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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Specification for Filled Compounds of Polytetrafluoroethylene (PTFE) Molding and Extrusion Materials[1]

This standard is issued under the fixed designation D 4745; 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 polytetrafluoroethylene (PTFE) filled molding compounds made with virgin PTFE resins defined in Specification D 4894, except Types I, IV, V, and VI.

NOTE 1—This specification can be used as a model for other PTFE compounds having particulate fillers that can survive the sintering temperatures of PTFE as can those listed in this specification. This specification is restricted to virgin PTFE for technical reasons. Recycled material cannot be processed successfully.

NOTE 2—The properties measured on commercially fabricated parts may differ from the listed values for samples prepared by the procedures given in this specification, depending on part geometry and processing parameters.

NOTE 3—There is no ISO equivalent to this specification.

1.2 The values stated in SI units as detailed in Practice E 380 are to be regarded as the standard and the practices of E380 incorporated herein.

1.3 The following statement applies to the test method portion, Section 12, 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.* See 9.5 and Note 4 for a specific warning statement.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 638 Test Method for Tensile Properties of Plastics[2]

D 792 Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement[2]

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[2]

D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials[2]

D 1898 Practice for Sampling of Plastics[2]

D 3892 Practice for Packaging/Packing of Plastics[3]

D 4894 Specification for Polytetrafluoroethylene (PTFE) Granular Molding and Extrusion Materials[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

E 380 Practice for the Use of the International System of Units (SI)[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[5]

3. Terminology

3.1 *Definitions*—The terminology given in Terminology D 883 is applicable to this specification unless otherwise specified.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *bulk density*, *n*—the mass in kilograms per cubic metre of resin compound measured under the conditions of the test.

3.2.2 *density*, *n*—the mass per unit volume in air in milligrams per cubic metre (grams per cubic centimetre) of the material at a temperature of 23 ± 2°C (73.4 ± 4°F).

3.2.3 *filled compound*, *n*—blend of PTFE resin as the matrix and particulate fillers, generally glass, other inorganic, metallic, or polymeric materials that withstand the sintering temperature of PTFE (327 to 380°C).

3.2.4 *free-flow resins (pelletized)*, *n*—generally made by treatment of finely divided resins to produce free-flowing agglomerates.

3.2.5 *lot*, *n*—one continuous production run or a uniform blend of two or more production runs of the compound.

3.2.6 *pigmented compound*, *n*—a compound in which a pigment is added for colorant purposes only.

3.2.7 *standard flow resins (nonpelletized)*, *n*—finely divided resin with an average particle size less than 100 µm.

3.3 *Abbreviations*—Abbreviations are in accordance with

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

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

[4] *Annual Book of ASTM Standards*, Vol 08.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

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

Terminology D 1600. PTFE is the acronym for polytetrafluoroethylene.

4. Classification

4.1 This specification covers the following two types of PTFE compounds

4.1.1 *Type I*—Nonpelletized material, for general-purpose compression molding.

4.1.2 *Type II*—Pelletized or free-flowing material, for molding, automatic molding, or ram extrusion.

4.2 Thirteen grades of each type distinguished by the nature of the filler(s) are listed in Tables 1-3.

4.3 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 the following illustrates:

Specification				
Standard Number : Type : Grade : Class : Special Block : : : : Notes				
:	:	:	:	:
Example: Specification D 4745 – 97	II	2		

4.3.1 For this example, the line callout would be Specification D 4745 – 97, II 2, and would specify a pelletized or free-flowing filled composition of polytetrafluoroethylene that has all of the properties listed for that type, and grade in the appropriate specified properties, tables, or both, in the specification identified. A comma is used as the separator between the standard number and the type. Separators are not needed between the type, grade, and class.[6] A provision for special notes is included so that other information can be provided when required. An example would be in Specification D 3295 – 81a where dimensions and tolerances are specified for each AWG size within type and class. When special notes are used, they should be preceded by a comma.

5. Ordering Information

5.1 The filled compounds of PTFE may be ordered using the type, (see 4.1) and the grade (see reference Table 1 and Table 3), or they may be ordered using the designation of the suppliers.

[6] See the *ASTM Form and Style Manual*, available from ASTM Headquarters.

6. Requirements

6.1 The PTFE compounds covered by this specification shall be uniform (filler and resin particles evenly distributed) and shall contain no foreign material.

