12086-2 Plastics—Fluoropolymer Dispersion and Molding and Extrusion Materials—Part 2: Preparation of Test Specimens and Determination of Properties[8]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of plastics terms used in this specification, see Terminology D 883.

3.2 *Abbreviations:Units, Symbols, and Abbreviations:*

3.2.1 For units, symbols and abbreviations used in this specification see Practice E 380.

4. Classification

4.1 This specification covers two types[9] of natural, unmodified PVDF fluoroplastics supplied in pellet form for molding and extrusion, and in powder form for solutions, dispersions, or coatings.

4.1.1 *Type I*—PVDF fluoroplastics are polymerized in emulsion. Depending upon the polymerization conditions, the peak melting point of the resin can be varied between 156 and 170°C. The diameter of the primary particle isolated from the emulsion is typically less than 1 μm; the dried powder has an average agglomerate diameter range of 3 to 15 μm.

4.1.1.1 Two distinctly different Type I emulsion PVDF resins are available commercially. These are differentiated by peak melting endotherm values, as shown in Table 1, and this difference is the basis for subdividing Type I resins into Grades 1 and 2. Table 1 shows the melt viscosity ranges encompassing resin grades available from several sources and are provided for information purposes only.

4.1.2 *Type II*—PVDF fluoroplastics are polymerized in suspension. Peak melting temperatures of these resins range from 164 to 180°C. The particles isolated from suspension are spherical and range typically from 20 to 150 μm in diameter.

4.1.2.1 Type II resins are available commercially, and the data of Table 1 reflect ranges encompassing values typical for the properties of available grades.

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

Specification				
Standard Number Block	Type	Grade	Class	Special notes
Example: Specification D 3222 – 97	I	2	...	...

For this example (D 3222 – 97, I2), the line callout describes a PVDF resin polymerized in emulsion, having a specific gravity between 1.75 and 1.79, and a peak melting endotherm between 162 to 170°C. 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, such as a preferred viscosity range, can be provided when required. When special notes are used, they should be preceded by a comma.

5. General Requirements

5.1 The material shall be of uniform composition and free of foreign matter to the contamination level agreed upon between the purchaser and seller.

6. Detail Requirements

6.1 *General Attributes*:

6.1.1 *Peak Melting Endotherm*—The material covered by this specification shall have a minimum peak melting endotherm for the type and class as shown in Table 1 when tested in accordance with Test Method D 3418. For Type I resins, this shall involve heating a solid specimen of 5 ± 1 mg from room temperature to 200°C at 10°C/min, maintaining the temperature at 200°C for 5 min, followed by cooling at a controlled rate of 10°C/min to about 30°C, then reheating at 10°C/min to 200°C. Record the peak melting endotherm during the second melting cycle.

6.1.1.1 *Temperature*—Test Type II resins likewise except that the maximum is 250°C.

6.1.2 *Specific Gravity*—A solid specimen of the material covered by this specification shall have the minimum specific gravity indicated in Table 1 (1.75 for Type I, Class 1 and 1.76 for all others) when tested in accordance with Test Methods D 792 or D 1505.

6.1.3 *Refractive Index*—The material covered in this specification shall have a refractive index of 1.42 when measured at

TABLE 1 Classification of PVDF Resins

Property	Typical Values or Ranges		
	Type I		
	Grade 1	Grade 2	Type II
Specific Gravity	1.75 to 1.79	1.75 to 1.79	1.76 to 1.79
Peak Melting Endotherm, °C	156 to 162	162 to 170	164 to 180
Apparent Melt Viscosity,[A] Pa·s:			
High Viscosity	2800 to 3800	2800 to 3100	2500 to 4000
Medium Viscosity	2300 to 2800	1300 to 2800	1300 to 2500
Low Viscosity	...	500 to 1300	500 to 1300

[A] Reported for a shear rate of 100 s^{-1} determined by capillary rheometry at 232°C (450°F) using 0.027 radian (60°) entrance angle die with L/D of 15 and according to procedures of Test Method D 3835. Multiply the pascal second values by ten to obtain poise values.

[6] *Annual Book of ASTM Standards*, Vol 08.03.
[7] *Annual Book of ASTM Standards*, Vol 14.02.
[8] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.
[9] Dohany, J. E., and Robb, L. E., "Poly(Vinylidene Fluoride)" *Kirk-Othmer Encyclopedia of Chemical Technology*, Vol 11, 3rd Edition, 1980, pp. 64–74.
[10] See the ASTM *Form and Style for ASTM Standards*, available from ASTM Headquarters.