



Designation: D4441 – 20

Standard Specification for Aqueous Dispersions of Polytetrafluoroethylene¹

This standard is issued under the fixed designation D4441; 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 aqueous dispersions of polytetrafluoroethylene (PTFE) resins. These resins are homopolymers of tetrafluoroethylene, or in some cases modified homopolymers containing not more than 1 % by weight of other fluoromonomers.

1.1.1 The dispersion is usually stabilized by one or more surfactants. Other non-PTFE materials may be present in total at 5 % or less by weight for special purposes.

1.1.2 This specification is for virgin material only and does not address recycled material as it is not appropriate for dispersions.

1.2 This specification is used to determine if a shipment, lot, or container of PTFE dispersion is suitable for its intended use. It covers only aqueous dispersions of polytetrafluoroethylene. It does not cover PTFE copolymers or filled dispersions. This specification includes test methods for both aqueous dispersion and the PTFE in the dispersion.

NOTE 1—Uses for the PTFE dispersion covered by this specification include, but are not limited to, casting of their films, impregnation of mechanical packings and other materials, surface coatings on various substrates, combined impregnation and coating of fibrous materials, and co-coagulation with other materials for subsequent processing into films, sheets, rods, and tubes.

NOTE 2—Information in this specification is technically equivalent to related information in ISO 20568-1 and ISO 20568-2.

1.3 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.

1.4 The following precautionary caveat pertains only to the test method portions 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.* Specific precautionary information is given in Notes 5 and 6.

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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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:²

D883 Terminology Relating to Plastics

D3892 Practice for Packaging/Packing of Plastics

D4464 Test Method for Particle Size Distribution of Catalytic Materials by Laser Light Scattering

D4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry

D4894 Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials³

D4895 Specification for Polytetrafluoroethylene (PTFE) Resin Produced From Dispersion³

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

IEEE/ASTM SI-10 Use of the International System of Units (SI): The Modern Metric System

2.2 ISO Standards:⁴

ISO 20568-1 Plastics—Fluoropolymer Dispersions and Molding and Extrusion Materials—Part 1: Designation and Specification

ISO 20568-2 Plastics—Fluoropolymer Dispersions and Molding and Extrusion Materials—Part 2: Preparation of Test Specimens and Determination of Properties

3. Terminology

3.1 Definitions:

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

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.

³ Specifications for other forms of polytetrafluoroethylene may be found in Specifications D4894 and D4895.

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

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

3.2 Definitions of Terms Specific to This Standard:

3.2.1 dispersion, *n*—a two phase system of which one phase consists of finely divided particles distributed throughout a bulk substance, the particles being the disperse phase and the bulk phase being the continuous phase.

3.2.2 dispersion particle size, *n*—a particle size measured in the presence of added surfactant.

3.2.3 raw dispersion particle size, *n*—a particle size measured in the absence of added surfactant (except for the small amount used in polymerization).

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

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

3.2.6 standard specific gravity (SSG), *n*—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.

4. Classification

4.1 This specification covers four types of virgin PTFE dispersions classified into those equal to or below 40 % PTFE and those above 40 % PTFE, and those containing added surfactant and those not containing added surfactant. The dispersions are classified into 13 grades depending on the level of added surfactant. Three classes of Surfactant Tolerance Level are provided to further differentiate the dispersion variability needed.

NOTE 3—The surfactant content is weight percent and is based on the solid, dry PTFE.

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

Standard Number Block: Example: Specification D4441 - XX,	Specification			Special Notes
	Type	Grade	Class	
	II	3	A	

For this example, the line callout would be Specification D4441 – XX, II3A and would specify a virgin PTFE dispersion that has all the properties listed for that type, grade, and class in the appropriate specified tables. A comma is used as the separator between the standard number and the type and class. Separators are not needed between the type, grade, and class.⁵ Provision for special notes is included so that other information can be provided when required.

5. Ordering Information

5.1 Orders for material under this specification shall contain the following information:

⁵ See the ASTM Form and Style Manual available from ASTM Headquarters.