6.2 The PTFE compounds shall conform to the requirements prescribed in Tables 1-3 when tested by the procedures specified herein. Table 1 and Table 3 list requirements for Type I. Table 2 and Table 3 reference requirements for Type II.

6.3 Other PTFE compounds are commercially available, but are not described in this specification.

7. Sampling

7.1 Sample the resin in accordance with the Sections covering General Sampling Procedures in Practice D 1898. Adequate statistical sampling prior to packaging shall be considered an acceptable alternative.

7.2 The producer shall take (and test) sufficient within-lot samples to ensure adequate in-process quality control and continuing conformance to the property requirements of this specification.

8. Number of Tests

8.1 Routine lot inspection tests shall consist of those carried out to determine the requirements specified in Table 1 or Table 3 depending on type. Periodic tests shall include using all the tests to determine the requirements in Table 3, depending on type.

8.2 The requirements listed in Tables 1-3, as they apply, are sufficient to establish conformity of a material to this specification. When the number of test specimens is not stated in the test method, single determinations may be made. If more than single determinations are made on specimens from separate portions of the same sample, the results shall be averaged. The single or average result shall conform to the requirements prescribed in this specification.

9. Test Specimens

9.1 Test specimens shall be cut from billets molded in accordance with the following procedures. An acceptable alternate procedure for molding the test plaque is described in Specification D 4894.

9.2 *Test Billets*:

TABLE 1 TFE Compounds, Type I, Standard Flow (Nonpelletized)

Type	Grade	Raw Resin Bulk Density, min, g/L	Molded Parts (Molded and Sintered)				
			Density, min, g/cm^3	SPG, max, g/cm^3	Tensile Strength		Elongation, min, %
					min, MPa	min, psi	
1	15 % glass fiber	400	2.150	2.25	19.6	2840	250
2	25 % glass fiber	425	2.150	2.250	15.7	2270	200
3	35 % glass fiber	450	2.200	2.300	10.3	1500	150
4	5 % glass fiber and 5 % MoS_2	350	2.150	2.300	20.7	3000	250
5	15 % glass fiber and 5 % MoS_2	375	2.150	2.300	17.2	2500	200
6	10 % graphite	350	2.100	2.220	17.9	2600	225
7	15 % graphite	300	2.100	2.200	16.6	2400	100
8	25 % carbon and graphite	350	1.950	2.100	11.0	1600	80
9	32 % carbon and graphite	325	1.900	2.100	6.9	1000	50
10	40 % bronze	500	2.900	3.200	17.2	2500	175
11	60 % bronze	650	3.800	4.000	13.8	2000	140
12	55 % bronze and 5 % MoS_2	700	3.500	4.000	10.3	1500	80
13	50 % stainless steel	500	3.200	3.600	17.2	2500	150

TABLE 2 TFE Compounds, Type II, Free-Flow (Pelletized)

Type	Grade	Raw Resin Bulk Density, min, g/L	Molded Parts (Molded and Sintered)				
			Density, min, g/cm³	SPG, max, g/cm³	Tensile Strength		Elongation, min, %
					min, MPa	min, psi	
1	15 % glass fiber	625	2.150	2.25	17.2	2500	200
2	25 % glass fiber	625	2.150	2.250	12.4	1800	180
3	35 % glass fiber	650	2.200	2.300	8.3	1200	100
4	5 % glass fiber and 5 % MoS_2	575	2.150	2.300	17.2	2500	220
5	15 % glass fiber and 5 % MoS_2	600	2.150	2.300	13.8	2000	180
6	10 % graphite	600	2.100	2.220	13.8	2000	180
7	15 % graphite	550	2.100	2.200	10.3	1500	100
8	25 % carbon and graphite	500	1.950	2.100	8.3	1200	20
9	32 % carbon and graphite	500	1.900	2.100	6.9	1000	20
10	40 % bronze	750	2.900	3.200	13.8	2000	100
11	60 % bronze	900	3.800	4.000	10.3	1500	100
12	55 % bronze and 5 % MoS_2	900	3.500	4.000	6.9	1000	50
13	50 % stainless steel	850	3.200	3.600	13.8	2000	100

TABLE 3 Required Filler Content

	Mass, %	Tolerance, ± , %
1	0 to 3	1
2	4 to 25	2
3	26 to 60	3
4	61 to 75	5

9.2.1 Prior to molding, screen the material through a 2.0-mm hand sieve.

9.2.2 Preform solid test billets in a mold (see Fig. 1) having a cross-sectional area not greater than 25.8 $cm^2 \leq (4\ in.^2)$ and of sufficient height to contain the sample. Clearance should be sufficient to ensure escape of entrapped air during pressing. The billet length may be varied in accordance with the amount of testing to be done. A mold length of 250 mm (9.8 in.) produces a billet approximately 75 mm (2 to 3 in.) long. Powder-charge weight may be varied according to the density of the material. The billet length should not exceed 75 mm (3 in.).

