Designation: ~~D7194 – 12~~ D7194 – 19

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,~~ stability, controlled crystallinity, 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 °C (160 °F),~~ 71°C (160°F), 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~~13, 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~~ safety, health, and ~~health~~environmental practices and ~~to~~ determine the applicability of regulatory limitations prior to use.*

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

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1.10 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[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. 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
D1204 Test Method for Linear Dimensional Changes of Nonrigid Thermoplastic Sheeting or Film at Elevated Temperature
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)
D3045 Practice for Heat Aging of Plastics Without Load
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. Significance and Use

4.1 Given the thermal instability of PCTFE in the melt, the primary intention of this specification is to ensure adequate molecular weight retention during molding, whereby as-polymerized resin, typically powder or coarse granular material, is thermoformed into semi-finished rod or sheet stock, typically by extrusion or compression molding.

4.2 Given the relatively low glass transition temperature of PCTFE ($T_g \approx 55°C$ (130°F)), the second intention of this specification is to ensure good dimensional stability of finished articles subjected to temperature excursions during service that approach or exceed the T_g.

4.3 Because the crystallinity can vary widely in PCTFE, which in turn can affect the performance of a finished PCTFE part in its intended application, nonmandatory guidance is given in Appendix X1. This guidance cites recommended ASTM Test Methods that measure the specific gravity (X1.1.4), melting point (X1.1.5), and tensile properties (X1.1.6) of PCTFE. Control of these properties is used to established consistency between different lots of finished parts where either the starting resin, molding method, thickness of the semifinished article, or machining method change.

4.4 Finished parts are categorized based on whether annealing of the finished parts is not performed (*Method A*), or is performed (*Methods B and C*), depending on the dimensional stability of a statistical sample of unannealed finished parts.

[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.

[3] Available from ~~the~~ U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, ~~Mail Stop: DE,~~ Washington, DC ~~20401.~~20401-0001, http://www.access.gpo.gov.

5. Classification

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

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

5.2.1 *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..~~testing.

5.2.2 *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.

5.2.3 *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.

6. Ordering Information

6.1 All parts covered by this specification shall be ordered by Specification D7194, Type, as listed in Section ~~4~~5, or as listed on the ~~procurement drawing~~ purchase contract or order when Type is not specified ~~explicitly..~~explicitly.

7. Apparatus

7.1 *Oven*—A convection oven capable of maintaining a temperature of 100 to 200 ± 2°C.

8. Materials and Manufacture

8.1 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.

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

8.3 No recycled polymer or regrind shall be permitted.

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

9. Property Requirements

9.1 Numerical values listed in this specification for properties or property changes are minimum values.

9.2 All requirements stipulated in Sections 9 and 10 shall be met, unless otherwise specified in the purchase contract or order. Any exceptions to the requirements stipulated in Sections 9 and 10 shall be noted in 14.2.1.12.

9.3 ~~Specification values listed in this specification are minimum specification values. Any additional requirement for specific tests or data~~ Any nonmandatory tests or data, such as those cited or alluded to in the nonmandatory Appendix X1, shall be specified at the time of the order.

10. 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~~ is responsible for insuring the requirements in ~~8.6~~10.4 are met. All other requirements listed in Section ~~8~~10 pertain to the finished part, and therefore, ~~will be~~ are the responsibility of the supplier of the finished, machined part.

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

10.2 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 ºC (73 ± 4 ºF) before and after being held for 48 ± 5 h at 71 ± 5 ºC (160 ± 9 ºF), as determined by the method in 12.5.~~

10.3 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~~13.1.

10.4 The maximum allowable ZST drop, ΔZST, shall be <20 % as determined in ~~12.1.4~~13.1.4.

10.5 No critical engineering dimension in a finished part shall change by more than 0.01 mm/mm (0.01 in./in.) when measured at 23 ± 2°C (73.4 ± 3.6°F) after being held for a predetermined time at 100 ± 2°C (212 ± 3.6°F) as determined in accordance with 13.2.

10.6 Finished parts meeting the general requirements of 10.1 to 10.5 shall be categorized as parts suitable for *Method A* (no annealing required).

10.7 Finished parts meeting the general requirements of 10.1 to 10.4, but which fail general requirement 10.5 (ΔL > 1.0 %), shall be categorized as *Method B* parts (annealing required to achieve best dimension stability) or *Method C* parts (annealing required to achieve best engineering tolerances). Annealing for *Methods B and C* parts shall be performed in accordance with 13.3.

10.8 For nonmandatory requirements; namely, specific gravity variation, melting point range, and minimum tensile strength, that can be imposed to help ensure lot-to-lot consistency, especially in regards to controlling the crystallinity of PCTFE semi-finished articles and parts machined therefrom subject to this ~~Specification,~~specification, refer to Nonmandatory Appendix X1.

