



Designation: D 4441 – 98

Standard Specification for Aqueous Dispersions of Polytetrafluoroethylene¹

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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 may be 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 12086-1 and ISO 12086-2.

1.3 The values stated in SI units, as detailed in Practice E 380, are to be regarded as the standard.

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 and health 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 D-20 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:

- D 883 Terminology Relating to Plastics²
- D 1898 Practice for Sampling of Plastics²
- D 3892 Practice for Packaging/Packing of Plastics³
- D 4464 Test Method for Particle Size Distribution of Catalytic Material by Laser Light Scattering⁴
- D 4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry⁵
- D 4894 Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Ram Extrusion Materials^{5,6}
- D 4895 Specification for Polytetrafluoroethylene (PTFE) Resins Produced from Dispersion^{5,6}
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode⁷
- E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)⁸

2.2 ISO Standards:⁹

- ISO 12086-1
- ISO 12086-2

3. Terminology

3.1 Definitions:

3.1.1 *General*—The terminology given in Terminology D 883 is applicable to this specification.

3.1.2 The terminology given in Specification D 4895 is applicable to this specification.

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.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 05.03.

⁵ Annual Book of ASTM Standards, Vol 08.03.

⁶ Specifications for other forms of polytetrafluoroethylene may be found in Specifications D 4894 and D 4895.

⁷ Annual Book of ASTM Standards, Vol 15.05.

⁸ Annual Book of ASTM Standards, Vol 14.02.

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

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

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

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

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 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, illustrated as follows:

Specification				
Standard Number Block:	Type	Grade	Class	Special Notes
Example: Specification D 4441 - 98,	II	3	A	

For this example, the line callout would be Specification D 4441 – 98, 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.¹⁰ 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:

¹⁰ 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 %
II	>40	2	≥2–<3	C	±2.0 %
		3	≥3–<4		
		4	≥4–<5		
III	≤40 with 0 % added surfactant	5	≥5–<6		
		6	≥6–<7		
		7	≥7–<8		
		8	≥8–<9		
		9	≥9–<10		
IV	>40 with 0 % added surfactant	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.

6.3 Measurements of pH and additives content may be a requirement.

7. Sampling

7.1 PTFE solids in the dispersion tend to settle upon standing. Therefore, the dispersion should be homogenized by gentle mixing before sampling. Gentle mixing can be accomplished by 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. **Caution**—Excessive agitation can 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. The ends of the tube should be smooth 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 may 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 E 70. 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—The PTFE dispersion can coat the electrode, so a thorough cleaning is necessary. Cleaning with toluene on a soft cloth or a concentrated surfactant on a soft cloth is suggested.¹¹

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*—The percent PTFE solids and the percent surfactant can be determined by successive evaporations

¹¹ 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.

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 $120 \pm 5^\circ\text{C}$ ($248 \pm 9^\circ\text{F}$), and an oven capable of reaching $380 \pm 10^\circ\text{C}$ ($716 \pm 18^\circ\text{F}$). 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 $120 \pm 5^\circ\text{C}$. 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 $380 \pm 5^\circ\text{C}$ ($716 \pm 9^\circ\text{F}$) 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:

$$\text{PTFE, \%} = \frac{(W_C - W_A) - [(W_C - W_D) \times (1 + k)]}{(W_B - W_A)} \times 100$$

$$\text{surfactant, \%} = \frac{(W_C - W_D) \times (1 + k)}{(W_C - W_A) - [(W_C - W_D) \times (1 + k)]} \times 100$$

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 according to 1.1.1 may increase the viscosity of the dispersion so much that it is unlikely that the hydrometer procedure can 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 $120 \pm 5^\circ\text{C}$ ($248 \pm 9^\circ\text{F}$) 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 $120 \pm 5^\circ\text{C}$ ($248 \pm 9^\circ\text{F}$) 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,

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 D 4895. 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.