Designation: D7194 – 12

Standard Specification for Aerospace Parts Machined from Polychlorotrifluoroethylene (PCTFE)[1]

This standard is issued under the fixed designation D7194; 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 is intended to be a means of calling out finished machined parts ready for aerospace use. Such parts may also find use in selected commercial applications where there are clear benefits derived from the use of parts with known or controlled crystallinity, high molecular weight, good molecular weight retention during processing, dimensional stability in the finished part, and tightly controlled engineering tolerances.

1.2 This specification establishes requirements for parts machined from virgin, unplasticized, 100 % polychlorotrifluoroethylene (PCTFE) homopolymers.

1.3 This specification does not cover parts machined from PCTFE copolymers, PCTFE film or tape less than 0.25-mm (0.010-in.) thick, or modified PCTFE (containing pigments or plasticizers).

1.4 This specification does not allow parts containing recycled material.

1.5 The specification does not cover PCTFE parts intended for general use applications, in which control of dimensional stability, molecular weight, and crystallinity are not as important. For machined PCTFE parts intended for general use, use Specification D7211.

1.6 This specification classifies parts into three classes based upon intended uses and exposures: oxygen-containing media, reactive media, and inert media.

1.7 *Application*—PCTFE components covered by this specification are virgin, 100 % PCTFE resin, free of plasticizers and other additives. The components are combustion resistant in oxygen, dimensionally stable, and meet other specific physical characteristics appropriate for their end use. They are used in valves, regulators, and other devices in oxygen, air, helium, nitrogen, hydrogen, ammonia, and other aerospace media systems. The components typically are used as valve seats, o-rings, seals, and gaskets. They are removed and replaced during normal maintenance procedures. The components provide reliable sealing surfaces resulting in proper closure of valves and related devices and no leakage from the system into the environment. They will experience static mechanical loading, cyclic mechanical loading, temperatures ranging from cryogenic to 71 ºC (160 ºF), and pressures up to 68.9 MPa (10,000, psig) for oxygen and air media, and 103.4 MPa (15,000 psig) for inert media.

1.8 The values stated in SI units are to be regarded as standard. The values in parentheses are for information only.

1.9 The following precautionary caveat pertains only to the test methods 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 to determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D618 Practice for Conditioning Plastics for Testing

D638 Test Method for Tensile Properties of Plastics

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

D1430 Classification System for Polychlorotrifluoroethylene (PCTFE) Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D1708 Test Method for Tensile Properties of Plastics by Use of Microtensile Specimens

D2512 Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)

[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.

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[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.

D4591 Test Method for Determining Temperatures and Heats of Transitions of Fluoropolymers by Differential Scanning Calorimetry
D7211 Specification for Parts Machined from Polychlorotrifluoroethylene (PCTFE) and Intended for General Use
G86 Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments

2.2 *Federal Standards*[3]

NASA-STD-6001B Flammability, Offgassing, and Compatibility Requirements and Test Procedures—Mechanical Impact for Materials in Ambient Pressure LOX (Test 13A) and Mechanical Impact for Materials in Variable Pressure GOX and LOX (Test 13B)

3. Terminology

3.1 *Definitions:*

3.1.1 Terms are defined in accordance with Terminologies D883 and D1600 unless otherwise indicated.

3.1.2 *air media, n*—liquid air, pressurized air, and breathing air.

3.1.3 *cognizant engineering organization, n*—the company, agency, or other authority responsible for the system or component in which aerospace grade PCTFE is used. This, in addition to design personnel, may include personnel from material and process engineering, or quality groups and others as appropriate.

3.1.4 *inert media, n*—gaseous helium (GHe) and gaseous nitrogen (GN_2) up to 103.4 MPa (15,000 psig).

3.1.5 *oxygen media, n*—liquid oxygen (LOX) and gaseous oxygen (GOX) up to 68.9 MPa (10,000 psig).

3.1.6 *processing route, n*—the method whereby a thermoplastic is taken above its melting point and processed into a semifinished article, typically sheet or rod stock. For PCTFE, the common processing methods are extrusion and compression molding.

3.1.7 *reactive media, n*—ammonia (NH_3) up to 3.5 MPa (500 psig), gaseous hydrogen (GH_2) up to 46.2 MPa (6700 psig), and liquid hydrogen (LH_2) up to 2.8 MPa (400 psig).