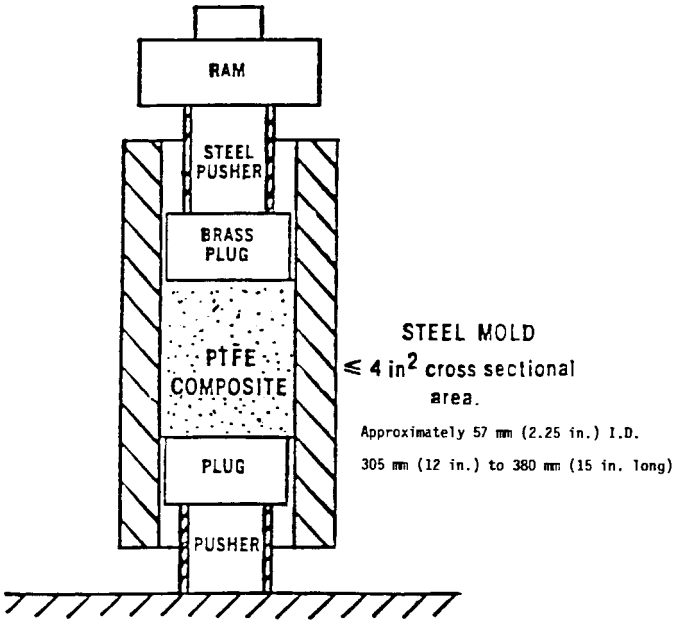


FIG. 1 Preforming of PTFE Composite Test Billet

9.2.3 Assemble the mold. Add the resin to the mold, taking care not to fill within 13 mm (0.5 in.) of the top of the cavity. Insert the top plug and apply hand pressure, making certain that the pusher is centered in the mold. Place the mold in a hydraulic press and remove the support ring or spacers. Do not allow the two end plugs to bottom on the mold shell. Apply an initial load to the mold of 3.45 MPa (500 psi) ± 10 % and hold for 1 to 2 min. Increase the loading smoothly to the final preforming pressure in 3 to 5 min. Use 35 MPa (5100 psi) for compounds containing 15 % by weight or less filler and 70 MPa (10150 psi) for composite compounds containing more than 15 % filler. For compounds containing bronze filler, use 60 MPa (8500 psi), and hold under maximum pressure for 2 to 5 min. Release the pressure gradually without apparent movement of the press platens. Then open the press, remove the top pusher from the mold, and force the preform vertically out of the mold, using a continuous, smooth movement.

9.2.4 Place the preform in a sintering oven and sinter in accordance with the procedures in Table 4. Use Procedure B for compounds containing molybdenum disulfide filler.

9.3 *Sectioning Test Billet*:

9.3.1 Remove and discard the top and bottom 2-mm (1⁄16 in.) section of the billet. Obtain transverse test specimens from as near the center of the billet as possible.

9.3.2 Prepare five test specimens, 1 ± 0.25 mm (0.040 ± 0.010 in.) in thickness for the determination of tensile strength and elongation and cut a piece of suitable thickness for density measurements. This piece should be approximately cubical in shape, weighing at least 10 g. All surfaces must be smooth. Take care to avoid wedge-shape cuts.

9.4 The alternative test billet is described in Specification D 4894.

TABLE 4 Sintering Procedures for Test Billets

	Procedure A	Procedure B
Initial temperature, °C (±)[A]	Ambient	Ambient
Rate of heating, °C/h (°F/h)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 10)
Hold temperature, °C (°F)	370± 6 (698 ± 10)	360 ± 6 (680 ± 10)
Hold time, min	120 ± 5	120 ± 5
Rate of cooling, °C/h (°F/h)	60 ± 5 (108± 9)	60 ± 5 (108 ± 9)
Final temperature, °C (°F)[A]	95 ± 6 (203± 10)	95 ± 6 (203± 10)
Time to cool to room temperature, h	24	24

[A] Oven can be opened safely at these temperatures.

