Designation: F 2026 – 00

Standard Specification for Polyetheretherketone (PEEK) Resins for Surgical Implant Applications[1]

This standard is issued under the fixed designation F 2026; 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 polyetheretherketone (PEEK) resins in virgin forms as supplied by a vendor (flakes, pellets, blocks, and so forth). It provides requirements and associated test methods for these thermoplastics when they are to be used in the manufacture of intracorporal devices such as surgical implants or components of surgical or dental devices.

1.2 As with any material, some characteristics may be altered by the processing techniques (molding, extrusion, machining, assembly, sterilization, and so forth) required for the production of a specific part or device. Therefore, properties of fabricated forms of these resins should be evaluated using test methods which are appropriate to ensure safety and efficacy as agreed upon by the vendor, purchaser, and regulating bodies.

1.3 The properties included in this specification are those applicable for PEEK resins only. Fabricated forms, material or forms containing colorants, fillers, processing aids, or other additives, as well as polymer blends which contain PEEK, are not covered by this specification.

1.4 This specification is designed to recommend physical, chemical, and biological test methods to establish a reasonable level of confidence concerning the performance of virgin PEEK resins for use in medical devices. The properties listed should be considered in selecting material in accordance with the specific end-use requirements.

1.5 When evaluating material in accordance with this specification, hazardous materials, operations, and equipment may be involved. *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies[2]

D 256 Test Method for Determining the Pendulum Impact Resistance of Notched Specimens of Plastics[3]

D 570 Test Method for Water Absorption of Plastics[3]

D 621 Test Methods for Deformation of Plastics Under Load[4]

D 638 Test Method for Tensile Properties of Plastics[3]

D 648 Test Method for Deflection Temperature of Plastics Under Flexural Load[3]

D 695 Test Method for Compressive Properties of Rigid Plastics[3]

D 696 Test Method for Coefficient of Linear Thermal Expansion of Plastics Between −30C and 30C[3]

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[3]

D 792 Test Methods for Density and Specific Gravity Relative Density) of Plastics by Displacement[3]

D 955 Test Method for Measuring Shrinkage from Mold Dimensions of Molded Plastics[3]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[3]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[3]

D 1898 Practice for Sampling of Plastics[5]

D 3417 Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC)[6]

D 3418 Test Method for Transition Temperatures of Polymers by Thermal Analysis[6]

D 4000 Classification System for Specifying Plastic Materials[6]

F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[7]

2.2 *ISO Standards:*

ISO 1628/1 Plastics—Guidelines for the Standardization of Methods for Determination of Viscosity Number and

[1] This specification is under the jurisdiction of ASTM Committee F-4 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.11 on Polymeric Materials.

Current edition approved May 10, 2000. Published August 2000.

[2] *Annual Book of ASTM Standards*, Vol 10.01.

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

[4] Discontinued; see *1993 Annual Book of ASTM Standards*, Vol 08.01.

[5] Discontinued; see *1997 Annual Book of ASTM Standards*, Vol 08.02.

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

[7] *Annual Book of ASTM Standards*, Vol 13.01.

Limiting Viscosity Number of Polymers in Dilute Solution—Part 1: General Conditions[8]

ISO 1133 Plastics—Determination of the Melt Mass-Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics[8]

ISO 10993 Biological Evaluation of Medical Devices, Parts 1-12[8]

2.3 *Other Document:*

United States Pharmacopeia, Vol. XXI, or latest edition[9]

Food and Drug Administration, Regulation 21 CFR 177.1580[10]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *fabricated forms*—those items into which the virgin forms may be converted. These include shapes and forms produced by means of machining, extruding, and compression molding virgin forms into a subsequent entity (for example, rods, slabs, sheets, film, or complex shaped parts and devices).

3.1.2 *formulated compound*—the PEEK materials, parts, or devices fabricated from virgin forms in such a way as to contain intentional or unintentional adjuvant substances.

3.1.3 *virgin forms*—that form of the PEEK resin as obtained by the synthesizer. It typically will be in the form of pellets, chips, or blocks. It is the material from which rods, slabs, sheets, films, or specific parts and devices are fabricated.

4. Classification

4.1 The PEEK resins in the scope of this specification are pure semicrystalline homopolymers consisting of phenylene rings connected by ether (E) and carbonyl (or ketone, K) groups along the polymer chain (see Appendix X2). Their polymeric structures are defined by the repeating unit EEK.

4.2 Types of PEEK plastics, molding, and extrusion grades are described in Classification System D 4000.

5. Properties

5.1 The PEEK resins used in medical applications may comply with the Food and Drug Administration (FDA) regulation 21 CFR 177.1580 which covers both wet and dry food contact applications.

[8] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[9] Available from U.S. Pharmacopeial Convention, Inc., Easton, PA.

[10] Available from the Food and Drug Administration.

TABLE 1 Required Properties of Virgin Resin

Parameter	Method	Requirement
T_g, ° C	DSC, 20°C/min, sealed sample	125 - 165
T_m, ° C	DSC, 20°C/min, sealed sample	320 - 360
Viscosity number, min, mL/g	ISO 1628, concentrated sulfuric acid, 0.5 % w/v, 25°C	77
Melt volume flow rate, cm^3/10 min	ISO 1133, 400°C, 10 Kg	25 - 60
Total heavy metals as lead, max, %	US Pharmacopeia, Test 231	0.1

TABLE 2 Typical Properties of Fabricated Forms

Parameter	Method	Requirement
Density, kg/m^3	ISO D 1505	1280 - 1320
Tensile Strength, min, MPA, Yield Break	Test Method D 638, Type IV, 5.08 cm/min	90 70
Percent elongation, min, %	Test Method D 638, Type IV, 5.08 cm/min	40
Izod Impact Strength, min, ft-lbf/in.	Test Method D 256, 0.254-cm depth, 0.025-cm radius	1.1
Deformation Under Load, max , %	Test Methods D 621 (A), 7 MPa for 24 h, 23°C, after 90-min recovery	2

5.2 The infrared spectrum[11] of these materials is characteristic of their molecular repeating units. A representative spectrum is listed in Appendix X3. The PEEK resin shall yield an infrared transmittance spectrum which exhibits major bands only at the wavelengths listed for a standard reference spectrum of that material.

