



# Standard Specification for Aqueous Dispersions of Polytetrafluoroethylene<sup>1</sup>

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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 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 12086-1 and ISO 12086-2.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary information is given in Notes 5 and 6.

<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).

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## 2. Referenced Documents

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

[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 Materials<sup>3</sup>](#)

[D4895 Specification for Polytetrafluoroethylene \(PTFE\) Resin Produced From Dispersion<sup>3</sup>](#)

[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:<sup>4</sup>

[ISO 12086-1 Plastics Fluoropolymer Dispersions and Molding and Extrusion Materials—Part 1: Designation and Specification](#)

[ISO 12086-2 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.

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

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Specifications for other forms of polytetrafluoroethylene may be found in Specifications [D4894](#) and [D4895](#).

<sup>4</sup> 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

3.2.3 *raw dispersion particle size*—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.

**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		

Separators are not needed between the type, grade, and class.<sup>5</sup> 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:

- 5.1.1 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.<sup>6</sup>

<sup>5</sup> See the ASTM Form and Style Manual available from ASTM Headquarters.  
<sup>6</sup> 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.

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  $105 \pm 5^\circ\text{C}$  ( $221 \pm 9^\circ\text{F}$ ), 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  $105 \pm 5^\circ\text{C}$  ( $221 \pm 9^\circ\text{F}$ ). 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  $380 \pm 5^\circ\text{C}$  ( $716 \pm 9^\circ\text{F}$ ) for  $35 \pm 1$  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  $\pm 0.001$ . 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  $1000 \pm 1$  g of dispersion; a filter screen, 80 mesh; a funnel, an oven capable of operating at  $105 \pm 5^\circ\text{C}$  ( $221 \pm 9^\circ\text{F}$ ) desiccator; balance capable of 1 mg.

8.5.3 *Materials*—Distilled water PTFE dispersion.

8.5.4 *Procedure*—Weigh a  $1000 \pm 1$  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  $105 \pm 5^\circ\text{C}$  ( $221 \pm 9^\circ\text{F}$ ) 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 **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 (302°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  $120 \pm 15$  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  $5^\circ\text{C}$  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

APPENDIX

(Nonmandatory Information)

**X1. TURBIDIMETRIC METHOD FOR DETERMINING THE AVERAGE PARTICLE SIZE OF AQUEOUS DISPERSIONS OF PTFE**

X1.1 *Scope:*

X1.1.1 This method is applicable only to aqueous PTFE dispersions with average particle diameters in the range of 0.10–0.35 microns.

X1.2 *Significance and Use:*

X1.2.1 This method is provided because particle size is an important parameter of the dispersion for some applications.

X1.2.2 This method is based on the principle that the turbidity of a dispersion of a given concentration increases with increasing particle size.

X1.3 *Interferences:*

X1.3.1 Instrument geometry can influence the results in this method for particle size determination. For this reason, cross checks should be run between laboratories that wish to compare results.

X1.4 *Apparatus:*

X1.4.1 Spectrophotometer capable of accurate measurement of absorbance at 546 nm.

X1.4.2 Cells, 1 cm, optical glass, Fisher Cat. No. 14-385-912B.

X1.4.3 Constant-temperature bath, set at  $25 \pm 1$  degree C. 500-mL volumetric flask, 5-mL pipet, and 10-mL pipet.

X1.5 *Sampling:*

X1.5.1 The sampling procedure given in paragraph 5 applies to samples for this test.

X1.6 *Reagents and Materials:*

X1.6.1 Deionized Water, Cheese Cloth, Dispersion.

X1.7 *Calibration and Standardization:*

X1.7.1 Set controls on the spectrophotometer as follows:

Concentration decimal point	3
Concentration switch	high
Scale-mode switch	abs
Wavelength	546 nm
Source selector	visible
Tungsten lamp	on
Filter selection	W2 (390–600)

X1.7.2 With the sample cell filled with deionized water, adjust the span and zero as follows:

X1.7.2.1 Close the sample compartment and adjust the display to zero, using first the coarse 100 % T/ABS 0 control and then the fine 100 % T/ABS 0 control.

X1.7.2.2 Open the sample compartment and adjust the display to zero using the 0 % T control.

X1.7.2.3 Repeat Steps X1.7.2.1 and X1.7.2.2 several times, if necessary to achieve proper adjustment. It should not be necessary to use the coarse 100 % T/ABS 0 control at this point.

X1.8 *Hazards:*

X1.8.1 Uncased cigarettes, pipes, etc., should not be carried in the pocket while working with PTFE. Toxic fumes are generated from burning tobacco which is contaminated with PTFE.

X1.9 *Procedure:*

X1.9.1 *Sample Preparation:*

X1.9.1.1 Allow the dispersion to air-cool slowly to room temperature.

X1.9.1.2 Filter the dispersion through a double layer of cheesecloth and place receiver in a 25°C water bath for 30 min.

X1.9.1.3 Determine specific gravity and percent solids by paragraph 8.4 of this specification.

