Designation: D5675 – 13

Standard Classification for Low Molecular Weight PTFE and FEP Micronized Powders[1]

This standard is issued under the fixed designation D5675; 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 classification system provides a method of adequately identifying low molecular weight polytetrafluoroethylene (PTFE) and fluorinated ethylene propylene (FEP) micronized powders using a system consistent with that of Classification System D4000. It further provides a means for specifying these materials by the use of a simple line callout designation. This classification covers fluoropolymer micronized powders that are used as lubricants and as additives to other materials in order to improve lubricity or to control other characteristics of the base material.

1.2 These powders are sometimes known as lubricant powders. The powders usually have a much smaller particle size than those used for molding or extrusion, and they generally are not processed alone. The test methods and properties included are those required to identify and specify the various types of fluoropolymer micronized powders. Recycled fluoropolymer materials meeting the detailed requirements of this classification are included (see Guide D7209).

1.3 These fluoropolymer micronized powders and the materials designated as filler powders (F) in ISO 12086-1 and ISO 12086-2 are equivalent.[2]

1.4 The values stated in SI units as detailed in IEEE/ASTM SI-10 are to be regarded as the standard.

1.5 *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 7.1.2.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D883 Terminology Relating to Plastics

D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

D1600 Terminology for Abbreviated Terms Relating to Plastics

D3892 Practice for Packaging/Packing of Plastics

D4000 Classification System for Specifying Plastic Materials

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

D4567 Test Method for Single-Point Determination of Specific Surface Area of Catalysts and Catalyst Carriers Using Nitrogen Adsorption by Continuous Flow Method

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

D4895 Specification for Polytetrafluoroethylene (PTFE) Resin Produced From Dispersion

D7209 Guide for Waste Reduction, Resource Recovery, and Use of Recycled Polymeric Materials and Products

D5740 Guide for Writing Material Standards in the Classification Format

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

2.2 *ISO Standards:*[4]

ISO 12086-1 Plastics-Fluoropolymer Dispersions and Moulding and Extrusion Materials Part 1: Designation and System and Basis for Specification

ISO 12086-2 Plastics-Fluoropolymer Dispersions and

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

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[2] Designations, specifications, and test methods are included in ISO 12086-1 and 12086-2.

[3] 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.

[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**

Moulding and Extrusion Materials Part 2: Preparation of Test Specimens and Determination of Properties

3. Terminology

3.1 *Definitions*—The terminology given in Terminology D883 is applicable to this classification unless otherwise specified.

3.1.1 *bulk density, n*—the mass per unit volume, in grams per litre of a loosely packed material, such as a molding powder. **D4895**

3.1.2 *lot, n*—one production run or uniform blend of two or more production runs. **D4895**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *direct polymerization powder, n*—fluoropolymer material based on polymerizations designed to produce low molecular weight PTFE resins with properties of materials described in this standard.

3.2.2 *dispersion-based powder, n*—fluoropolymer material based on the type of polymerization normally related to the production of "paste or coagulated dispersion type" fluoropolymer resins.

3.2.3 *micronized powder, n*—a material comprised of particles reduced in average size to a dimension typically between 1 and 100 µm.

3.2.4 *reground PTFE, n*—PTFE material produced by grinding polytetrafluoroethylene (PTFE) material that has been preformed but has never been sintered.

3.2.5 *reprocessed PTFE, n*—PTFE material produced by grinding PTFE material that has been both preformed and sintered.

3.2.6 *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.

3.2.7 *suspension-based powder, n*—fluoropolymer material based on the type of polymerization normally related to the production of granular PTFE resins.

3.3 *Abbreviations*—Abbreviated terms are in accordance with Terminology D1600.

4. Classification

4.1 This classification covers two groups of fluoropolymer micronized powders. Fluoropolymer micronized powders are classified into groups according to their base fluoropolymer. These groups are further subdivided into classes and grades as shown in Table 1.

NOTE 1—An example of this classification system is as follows:
The designation ASTM D5675 PTFE0111 indicates PTFE micronized powder in accordance with Specification D5675:
01 = PTFE resin,
1 = suspension polymerization based, and
1 = formerly Type I, Grade 1, Class A, in Specification D5675 - 95a with a particle size of 1 to <10 µm (average diameter), a surface area of 0.8 to 4.5 m^2/g, and a mass flow rate of >1 g/10 min using a load of 5 kg.