11. Specific Requirements

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

11.2 *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~~13.4 or ~~12.3~~13.5 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~~13.4 or ~~12.3~~13.5 shall be assigned a unique dash number or part number.

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

12. Test Specimens and Number of Tests

12.1 In cases where measured values depend on orientation, for example, tensile properties and dimensions of test specimens made from extruded material, the extrusion direction relative to the measurement direction shall be tracked.

12.2 When the number of test specimens is not stated in the test method, a single determination may be made. If more than a single determination or determinations ~~and~~on 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.

~~11. Test Conditions~~

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

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.

13. Test Methods

13.1 *Zero Strength Time (ZST)*

13.1.1 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 °C~~ 1°C (482 ± ~~2 °F)~~ 2°F) using a 7.5 ± 0.1-g weight.

13.1.2 When determining the *ZST* of the as-molded semifinished 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:

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

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

13.1.3 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.

13.1.4 The *ZST* drop, Δ*ZST*, will be calculated as:

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

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

13.2 *Dimensional Stability:*

13.2.1 *Conditioning Temperature and Humidity*—Test specimens used to determine dimensional stability shall be conditioned before test at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 10 % relative humidity in accordance with Test Method D1204 or Practice D618 unless otherwise specified in the purchase contract or order. In cases of disagreement, the tolerances shall be ±2°C (±3.6°F) and ±10 % relative humidity.

13.2.2 *Conditioning Time*—Condition test specimens 7 mm (0.25 in.) or under in thickness in for a minimum of 40 h, and test specimens over 7 mm in thickness in for a minimum of 88 h immediately prior to testing in accordance with Procedure A of Practice D618.

13.2.3 Dimensional stability shall be determined on a statistical sample of finished parts chosen per the discretion of the finished part manufacturer.

NOTE 3—Good practice when determining the dimensional stability of parts machined from rod stock is to measure representative part(s) taken from rod ends, and if results differ, to measure additional representative part(s) taken midway between the rod ends.

13.2.4 Linear dimensional change shall be determined by comparing selected dimensions measured before and after heating in an oven. Measurements of dimensions are made at locations distributed equidistantly about the specimen. After initial dimensions are measured, specimens are placed in a preheated oven at 100 ± 2°C (212 ± 3.6°F) for 2 h, after which the specimens are removed and allowed to cool for a minimum of 1 h at the same temperature and humidity used during the initial conditioning process. The percent dimensional change, Δ*L*, is calculated as:

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

where:

L_i *and* L_f = initial and final dimensions (length, thickness, inner diameter, outer diameter, etc., as applicable) before and after heating.

A negative value for Δ*L* denotes shrinkage, and a positive value indicates expansion.

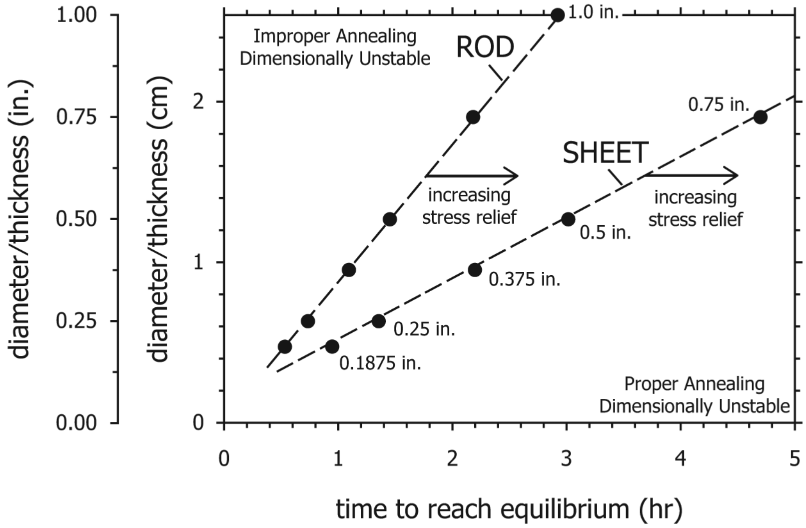
NOTE 4—Heating at 100°C for 2 h is deemed sufficient to cause adequate dimensional relaxation in PCTFE parts with thicknesses and diameters less than 0.9 to 1.7 cm (0.35 to 0.67 in.), respectively, while heating for 30 min is deemed sufficient for PCTFE part made from sheet or film with thicknesses less than about 3 mm (0.125 in.) (see Fig. 1).