TABLE 1 PTFE Level, Surfactant Level, and Tolerance Level Requirements

Type	% PTFE	Grade	Nominal % Added Surfactant	Class	Surfactant Tolerance Level
I	≤40	0	>0–<1	A	±0.5 %
		1	≥1–<2	B	±1.0 %
		2	≥2–<3	C	±2.0 %
II	>40	3	≥3–<4		
		4	≥4–<5		
		5	≥5–<6		
III	≤40 with 0 % added surfactant	6	≥6–<7		
		7	≥7–<8		
		8	≥8–<9		
IV	>40 with 0 % added surfactant	9	≥9–<10		
		10	≥10–<11		
		11	≥11–<12		
		12	≥12–<13		

- 5.1.1 Quantity, Grade, Class,
- 5.1.2 Name of material (Aqueous dispersion of PTFE),
- 5.1.3 ASTM designation and year,
- 5.1.4 Type,
- 5.1.5 Additions to the specification and supplementary requirements, if required.

6. Other Requirements

6.1 Significance—In addition to tests on the dispersion and the polymer, the following requirements apply:

6.2 The dispersion shall be free of contamination, dirt, mold, etc. It shall be of uniform composition. The expected level of coagulated polymer shall be specified.

7. Sampling

7.1 PTFE solids in the dispersion tend to settle upon standing. Therefore, the dispersion shall be homogenized by gentle mixing before sampling. Gentle mixing includes rolling a drum for 5 min at 3 to 4 rpm, by stirring with a smooth rod for 3- to 4-min, or by other types of gentle agitation. (Warning—Vigorous agitation will coagulate the dispersion.)

7.2 After blending, take the sample by removing an aliquot. A suitable method is by inserting a clean, smooth, dry glass tube, open at both ends, until it reaches the bottom of the container. An internal diameter of 6 or 7 mm (0.25 in.) is suitable. Smooth the ends of the tube to prevent injury. Close the upper end of the tube, remove the tube from the container, and transfer the contents to a clean dry glass jar. Repeat until the desired sample size is reached.

7.3 When samples are drawn from several containers, the individual samples shall be combined and thoroughly mixed by gentle stirring when the samples are combined and again before the combined sample is tested.

8. Test Methods for Dispersions

8.1 Scope—The specification contains test methods to define characteristics of the polymer and of the dispersion of the polymer. This section defines tests on the dispersion of the polymer.

8.2 *pH Value*—The test method shall be in accordance with Test Method E70. Some buyers or sellers attach importance to the pH of the PTFE dispersion. The pH value is not specified in this ASTM Standard.

NOTE 4—If the PTFE dispersion coats the electrode, clean the electrode thoroughly using a soft cloth wet with either toluene or concentrated surfactant.⁶

NOTE 5—**Warning:** Toluene is hazardous. Consult appropriate safety information on the material Safety Data Sheet before using.

8.3 *Solid Content and Surfactant Content by Gravimetric Weight Loss:*

8.3.1 *Significance*—Determine the percent PTFE solids and the percent surfactant by successive evaporations of water and surfactant. The percent surfactant is based on the weight of the PTFE. All percentages are based on weight.

8.3.2 *Apparatus*—An aluminum weighing dish, an oven capable of reaching 105 ± 5°C (221 ± 9°F), and an oven capable of reaching 380 ± 10°C (716 ± 18°F). Balance capable of weighing down to 0.0001 g.

8.3.3 *Procedure*—Weigh the aluminum weighing dish to 0.0001 g (W_A). Add 10 g of PTFE dispersion and weigh immediately to 0.0001 g (W_B). Dry the sample for 2 h at 105 ± 5°C (221 ± 9°F). Weigh the sample to 0.0001 g (W_C) after cooling to room temperature in a desiccator. After weighing, evaporate the surfactant by placing the sample in an oven at 380 ± 5°C (716 ± 9°F) for 35 ± 1 min. Allow the sample to cool in a desiccator to room temperature and weigh to 0.0001 g (W_D).