4. Classification

4.1 Part shape and size shall be defined by the applicable purchase order.

4.2 The type of product shall be categorized by the intended use category:

4.2.1 *Type I* for use in air and oxygen media (see 3.1.2 and 3.1.5) at service pressures above 11.4 MPa (1650 psi) that require batch testing..

4.2.2 *Type II* for use in *(1)* air and oxygen media (at service pressures below 11.4 MPa (1650 psi), or at service pressures above 11.4 MPa (1650 psi) that do not require batch testing); or *(2)* inert and reactive media up to the pressures specified in 3.1.4 and 3.1.7.

4.2.3 *Type III* for use in media other than air, oxygen, GHe, GN_2, ammonia, GH_2 and LH_2, at service pressures specified by the cognizant engineering organization.

5. Ordering Information

5.1 All parts covered by this specification shall be ordered by Specification D7194, Type, as listed in Section 4, or as listed on the procurement drawing when Type is not specified explicitly..

6. Materials and Manufacture

6.1 Parts shall be made from as-polymerized resin meeting all requirements of Classification System D1430. Type *I*, *II* and *III* parts shall be fabricated from as-polymerized resin classified as meeting Classification System D1430, Group 01, Class 1, Grade 3.

6.2 Parts shall be made from virgin, unplasticized, 100 % polychlorotrifluoroethylene (PCTFE) homopolymer.

6.3 No recycled polymer or regrind shall be permitted.

6.4 The base material shall be free of all defects or contaminants that would be detrimental to final fabrication or performance of the finished parts.

7. Property Requirements

7.1 Specification values listed in this specification are minimum specification values. Any additional requirement for specific tests or data shall be specified at the time of the order.

8. General Requirements

NOTE 2—Unless otherwise specified in the purchase contract or order, the molder producing the semifinished article from which finished parts are made will be responsible for insuring the requirements in 8.6 are met. All other requirements listed in Section 8 pertain to the finished part, and therefore, will be the responsibility of the supplier of the finished, machined part.

8.1 Finished parts shall have a natural translucent appearance. The color shall be white or gray with no yellowing or other unnatural color.

8.2 Finished parts shall be free of voids, scratches, fissures, inclusions, or entrapped air bubbles that will affect serviceability. No particles (for example, black specks) shall be visible to the naked eye.

8.3 All finished parts are to be supplied after being annealed in accordance with 12.4.

8.4 No dimension of a finished part shall change more than 0.003 mm/mm (0.003 in./in.) measured at 23 ± 2 °C (73 ± 4 °F) before and after being held for 48 ± 5 h at 71 ± 5 °C (160 ± 9 °F), as determined by the method in 12.5.

8.5 Finished parts shall be made from semifinished articles having a zero strength time (ZST_{stock}) of 300 to 450 s (Grade 3) when determined in accordance with 12.1.

8.6 The maximum allowable ZST drop, ΔZST, shall be <20 % as determined in 12.1.4.

8.7 For nonmandatory requirements; namely, specific gravity variation, melting point range, and minimum tensile strength, that can be imposed to help ensure lot-to-lot

[3] Available from the U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: DE, Washington, DC 20401.

consistency, especially in regards to controlling the crystallinity of PCTFE semi-finished articles and parts machined therefrom subject to this Specification, refer to Nonmandatory Appendix X1.

9. Specific Requirements

9.1 Specific requirements for *Type I*, *II* and *III* material are summarized in Table 1.

9.2 *Type I*—Each batch or lot of parts, or semifinished articles from which the finished parts are made, shall meet the test and criteria of 12.2 or 12.3 in accordance with NASA-STD-6001B and Test Methods D2512 and G86 according to the discretion of the procuring agency. Finished parts meeting the criteria of 12.2 or 12.3 shall be assigned a unique dash number or part number.

9.3 *Types II and III*—No requirements exist either for mechanical impact testing and assigning of a unique dash number or part number.

10. Number of Tests

10.1 When the number of test specimens is not stated in the test method, a single determination may be made. If more than single determinations and separate portions of the same sample are made, the results shall be averaged. The final result shall conform to the requirements prescribed in this specification.