5.2.1 The infrared spectrum, as used in this specification, is to identify the specific type of poly aryl ether ketone (PAEK) present and does not necessarily indicate an acceptable degree of material purity.

5.2.2 The presence of additional bands in the sample's infrared spectrum compared to that of the reference material may indicate a different PAEK or impurities, or both.

5.3 The physical and chemical property requirements for the virgin resin are listed in Table 1. If additional characteristics are necessary because of a specific application, the procedures referenced in 5.7 are recommended, or as agreed upon between the vendor and the purchaser.

5.4 The viscosity requirements listed in Table 1 may be supplemented, or replaced, by rheological or complex viscosity data as agreed upon between the vendor and the purchaser.

5.5 The chemical, physical, and mechanical properties of fabricated forms are related to the processes utilized in producing the fabricated form (for example, molding, machining, sterilization, and so forth). Additionally, the properties necessary for a particular device to perform properly will vary from one device type to another. Table 2 lists some typical properties of non-sterilized injection molded material.

5.6 Test specimens shall be fabricated (machined, injection molded, and so forth) from the virgin resin, or finished part, in such a way to effectively represent the material characteristics of the non-sterilized finished part.

5.7 Tests and test procedures shall be such as to ensure a high level of control and characterization of the virgin resin as received from the supplier. The following are some test methods that may be appropriate: Test Method D 149, Test Method D 256, Test Method D 570, Test Method D 638, Test Method D 648, Test Method D 695, Test method D 696, Test Methods D 790, Test Methods D 792, Test Method D 955, Test Method D 1238, Test Method D 1505, Test Method D 3417, Test Method D 3418, and Classification System D 4000.

6. Sampling

6.1 The material should be sampled in accordance with the

[11] Silverstein, R. M., Bassler, G. C., and Morrill, T. C., "Spectroscopic Identification of Organic Compounds," 5th ed., John Wiley & Sons, New York, NY.

standard sampling procedures, such as those described in Practice D 1898, or other sampling techniques unless otherwise agreed upon between the consumer and the supplier.

7. Biocompatibility

7.1 Biocompatibility of PEEK resins and implant devices made using these materials shall be determined in accordance with Practice F 748 or the ISO 10993 series, unless otherwise agreed upon between the packager and the consumer and regulating bodies.[12]

[12] Other useful references for testing biocompatibility of materials include:

Autian, J., "Toxicological Evaluation of Biomaterials: Primary Acute Toxicity Screening Program," *Journal of Artificial Organs*, Vol 1, No. 1, 1977, p. 53.

Autian, J., "The New Field of Plastic Toxicological Methods and Results," *CRC Critics Review in Toxicology*, 1973, p. 18.

Homsy, C. A., Ansevin, K. D., O'Brannon, W., Thompson, S. H., Hodge, R., and Estrella, M. E., "Rapid In Vitro Screening of Polymers for Biocompatibility," *Journal of Macromolecular Science Chemistry*, Vol A4, No. 3, May 1970, pp. 615-634.

Rice, R. M., Hegyeli, A. F., Gourlay, S. J., Wade, C. W. R., Dillon, J. G., Jaffe, H., and Kulkarni, R. K., "Biocompatibility Testing for Polymers: In Vitro Studies With In Vivo Correlation," *Journal of Biomedical Materials Research*, Vol 12, 1978, p. 43.

8. Keywords

8.1 PEEK; polyetheretherketone

APPENDIXES

(Nonmandatory Information)

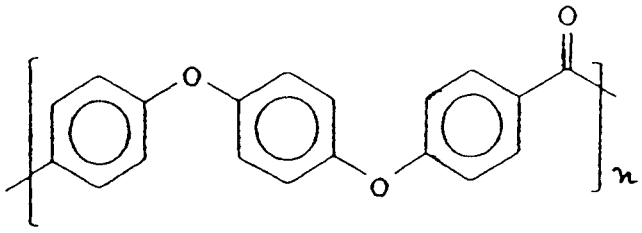
X1. RATIONALE

X1.1 The PEEK resins may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of PEEK resins may be sterilized. Sterilization methods successfully used include steam, ethylene oxide, and irradiation. Repeated sterilization may weaken parts fabricated of any plastic material. The number of times a given part may be sterilized safely without fear of subsequent failure depends on a number of factors including the molecular weight of resin and design, fabrication, intended function, and method of sterilization of the device. Therefore, it is imperative that the manufacturer test the device in order to determine the maximum number of sterilization cycles to which it can be safely subjected.

X1.2 The potential to develop a significant level of crystallinity is an important characteristic of these materials. Performance characteristics are related to the percent crystallinity. Certain additives and processes (for example, excessive cross linking) can limit these materials' ability to crystallize. Therefore, this feature of the resin and its fabricated form should be evaluated, using appropriate test methods, to ensure efficacy.

X1.3 A formulated compound or fabricated part or device may contain optional adjuvant substances required for the fabrication or intended use of the end product. The biocompatibility of these adjuvant substances, and subsequent formulated compounds, parts, and devices shall be established in accordance with Practice F 748 or the ISO 10993 series.

X2. CHEMICAL STRUCTURE OF PEEK



X3. REPRESENTATIVE INFRARED SPECTRA OF PEEK