8.3.4 *Calculations*—For surfactants that are completely volatile use the following:

$$\text{PTFE, \%} = \frac{(W_D - W_A)}{(W_B - W_A)} \times 100$$

$$\text{surfactant, \%} = \frac{(W_C - W_D)}{(W_D - W_A)} \times 100$$

8.3.4.1 For surfactants that are not completely volatile use the following:

$$\text{PTFE, \%} = \frac{(W_C - W_A) - [(W_C - W_D) \times (I+k)]}{(W_B - W_A)} \times 100$$

$$\text{surfactant, \%} = \frac{(W_C - W_D) \times (I+k)}{(W_C - W_A) - [(W_C - W_D) \times (I+k)]} \times 100$$

where:

k = weight of nonvolatile portion of the surfactant/weight of volatile portion of the surfactant.

NOTE 6—Upon request, the supplier shall inform the user whether the surfactant can be completely removed by the procedures of this specification; and if not, shall define the surfactant or the volatile and nonvolatile portions of the surfactant.

8.3.5 *Precision and Bias*—No information can be presented on the precision and bias because of insufficient participation in the investigation.

8.3.6 The results of these tests on solids contents and surfactant shall conform to one of the types of dispersion listed in 4.1.

8.4 *Solids Content by Hydrometer:*

8.4.1 *Significance*—An approximate solids content in a dispersion is commonly determined from the specific gravity of the dispersion. The hydrometer reading is a function of the solids content, the surfactant content, and other parameters of the dispersion. Therefore, any single conversion table has inherent error and cannot be universally applicable. A table should be obtained from the supplier.

8.4.2 *Apparatus*—A hydrometer or set of hydrometers capable of measuring specific gravity from 1.000 to 1.500. Accuracy of the hydrometer shall be ±0.001. A graduated cylinder large enough to hold the hydrometer.

8.4.3 *Procedure*—Fill the graduated cylinder with PTFE dispersion to float the hydrometer. Place the hydrometer into the cylinder. Add dispersion until the cylinder is full and the meniscus is slightly convex. Read the hydrometer at the top of the dispersion. The reading shall be accurate to 0.001. Translate the hydrometer reading to solids content using the table.

8.4.4 *Precision and Bias*—No information can be presented on the precision and bias because of insufficient participation in the investigation.

NOTE 7—Some of the additives permitted in accordance with 1.1.1 may increase the viscosity of the dispersion so much that it is unlikely that the hydrometer procedure will be used to determine the density in a reliable manner.

8.5 *Test for Coagulated Polymer:*

8.5.1 *Significance*—Polymer that has coagulated may not be useful to the purchaser. This test will determine the percent of coagulated polymer.

8.5.2 *Apparatus*—A tared beaker to hold 1000 ± 1 g of dispersion; a filter screen, 80 mesh; a funnel, an oven capable of operating at 105 ± 5°C (221 ± 9°F) desiccator; balance capable of 1 mg.

8.5.3 *Materials*—Distilled water PTFE dispersion.

8.5.4 *Procedure*—Weigh a 1000 ± 1 g of dispersion (W_D) into the tared beaker. Weigh the screen to 1.0 mg and record the weight (W_S). Secure the screen to the funnel and filter the dispersion through the screen. Rinse the beaker with 25 mL of distilled water and use this rinse water to wash the coagulum on the screen. Gently wash the coagulated polymer on the screen with 25 mL of distilled water from a wash bottle. Carefully remove the screen from the funnel and dry at 105 ± 5°C (221 ± 9°F) for 2 h. Weigh the screen and coagulum to 1 mg after allowing it to cool to room temperature in a desiccator. Record the weight as W_C .

8.5.5 *Calculation*—Calculate the coagulum content as follows:

$$\text{coagulum content, \%} = \frac{(W_C - W_S)}{(W_D \times \text{PTFE content, \%})} \times 100$$

where:

W_D = mass of dispersion sample, g,

W_S = mass of screen, g,

⁶ Examples of a suitable surfactant are “Glim” detergent, B. J. Babbitt, Inc., “Joy” detergent, Proctor and Gamble, Inc., and “Triton” X-100, Rohm and Haas Co.

W_C = mass of screen and coagulated polymer, g, and PTFE content, %, = PTFE, % in the dispersion as determined in 8.3. It is to be expressed as a decimal fraction.