TABLE 1 Type I, II and III Material Requirements[A]

Type	Service Pressure (MPa (psi)) and affected parts	Media	Mechanical Impact Test Required	Assign Unique Part Number or Dash Number for the Finished Part
I	>11.4 (1650): only parts for which mechanical impact testing is required	oxygen, air	yes	yes
II	oxygen, air: < 11.4 (1650): all parts > 11.4 (1650): only parts for which no mechanical impact testing is required He: inert, reactive: pressures specified in 3.1.4 and 3.1.7: all parts	oxygen, air, inert, reactive	no	no
III	all pressures[B] and parts	other (for example, nitrox)	no	no

[A] Finished parts made from *Type I*, *II* and *III* material will be in an annealed condition, have a ZST_{stock} from 300 to 450 s, and have a ZST drop no greater than 20 %.
[B] As specified by the cognizant engineering organization.

11. Test Conditions

11.1 *Standard Temperature*—The tests shall be conducted at the standard laboratory temperature of 23 ± 2 ºC (73 ± 4 ºF) and 50 ± 10 % relative humidity per Practice D618.

12. Test Methods

12.1 *Zero Strength Time (ZST)*

12.1.1 The *ZST* apparent molecular weight of the as-molded semifinished article, denoted ZST_{stock} shall be determined by the molder procedures in Classification System D1430 using 50-mm (2-in.) long by 4.8-mm (3⁄16-in.) wide by 1.58-mm (0.062-in.) thick V-notched test strips. The ZST will be determined at 250 ± 1 ºC (482 ± 2 ºF) using a 7.5 ± 0.1-g weight.

12.1.2 When determining the *ZST* of the as-molded semi-finished article, ZST_{stock}, the molding method used to fabricate the semifinished article from which the finished, machined part is made shall be used. Several options exist for sample preparation:

12.1.2.1 For 1.58-mm (0.062-in.) thick sheet stock, *ZST* specimens may be die cut, and

12.1.2.2 For thicker sheet stock or rod stock, it will be necessary to machine the *ZST* specimens directly from the stock.

12.1.3 When determining the *ZST* of the as-polymerized resin, ZST_{resin}, V-notched test strips shall be cut from compression-molded sheet prepared in accordance with 9.1.2 in Classification System D1430.

12.1.4 The *ZST* drop, ΔZST, will be calculated as:

$$\Delta ZST = \frac{ZST_{resin} - ZST_{stock}}{ZST_{resin}} \times 100\,\% \quad (1)$$

12.2 *Mechanical Impacts/Ambient Pressure/LOX*

12.2.1 The decision to perform mechanical impact shall be made by the cognizant engineering organization within the procuring agency per the procurement drawing.

12.2.2 Test Method D2512 shall be used.

12.2.3 Samples shall be prepared in accordance with Test Method D2512.

12.2.4 Surface contamination shall be removed by filtered, gaseous nitrogen.

12.2.5 Test conditions (pressure and temperature) shall be at the ambient temperature of the test facility and the boiling point of LOX at that pressure.

12.2.6 Twenty separate samples shall be impacted at 98 J (72 lb_f) using a 12.7-mm (½-in.) diameter contact without any adverse reactions.

12.2.7 Reactions shall include a single audible, explosion, visual flash, burning or charring of samples, or major discoloration.

12.2.8 If one sample out of 20 reacts, 40 additional samples shall be tested without any reactions.

12.3 *Mechanical Impacts/Variable Pressure/GOX or LOX*

12.3.1 The decision to perform mechanical impact shall be made by the cognizant engineering organization within the procuring agency per the procurement drawing.

12.3.2 Test Method G86 shall be used.

12.3.3 Samples shall be prepared in accordance with Test Method G86.

12.3.4 Surface contamination shall be removed by filtered, gaseous nitrogen; or oxygen-compatible solvent, detergent and distilled water, as appropriate.

12.3.5 Pure GOX or LOX shall be used (choice depends on end-use application).

12.3.6 Test conditions shall be at the ambient temperature of the test facility and a pressure of 11.4 MPa (1650 psi).

12.3.7 Twenty separate samples shall be impacted at 98 J (72 lb_f) using a 12.7-mm (½-in.) diameter contact.

12.3.8 No advance reactions shall be observed. This includes a single audible explosion, visual flash, burning or charring of samples, or major discoloration.