9.5 **Safety Warning**—At normal processing temperatures, PTFE liberates vapors that may be harmful. Provide adequate ventilation in areas where PTFE compounds are exposed to elevated temperatures. Avoid contaminating smoking materials with PTFE compounds.

10. Conditioning Test Specimens

10.1 For density and tensile properties the test specimens shall be conditioned in accordance with Procedure A of Practice D 618 for a period of at least 4 h prior to test.

11. Test Conditions

11.1 Tests shall be conducted at 25 ± 2°C (77 ± 3.6°F) instead of the standard laboratory temperature of 23 ± 2°C (73.4 ± 3.6°F) unless otherwise specified in the test methods or in this specification. This deviation from the standard laboratory temperature is made because of the necessity for maintaining test temperatures above approximately 22°C (71.6°F) to avoid problems related to the crystalline transition that occurs in PTFE in this temperature region. Since the resin does not absorb water, the maintenance of constant humidity during testing is not required.

12. Test Methods

12.1 *Filler Content*:

12.1.1 *Scope*—This burn-out procedure for filler content may be used for determining the amount of a filler in a compound. The procedure shall be carried out in an inert atmosphere, especially when fillers that react with PTFE and oxygen in the air are present. This reaction produces volatile products that cause incorrect results.

12.1.2 The equipment used is a tube furnace suitable for use at 680 ± 25°C (1250 ± 50°F). The furnace should have a combustion tube made of ceramic or Vycor glass. Two nickel or platinum combustion boats should be used to enable the test to run in duplicate. A system is needed to supply a constant purge of dry nitrogen through the furnace at a rate of 10 to 50 mL/min.

12.1.3 Scrub the pyrolysis gases with a base, such as a dilute solution of sodium hydroxide, and vent to the outside atmosphere.

NOTE 4—Take care to avoid leakage of fumes into the work area. Take precautions to avoid the inhalation of, or exposure to gases from the pyrolysis since these gases may be hazardous.

12.1.4 Add approximately 2 g of the sample to each of the combustion boats, using standard quantitative laboratory weighing practices. Place boats and samples into the furnace. Heat and hold at 680 ± 25°C (1250 ± 50°F) until complete degradation of the PTFE has occurred. One hour is sufficient time. Maintain an inert atmosphere during the heating, pyrolysis, and cooling steps. Calculate the percentage of filler as the net weight of the residue multiplied by 100, divided by the net weight of the original sample.

12.1.5 Calculate the filler content by volume as follows:

$$E = \frac{F}{U + F} \times 100 \quad (1)$$

where:

F = grams per liter of the filler.
U = grams per liter of the unfilled PTFE.
E = equivalent filler content (% by volume).

12.1.6 The percent by volume of filler in a finished piece is lower than that in the powder compounds due to the increase in volume of PTFE that results from the change in crystalline content that occurs during sintering.

12.2 *Filler Content (Alternate Method)*:

12.2.1 *Scope*—This burn-out procedure for filler content may be used as alternate to the split tube furnace method of 12.1. The procedure shall be carried out in an inert atmosphere, especially when fillers that react with PTFE and oxygen in the air are present. This reaction produces volatile products that cause incorrect results.

12.2.2 *Equipment*—Thermogravimetric analyzer, capable of weighing a sample as heat is applied. The analyzer should be programmable to apply 680 ± 20°C (1250 ± 50°F). A constant supply of nitrogen, or suitable purge gas, is needed to flow through the furnace at a rate of 20 ± 10 mL/min. The crucible should be constructed of aluminum oxide or platinum.

12.2.3 The pyrolysis gases should be scrubbed in accordance with 12.1.3.

12.2.4 Add 50 ± 10 mg to a crucible. Place crucible and sample in furnace. Heat and hold at 680 ± 25°C (1250 ± 50°F) until complete degradation of the PTFE has occurred (one hour is sufficient). Maintain an inert atmosphere during the heating, pyrolysis, and cooling steps. The analyzer should be capable of calculating the residue left after the test cycle is complete. Follow the manufacturer's instructions.