9. Methods for Tests on PTFE in the Dispersion

9.1 *Scope*—The specification contains test methods to define characteristics for the dispersion and for the PTFE in the dispersion. This section specifies tests that will define the solids in the dispersion to be PTFE and to characterize the PTFE.

9.2 *Standard Specific Gravity*—The test method for standard specific gravity is the same as given in Specification D4895. However, it is necessary to remove all of the surfactant before sintering in order to obtain meaningful results.

9.2.1 *Apparatus*—A 475-mL (16-oz) widemouth bottle with sealable top. A 5-in. Buchner funnel; 100-mL graduate cylinder, watchglass or aluminum pan. A vacuum oven capable of operating at 150°C (362°F) and an absolute pressure of 48 mm (1.9 in.) of mercury (26 in. of water); desiccator, balance.

9.2.2 *Materials*—Methanol, acetone, cheesecloth, PTFE dispersion, and deionized water.

9.2.3 *Procedure to Isolate PTFE as a Powder*:

9.2.3.1 Filter the PTFE dispersion through a double layer of cheese cloth. A convenient amount is enough dispersion to isolate 35 g of solids. The test requires 12.6 g solids. Add the appropriate amount of filtered dispersion to a 475-mL (16-oz) widemouth bottle.

9.2.3.2 Add the following in the order indicated; 50 mL of acetone, 75 mL of deionized water, and 75 mL methanol to the filtered sample.

9.2.3.3 Seal the bottle and shake until the sample is coagulated.

9.2.3.4 Place 8 layers of cheesecloth over the open end of the bottle and position the inverted bottle and cheesecloth in the Buchner funnel.

9.2.3.5 Remove bottle from Buchner funnel before filtering the liquid portion by opening the vacuum valve.

9.2.3.6 Release the vacuum and return the resin to the 475-mL (16-oz) bottle. Add 200 mL of methanol. Shake for 120 ± 15 s. Remove the methanol by vacuum filtering, as in 9.2.3.4 and 9.2.3.5.

9.2.3.7 Repeat 9.2.3.6.

9.2.3.8 Repeat 9.2.3.6 twice, using 200 mL of $85^\circ \pm 5^\circ\text{C}$ deionized or distilled water. Then repeat 9.2.3.6 once, using 150 mL of acetone.

9.2.3.9 Place the washed sample in an aluminum pan or watchglass and cover to prevent contamination.

9.2.3.10 Dry the PTFE powder to 0.04 %, or less, moisture.

NOTE 8—A vacuum oven at an absolute pressure of 10 mm Hg and a temperature of 150°C (302°F) is recommended to achieve dryness. Cool to room temperature in a desiccator before weighing. Repeated drying, cooling, and weighing may be necessary.

9.2.4 Determine the standard specific gravity in accordance with the method in Specification D4895. The standard specific gravity shall be between 2.14 and 2.27.

9.3 *Melting Characteristics*:

9.3.1 *Significance*—The melting peak temperature characteristics are specific for PTFE and serve to identify the material as PTFE.

9.3.2 The melting point of the PTFE should be determined on a sample. A sample may be obtained from 9.2.3.10. Two specimens should be tested, each twice. Specimens will show different melting points in the two meltings, the virgin melting point and a lower melting point for the recrystallized PTFE.

9.3.3 The test method shall be in accordance with Specification D4895. The first and second melting points should be measured.

9.3.4 The second melting point should be $327 \pm 10^\circ\text{C}$. The first melting point shall be at least 5°C greater than the second melting point.

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 As the dispersion is sensitive to settling, it is recommended that the dispersion be sampled prior to use and tested.

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

10.3.1 PTFE content.

10.3.2 Surfactant content.

10.4 Periodic check inspection with reference to a specification based upon this classification system 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 10.5.

10.5 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 the specification (line callout).

10.6 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 *Packaging*—The dispersion shall be packaged in 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.

11.2 *Marking*—Shipping containers shall be marked with the name of the dispersion, type, and quantity contained therein.

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

12. Keywords

12.1 fluoropolymers; polytetrafluoroethylene; polytetrafluoroethylene dispersions; PTFE