5. General Requirements

5.1 The resin shall be uniform and shall contain no additives or foreign material.

5.2 The color of the material as shipped by the seller shall be white to gray.

TABLE 1 Basic Properties

Group	Description	Class	Description	Grade	Description	Particle Size, Average Diameter, µm	Surface Area, m^2/g	Melt-Flow Rate,[A] g/10 min	Mass[B]
01[C]	PTFE	1	suspension based	1	[D]	1 to <10	0.8 to 4.5	>1	5
				2	[E]	10 to 25	0.8 to 4.5	>0.1	10
		2	dispersion based	1	[F]	1 to <10	4.6 to 15	>1	5
				2	[G]	10 to 30	4.6 to 15	>0.1	10
				3	[H]	25 to 50	4.6 to 15	>1	5
				4	[I]	50 to 150	4.6 to 15	>1	5
		3	direct polymerization	1	[J]	2 to 15	4.6 to 15	>1	5
		4	reground suspension	1	[K]	1 to <10	0.8 to 4.5	>1	5
		5	reground dispersion	1	[L]	1 to 25	0.8 to 4.5	>1	5
		6	previously sintered	1	[M]	10 to 50	<1.5	>20	5
02[N]	FEP	1		1	[O]	10 to 30	4.6 to 15	4 to 12	5

[A] Orifice diameter of 2.0955 mm and temperature of 372°C.
[B] Kilogram load on plastometer.
[C] Group 01 materials have a specific gravity of 2.10 to 2.30 g/cm^3, a water content (maximum) of <0.1 %, a melting point (peak temperature) of 315 to 340°C, and a bulk density of 225 to 600 g/L.
[D] Formerly Specification D5675–95a, Type I, Grade 1, Class A.
[E] Formerly Specification D5675–95a, Type I, Grade 1, Class B.
[F] Formerly Specification D5675–95a, Type I, Grade 2, Class A.
[G] Formerly Specification D5675–95a, Type I, Grade 2, Class B.
[H] Formerly Specification D5675–95a, Type I, Grade 2, Class C.
[I] Formerly Specification D5675–95a, Type I, Grade 2, Class D.
[J] Formerly Specification D5675–95a, Type I, Grade 3, Class A.
[K] Formerly Specification D5675–95a, Type I, Grade 4, Class A.
[L] Formerly Specification D5675–95a, Type I, Grade 4, Class B.
[M] Formerly Specification D5675–95a, Type I, Grade 5, Class A.
[N] Group 02 materials have a specific gravity of 2.10 to 2.20 g/cm^3, a water content (maximum) of <0.1 %, a melting point (peak temperature) of 250 to 280°C, and a bulk density of 200 to 600 g/L.
[O] Formerly Specification D5675–95a, Type II, Grade 1, Class A.

6. Sampling

6.1 Sampling shall be statistically adequate to satisfy the requirements of 9.4.

7. Sample Preparation

7.1 *Test Samples:*

7.1.1 For each lot of fluoropolymer micronized powder, collect a suitable sample for testing. The sample powder shall be at the standard laboratory temperature of 23 ± 2°C (73.4 ± 3.6°F) prior to testing.

7.1.2 Screen the powder through a No. 10 hand sieve in order to break up or remove any lumps. (**Warning—** Fluoropolymers have the potential to evolve small quantities of gaseous products when heated above 200°C (400°F). It is possible that some of these gases will be harmful. Exhaust ventilation must consequently be used whenever the resins are heated above this temperature, as they are during the melting operations that are part of this classification. Since the temperature of burning tobacco exceeds 200°C, those working with fluoropolymer resins shall ensure that the tobacco is not contaminated.)

8. Test Methods

8.1 *Melting Characteristics by Thermal Analysis:*

8.1.1 *Significance and Use*—Most of the PTFE resins that fall within the scope of this classification have never been melted. An exception is Group 01 Class 6 micronized powders. However, these micronized powders generally do not have higher melting peak temperatures upon initial melting than upon second or subsequent meltings. Except for Group 01 Class 4 and 5 powder, melting-peak temperatures cannot be used to determine whether the PTFE resins have been melted previously. Melting peak temperatures are used to determine the conformance of a micronized powder to the melting peak temperature requirements given in Table 1 of this classification.