NOTE 5—Finished parts which exhibit Δ*L* < 1.0 % after heating at 100°C for 2 h shall only be qualified for use in applications where temperature excursions above 100°C are not expected. To qualify finished parts for higher service temperatures, dimensional stability shall be determined by heating specimens in the oven for 2 h at the higher temperature.

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

NOTE 6—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 1.0 %. For example, a precision micrometer with 0.0127 mm (0.0005 in.) sensitivity will not be able to detect dimensional changes smaller than 1.0 % if the original dimension is less than 1.27 mm (0.050 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.

13.3 *Annealing—*



Source: Waller, J. M., Julien, H. L., Newton, B. E., Beeson, H. D., "Proactive Mitigation of PCTFE-Related Ignition Hazards in Oxygen Systems: I. Development of a Voluntary Consensus Material Specification to Control Property Variation in Finished PCTFE Parts," Journal of ASTM International, 3 (9), 2006.

FIG. 1 Time to Reach Thermal Equilibrium for Various Polychlorotrifluoroethylene Semifinished Rod Diameters and Sheet Thicknesses

13.3.1 Method A (no annealing required) shall be the preferred method. However, in cases where unacceptable dimensional stability is observed on as-machined finished parts as determined in 13.2, Method B (best dimensional stability) shall be used instead of Method A. When annealing of finished parts is required, and it imperative to maintain closest tolerances, Method C shall be preferred over Method B.

13.3.2 The appropriate annealing method shall be chosen by the cognizant engineering organization within the PCTFE part manufacturer unless specifically stated in the purchase contract or order.

13.3.3 Finished parts fall into two categories depending on the result obtained in 13.2 (see Fig. 2):

13.3.3.1 *Method A*, $\Delta L < 1.0$ % for all critical engineering dimensions, annealing is not required.

13.3.3.2 *Methods B and C*, $\Delta L > 1.0$ % for any critical engineering dimension, annealing of finished parts is required.

13.3.4 The effect of annealing (*Methods B and C*) on dimensional stability shall be determined on a statistical sample of finished parts chosen per the discretion of the finished part manufacturer.

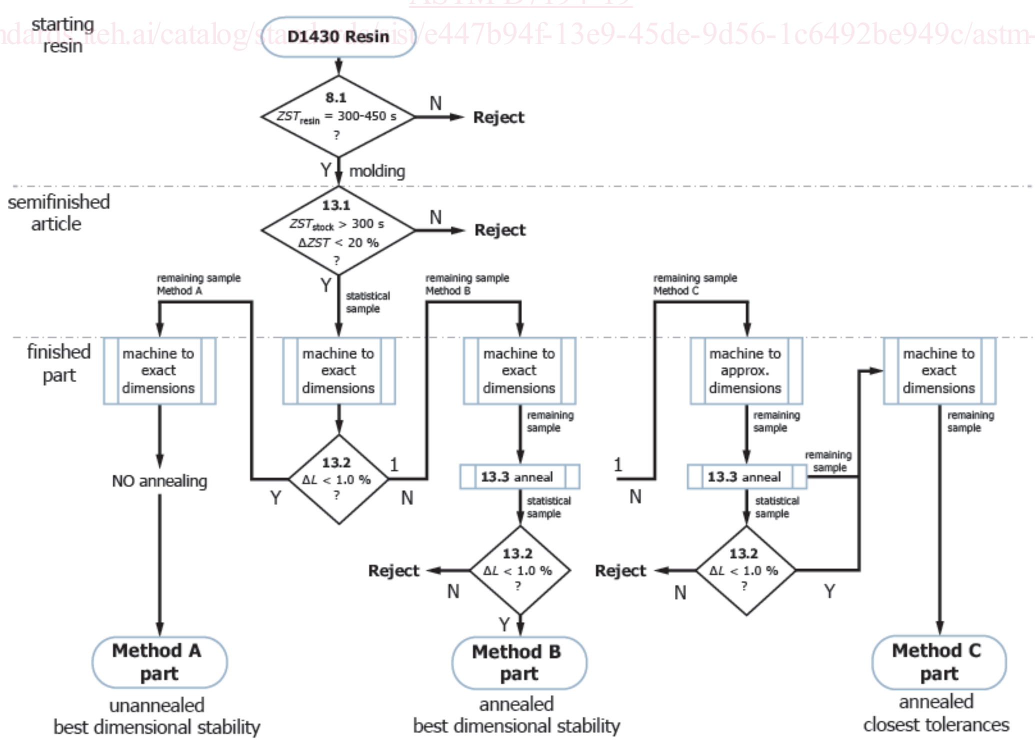


FIG. 2 Flow Chart for D7194 Polychlorotrifluoroethylene Methods A, B, and C