**TABLE X1.1 CONVERSION TABLE**

A/C to Average Particle Diameter							
A/C	d, μm	A/C	d, μm	A/C	d, μm	A/C	d, μm
11.5	0.100	27.5	0.147	43.5	0.188	69	0.253
12	0.102	28	0.148	44	0.189	70	0.256
12.5	0.103	28.5	0.149	44.5	0.190	71	0.258
13	0.105	29	0.150	45	0.191	72	0.260
13.5	0.106	29.5	0.152	45.5	0.193	73	0.263
14	0.108	30	0.153	46	0.194	74	0.266
14.5	0.110	30.5	0.154	46.5	0.195	75	0.269
15	0.112	31	0.155	47	0.196	76	0.271
15.5	0.113	31.5	0.157	47.5	0.197	77	0.274
16	0.114	32	0.158	48	0.199	78	0.277
16.5	0.116	32.5	0.159	48.5	0.200	79	0.279
17	0.118	33	0.160	49	0.202	80	0.282
17.5	0.119	33.5	0.161	49.5	0.203	81	0.285
18	0.121	34	0.163	50	0.204	82	0.288
18.5	0.122	34.5	0.164	51	0.206	83	0.291
19	0.124	35	0.166	52	0.209	84	0.294
19.5	0.125	35.5	0.167	53	0.212	85	0.297
20	0.126	36	0.168	54	0.214	86	0.300
20.5	0.127	36.5	0.169	55	0.217	87	0.303
21	0.129	37	0.171	56	0.219	88	0.306
21.5	0.130	37.5	0.172	57	0.221	89	0.310
22	0.132	38	0.174	58	0.224	90	0.314
22.5	0.133	38.5	0.175	59	0.227	91	0.317
23	0.135	39	0.176	60	0.229	92	0.320
23.5	0.136	39.5	0.177	61	0.232	93	0.323
24	0.137	40	0.178	62	0.234	94	0.327
24.5	0.139	40.5	0.180	63	0.236	95	0.330
25	0.140	41	0.182	64	0.239	96	0.333
25.5	0.141	41.5	0.183	65	0.242	97	0.336
26	0.142	42	0.184	66	0.245	98	0.339
26.5	0.144	42.5	0.186	67	0.247	99	0.343
27	0.145	43	0.187	68	0.250	100	0.346

X1.9.1.4 Fill a 500 mL volumetric flask about halfway with deionized water that has been brought to 25°C in the constant-temperature bath.

X1.9.1.5 If the percent solids is 25 % or greater (Step X1.9.1.3), transfer 5 mL (pipet) of the filtered dispersion to the flask. If the percent solids is less than 25 % transfer 10 mL (pipet).

NOTE X1.1—In order to eliminate coagulation, keep the tip of the pipet below the water level in the flask until all but the last few drops of dispersion have run out. To maintain a clean pipet, rinse well with water after each use and store in water. A periodic check of the pipet should be made using pure water to check accuracy and reproducibility of delivery.

X1.9.1.6 Dilute to the mark with 25°C deionized water and mix well.

#### X1.9.2 Analysis:

X1.9.2.1 Use a water blank to zero the instrument. Measure the absorbance of the diluted dispersion at 546 nm.

#### X1.10 Calculations:

X1.10.1 Calculate the parameter ( $A/C$ ) as follows:

$$\frac{A}{C} = \frac{A}{G \times S \times D} \times 50\,000$$

where:

- A = absorbance (Step X1.9.2.1)
- C = conc of particles, g/g emulsion
- G = specific gravity of dispersion, g/mL (Step X1.9.1.3)
- S = solids content, % (Step X1.9.1.3)
- D = vol of dispersion added, mL (Step X1.9.1.5)
- 50 000 = (dilution vol. 500 mL) × (100 %)

X1.10.2 From the attached table, determine the average particle diameter,  $d$ , corresponding to the value of ( $A/C$ ) calculated in Step X1.10.1.

#### X1.11 Report:

X1.11.1 The report should contain proper identification of the sample and the particle size. The particle size should be reported to 0.001 micrometer.

#### X1.12 Precision and Bias:

X1.12.1 The precision of the particle size method is determined by the precision with which the absorbance is measured. On runs of one sample using one operator, a standard deviation of 0.001 on the 0.2- $\mu$ m level was obtained.

X1.12.2 Accuracy varies from one dispersion product to another, depending on how closely the refractive index difference between the solid and liquid phases matches an assumed value for this parameter. A surfactant, if present, can affect a light scattering particle size measurement by changing the index of refraction. Generally the “dispersion particle size” is about 0.02  $\mu$ m greater than the “raw dispersion particle size” due to the change in index of refraction. The presence of small amounts of comonomer can also change the index of refraction.

X1.12.3 Section 8.4 of this specification for determining solids content and specific gravity by hydrometer is valid only for raw dispersions containing no surfactants. However, solids content determination by hydrometer is used in conjunction with this method on stabilized dispersion products. A solution of 4 % TRITON X-100(9) in water was both calculated and measured to have a specific gravity of 1.002 instead of the value of 0.9971 for pure water anticipated by the method. Based on 60 % solids dispersion, this discrepancy would cause an error in dispersion particle size of about 0.001  $\mu$ m.

## SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4441–04(2010)) that may impact the use of this standard. (May 1, 2015)

- (1) Changed “120 ± 5°C (248 ± 9°F)” to “105 ± 5°C (221 ± 9°F)” in 8.3.2, 8.3.3, 8.5.2 and 8.5.4.
- (2) Changed “2.15” to “2.14” in 9.2.4.

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