12.3 *Bulk Density*:

12.3.1 Bulk density gives some indication, on a volumetric basis, of how the resin may perform during feeding of molding and extrusion equipment. The PTFE resins have a tendency to compact during shipment and storage, and even though the material is broken up by screening or other means, original as-produced results may not be duplicated. Because of this tendency to pack under small amounts of compression or shear, Test Methods D 1895 are not applicable to these resins. Follow the procedure given in the following paragraph.

12.3.2 *Apparatus and Test Method*—Perform the test in accordance with instructions titled *bulk density* in Specification D 4894.

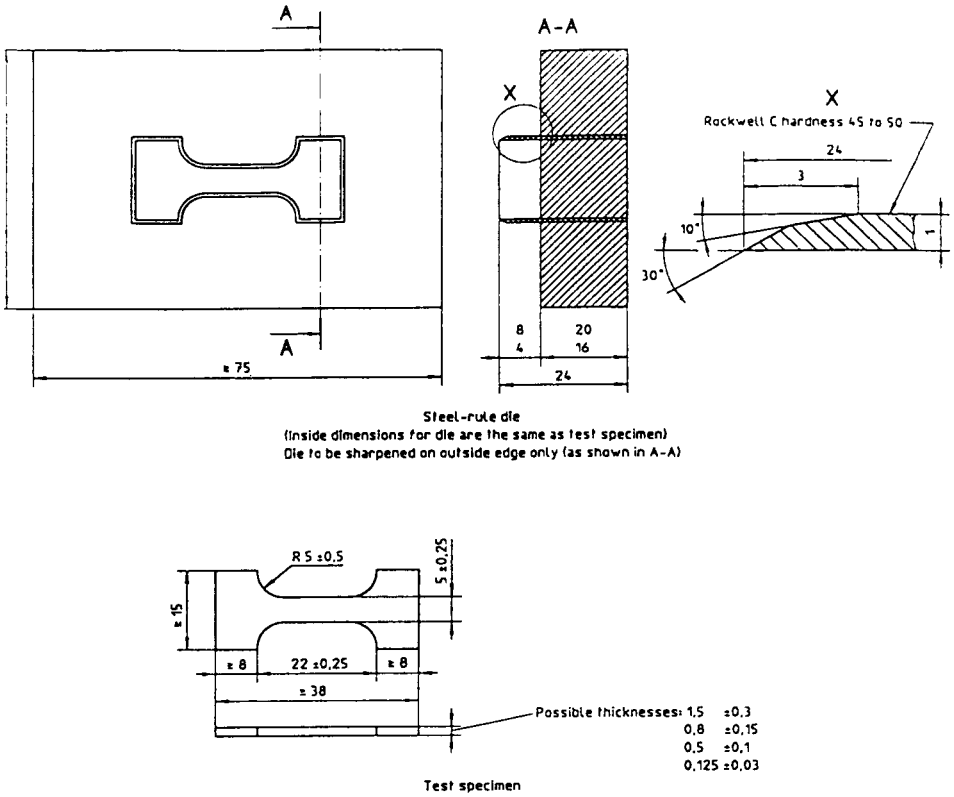
12.4 *Tensile Properties*:

12.4.1 *Procedure*:

Using the microtensile cutting die shown in Fig. 2, cut five tensile specimens from the slices prepared as in 9.3.2. Determine the tensile properties in accordance with the procedures described in Test Method D 638, except the initial jaw separation shall be 22.2 ± 0.13 mm (0.875 ± 0.005 in.), and the speed of testing shall be 50 mm (2 in.)/min. Clamp the specimens with essentially equal lengths in each jaw. Determine elongation from the chart, expressed as a percentage of the initial jaw separation.

12.4.1.1 *Sample Calculation*:

$$E = \frac{F - I}{I} \times 100 \quad (2)$$



NOTE 1—Dimensions in millimetres.

FIG. 2 Microtensile Die—ISO

where:

E = elongation, %,
F = final length of the jaw separation, mm, and
I = initial length of the jaw separation, mm.

12.5 *Density*:

12.5.1 Determine the density of specimens taken from the center part of the billet described in 9.2.2.

12.5.2 Make density determinations in accordance with the procedures described in Test Methods D 792. Add two drops of a wetting agent to the water in order to reduce surface tension and ensure complete wetting of the specimen. Report the value in grams per cubic centimetre.