8.1.2 *Apparatus*—Use the apparatus described in Test Method D4591.

8.1.3 *Procedure*—Measure the melting peak temperatures in accordance with the procedures given in Test Method D4591. An initial melting peak temperature above the melting peak temperature obtained on the second and subsequent melting (defined as the second melting peak temperature) probably indicates that the PTFE resin is Group 01, Classes 5 and 6 micronized powder. The second melting peak temperature occurs at approximately 327°C (621°F). The difference between the initial and second melting peak temperatures is usually greater than 5°C (9°F) but might be slightly less. There is no significant difference between the initial and second melting peak temperatures for all of the other fluoropolymer micronized powders. If peak temperatures are difficult to discern from the curves because the peaks are rounded, straight lines shall be drawn tangent to the sides of the peak. The peak temperature shall be taken to be where these lines intersect beyond the peak.

8.2 *Melt Flow Rate:*

8.2.1 *Test Method*—Determine the melt flow rate at 372 ± 1°C using a modification of the extrusion plastometer described in Test Method D1238. Measure flow rate using a load of 5000 or 10000 g.

8.2.2 *Apparatus*—The apparatus shall consist of an extrusion plastometer, described in Test Method D1238, modified by use of corrosion-resistant[5] alloy for the barrel lining, orifice, orifice securing device, and piston tip.

8.2.3 *Measurement of Flow Rate*—Proceed in accordance with Test Method D1238 using the appropriate sample weight and testing time from Table 2 of Test Method D1238. The test temperature shall be 372 ± 1°C and the load of 5000 or 10000 g in accordance with Table 1 of this classification.

8.3 *Specific Gravity*—Measure specific gravity using the procedures described in Test Method A of Test Method D854, except use Isopar K[6] instead of water in order to provide proper wetting of the powder.

8.4 *Water Content:*

8.4.1 *Significance and Use*—The presence of an excessive amount of water in fluoropolymer resin has a significant adverse effect on the processing characteristics of the resin and the quality of the products made using the resin. A sample of the powder of known weight is dried in a vacuum oven in a tared aluminum weighing dish. When the powder is dry, it is removed from the oven, placed in a desiccator, allowed to cool, and then reweighed. The water content is calculated from the weight lost during drying.

8.4.2 *Apparatus*—Use the apparatus described in Specification D4895.

8.5 *Particle Size*—Measure particle size using the procedures described in Test Method D4464, except use methanol instead of water.

8.6 *Surface Area*—Measure surface area using the procedures described in Test Method D4567, except use a 20-min degas period at 200°C rather than 60 min at 300°C.

8.7 *Bulk Density*—Measure bulk density using the procedures described in Specification D4895.

9. Inspection and Certification

9.1 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.

9.2 Lot acceptance shall be the basis on which acceptance or rejection of the lot is made. The lot-acceptance inspection shall consist of particle size determination.

9.3 Periodic check inspection with reference to a specification based on 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 9.4.

[5] "Stellite" Grade No. 18, Stellite Division of Cabot Corp., Kokomo, IN 46901, and "Duranickel" No. 301, Huntington Alloy Co., Huntington, WV 25720, have been found to be resistant to fluorocarbon resins.

[6] Isopar is available from Exxon Co. and is also used in Specification D4895.

9.4 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).

9.5 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.

10. Packaging and Marking

10.1 The provisions of Practice D3892 apply to packaging, packing, and marking of containers for plastic materials.

11. Keywords

11.1 fluorinated ethylene propylene, FEP; fluoropolymer; line callout; lubricant powder; micronized powder; polytetrafluoroethylene; PTFE

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D5675 – 04(2010)) that may impact the use of this standard. (September 1, 2013)

(1) The scope of materials covered by this standard have been clarified from the generic and poorly defined "fluoropolymer micropowders" to be more specific; namely, "low molecular weight polytetrafluoroethylene (PTFE) micronized powders" and "low molecular weight fluorinated ethylene propylene (FEP) micronized powders."
(2) The term "micronized powder" was redefined.