12.3.9 If any sample fails, 40 additional samples shall be tested without any reactions.

12.4 *Annealing*

12.4.1 *Method A* (best dimensional stability) shall be the preferred annealing method. However, in cases where it imperative to maintain closest tolerances, *Method B* shall be preferred.

12.4.2 The appropriate annealing method shall be chosen by the cognizant engineering organization within the PCTFE part manufacturer unless specifically stated in the procurement drawing from the procuring agency.

12.4.2.1 *Method A*—For the best dimensional stability, parts shall be machined to final dimensions with appropriate allowances for contraction or expansion during annealing, annealed at 120 ± 5 ºC (248 ± 9 °F) for 2 h, cooled to room temperature, after which dimensional stability shall be determined (12.5).

12.4.2.2 *Method B*—For closest tolerances on a finished part, parts shall be machined to approximate dimensions, annealed at 120 ± 5 ºC (248 ± 9 °F) for 2 h, cooled to room temperature, finished to final dimensions, after which dimensional stability shall be determined (12.5).

NOTE 3—*Method B* is more practical for larger PCTFE parts.

12.5 *Dimensional Stability*

12.5.1 Linear dimensional change shall be determined by comparing selected dimensions measured before and after oven heating to simulate accelerated use. Pre- and post-heating dimensional measurements will be at several reference locations and at multiple locations or both distributed equidistantly about the specimen. After the initial dimensions have been made at 20 to 30 ºC (68 to 86 ºF), specimens will be placed in the preheated oven at 71 ± 5 ºC (160 ± 9 ºF), heated for 48 ± 5 h, after which the specimens will be removed and allowed to cool. After resting for 16 to 24 h (overnight), the final dimensions will be measured at 20 to 30 ºC (68 to 86 ºF), and the percent dimensional change, ΔL, calculated as:

$$\Delta L = \frac{L_f - L_i}{L_i} \times 100\,\% \qquad (2)$$

where:

L_i *and* L_f = initial and final dimensions before and after heating.

12.5.2 Measurement of dimensions will always be made with respect to a known direction such as the MD, TD, or sealing surface.

NOTE 4—Conventional metrological methods (coordinate measuring machines and dial micrometers) may not have the requisite sensitivity to detect dimensional changes in small parts of the order of 0.3 %. For example, a precision micrometer with 0.013-mm (0.0005-in.) sensitivity will not be able to detect dimensional changes smaller than 0.3 % if the original dimension is less than 4.3 mm (0.17 in.). An example would be PCTFE gaskets with thicknesses of the order of 0.50 mm (0.020 in.). In such cases, dimensional stability shall be indeterminate and shall not constitute grounds for nonprocurement.

13. Packaging and Package Marking

13.1 Finished parts shall be individually packaged in sealed envelopes, pouches, bags, or other containers in accordance with the manufacturer's commercial practice.

13.2 *Marking for Shipment*

13.2.1 Each container shall be legibly and permanently labeled with the following information:

13.2.1.1 *Material*—PCTFE parts,

13.2.1.2 *Type I, II or III*

13.2.1.3 *Annealing Method:* A or B

13.2.1.4 ZST_{stock}

13.2.1.5 *Specification number*—D7194

13.2.1.6 *Part manufacturer's name*

13.2.1.7 *Molder's name*

13.2.1.8 *Processing route and date*

13.2.1.9 *Lot number*

13.2.1.10 *Purchase order number*

13.2.2 For each batch or lot of PCTFE used in oxygen and air media operated at pressures greater than 11.4 MPa (1650 psi) (*Type I* material) that is subjected to mechanical impact testing, a unique dash number or part number shall be assigned by the part manufacturer to distinguish it from like PCTFE parts (*Type II* and *III* material) for which a unique dash number or part number shall not be assigned used in *(1)* oxygen and air media at pressures less than 11.4 MPa (1650 psi), *(2)* oxygen and air media at pressures greater than 11.4 MPa (1650 psi) but for which mechanical impact testing is not required, *(3)* inert and reactive media at the pressures specified in 3.1.4 and 3.1.7, and *(4)* other media at pressures specified by the cognizant engineering organization.