13. Inspection

13.1 Inspection shall be conducted to determine that the material meets the requirements of this specification.

13.2 Manufacturers' test data for density, tensile strength, and elongation shall be available for each lot of product and shall be furnished at a frequency agreed upon between the seller and the purchaser.

14. Packaging and Package Marking

14.1 Packaging and packing provisions shall be in accordance with Practice D 3892.

14.2 *Marking*—Shipping containers shall be marked with the name of the material, type, and quantity contained therein, and appropriate cautionary information.

15. Precision and Bias

15.1 *Filler Content*—The test precision and bias are to be determined by round-robin testing.

15.2 *Tensile Properties*—Table 5 is based on a round robin conducted in 1985 through 1986 in accordance with Practice E 691, involving seven materials each tested by six laboratories. For each material, the sheeting from which the test specimens were to be cut was obtained from one source. Using a steel-rule die, one set of test specimens for each laboratory was cut by one of the laboratories. Sheeting and a duplicate die were furnished to each participating laboratory and used to cut a second set of test specimens. Each test result was the average of five individual determinations. Each laboratory obtained four test results on each material, two test results each on the specimens furnished and two on the specimens cut by the laboratory doing the testing.

15.2.1 The properties used in the analysis are: tensile strength and elongation at break. The stress-strain curves of the

TABLE 5 Precision Summary, Tensile Strength and Elongation at Break[A]

Material	Tensile Strength				
	Mean psi	CV_r%	CV_R%	I_r [B] %	I_R [C] %
Granular PTFE with 25 % Glass Fiber	2081	1.73	4.12	4.84	11.54
Granular PTFE	4801	2.79	8.85	7.81	24.78
Material	**Percentage Elongation at Break**				
	Mean psi	CV_r%	CV_R%	I_r [B] %	I_R [C] %
Granular PTFE with 25 % Glass Fiber	198	3.35	15.56	9.38	43.57
Granular PTFE	337	2.83	16.43	7.29	46.00

[A] Virgin granular PTFE data are given for comparison.
[B] $I_r = 2.8 \times CV_r$.
[C] $I_R = 2.8 \times CV_R$.

fluorocarbon polymers, including the glass-fiber filled material, are similar in shape. Based on advice from experts in statistical analysis of round-robin data, the information from the samples cut in one laboratory and tested by all the laboratories is not included in Table 5.

15.2.2 **Caution**—The following explanations of I_r and I_R (15.2.3-15.2.3.3) are intended only to present a meaningful way of considering the approximate precision of this test method. The data in Table 4 should not be applied rigorously to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories.

15.2.2.1 Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 15.2.3-15.2.3.3 would then be valid for such data.

15.2.3 *Concept of I_r and I_R* —If CV_r and CV_R have been calculated from a large enough body of data, and for test results that were averages from testing five specimens, the following applies:

15.2.3.1 I_r *: Repeatability*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the I_r value for that material.

15.2.3.2 I_R *: Reproducibility*—In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R value for that material.

15.2.3.3 Any judgment in accordance with 15.2.3.1 and 15.2.3.2 would have an approximate 95 % (0.95) probability of being correct.

15.2.4 Bias is systematic error which contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for this test procedure.

15.3 *Density*—Project X-15-289 has been established to review the status of and determine the need for additional precision and bias statements for tests in fluoropolymer standards.

16. Keywords

16.1 filled compounds; filled polytetrafluoroethylene; fluorocarbon polymers; fluoropolymer; fluoropolymer composites; polytetrafluoroethylene; PTFE

SUMMARY OF CHANGES

This section identifies the location of selected changes to this specification. For the convenience of the user, Committee D-20 has highlighted those changes that may impact the use of this specification. This section may also include descriptions of the changes or reasons for the changes, or both.

D 4745 – 97:

(*1*) Deleted reference to Specification D 1457 in 1.1 and throughout the entire specification.

(*2*) Added ISO equivalency statement as Note 3.

(*3*) Deleted MIL-STD-105 from 2.2.

(*4*) Added definitions for *free-flow resins (pelletized), pigmented compound, and standard flow resins (nonpelletized)* to Section 3.

(*5*) Updated Table 1 and Table 3, renamed to Table 1 and Table 2; combined Table 2 and Table 4 into Table 3.

(*6*) Added alternative method for filler content to Section 12.

(*7*) Changed Fig. 2 to ISO microtensile die.

(*8*) Changed *fabricator* and *supplier* to *seller* and *purchaser* in 13.2.