14. Keywords

14.1 annealing; dimensional stability; finished parts; PCTFE; polychlorotrifluoroethylene

APPENDIX

(Nonmandatory Information)

X1. SUPPLEMENTARY INFORMATION FROM CORRESPONDING SECTIONS OF PRACTICE (NOTE SECTION NUMBERS REFER TO PARTS OF THE PRACTICE)

X1.1 (Section 8.7) Supplementary Property Evaluations on PCTFE semifinished articles and finished parts machined therefrom

X1.1.1 The crystallinity, hence mechanical properties of PCTFE can vary significantly depending on molding method, molding temperature, residence time in the melt, thickness of the semifinished article (rod or sheet), and quenching method. Therefore, when questions arise as to the lot-to-lot consistency of PCTFE parts machined therefrom, it is recommended that some or all of the following property checks be performed.

X1.1.2 Specific gravity (X1.1.4) and melting point (X1.1.5) shall be performed on the machined, finished PCTFE part in its final, annealed condition per 12.4.

X1.1.3 Tensile strength and percent elongation (X1.1.6) shall be performed on test specimens prepared by machining operations, or die cutting, from the semifinished article (materials in sheet, plate, slab, or similar form).

X1.1.4 *Specific Gravity*

X1.1.4.1 The specific gravity variation, Δsp. gr. 23/23 ºC for finished parts shall not exceed ±0.01 (the weight percent crystallinity, W^c, of a given finished part shall not vary more than ±10 %).

X1.1.4.2 Specific gravity of finished parts shall be determined in accordance with Test Methods D792, Method A, with the following modifications. The submersion medium (deionized 18 MΩ cm water) is boiled; then one to two drops of Zonyl fluorosurfactant[4] (or equivalent) wetting agent is added per 100 mL of water. A magnifying glass is used to insure further that no air bubbles cling to submerged parts during weighings. Specimens shall also be free of internal voids in accordance with section 8.2. The test temperature shall be 23 ± 2 ºC (73 ± 4 ºF).

X1.1.4.3 Weight percent crystallinity, W^c, shall be calculated as:[5]

$$W^c = \frac{\rho_c}{\rho}\left(\frac{\rho - \rho_a}{\rho_c - \rho_a}\right) \times 100\,\% \qquad \text{(X1.1)}$$

$$\rho_c = \frac{1}{0.45563 + 0.8079 \times 10^{-4}\,T + 0.874 \times 10^{-7}\,T^2} \qquad \text{(X1.2)}$$

$$\rho_a = \frac{1}{0.47884 + 1.186 \times 10^{-4}\,T + 2.20 \times 10^{-7}\,T^2} \qquad \text{(X1.3)}$$

where:

ρ = density (specific gravity) of the finished part,
ρ_a = density of pure amorphous phase (ρ_a = 2.0760 g cm^{-3} at 23 ºC),
ρ_c = density of the pure crystalline phase (ρ_c = 2.1856 g cm^{-3} at 23 ºC), and
T = analysis temperature (°C).

X1.1.5 *Melting Point*

X1.1.5.1 The melting point of finished parts shall be determined to be in the range of 210 to 220 ºC (410 to 428 ºF), by determining the point at which all spherulitic order disappears as determined by polarized light microscopy in accordance with Test Method, alternatively, by determining the peak crystal melting temperature, $T_{m,\,peak}$, using DSC in accordance with Test Method D4591.

X1.1.6 *Tensile Strength and Elongation*

X1.1.6.1 The minimum tensile strength and percent elongation of the semifinished article from which the finished part was made shall be 33.1 MPa (4800 psi) and 100 %, respectively, as determined by Test Method D638, at a crosshead speed of 25 mm (1.0 in.) per minute. If product size does not permit testing in accordance with Test Method D638, tensile testing will be conducted in accordance with Test Method D1708 using machined microtensile specimens, also at a crosshead speed of 25 mm (1.0 in.) per minute.

[4] The sole source of supply of the wetting agent known to the committee at this time is E. I. Du Pont de Nemours and Company, DuPont Corporate Information Center, Chestnut Run Plaza 705/GS38, Wilmington, DE 19880-0705. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] Hoffman, J. D. and Weeks, J. J., "The Specific Volume and Degree of Crystallinity of Polychlorotrifluoroethylene," *Journal of Polymer Science*, 1958, 28, 472-475.