Designation: F2026 – 16

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

This standard is issued under the fixed designation F2026; 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) polymer in virgin forms as supplied by a vendor (pellets, powder, fabricated forms, and so forth). It provides requirements and associated test methods for these thermoplastics when they are to be used in the manufacture of intracorporeal devices such as surgical implants or components of surgical or dental devices.

1.2 The properties included in this specification are those applicable for PEEK polymers only. Indicated properties are for fabricated forms. Materials or forms containing colorants, fillers, processing aids, or other additives, as well as polymer blends which contain PEEK, or reclaimed materials, are not covered by this specification.

1.3 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 polymers for use in medical implant devices.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[2]

D256 Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics
D638 Test Method for Tensile Properties of Plastics
D648 Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position
D695 Test Method for Compressive Properties of Rigid Plastics
D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D1505 Test Method for Density of Plastics by the Density-Gradient Technique
D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry
D4000 Classification System for Specifying Plastic Materials
F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

2.2 *ISO Standards:*[3]

ISO 178 Plastics—Determination of Flexural Properties
ISO 180 Plastics—Determination of Izod Impact Strength
ISO 527 Plastics—Determination of Tensile Properties—Part 1: General Principles
ISO 1183 Plastics—Methods for Determining the Density of Non-cellular Plastics—Part 2: Density Gradient Column Method
ISO 10993 Biological Evaluation of Medical Devices, Parts 1-12
ISO 13485 Medical Devices—Quality Management Systems—Requirements for Regulatory Purposes

2.3 *Other Documents:*

United States Pharmacopeia, Vol. XXI, or latest edition[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

Current edition approved April 1, 2016. Published May 2016. Originally approved in 2000. Last previous edition approved in 2014 as F2026 – 14. DOI: 10.1520/F2026-16.

[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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852-1790, http://www.usp.org.

3.1.1 *fabricated forms, n*—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, fibers, tubes, rods, slabs, sheets, film, or complex shaped parts and devices).

3.1.2 *formulated compound, n*—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, n*—the initially delivered form of the polymer as synthesized from its monomers prior to any processing or fabrication into a medical device. The provided resin is typically in the form of pellets, granules, or powder and is the material from which fibers, tubes, rods, slabs, sheets, films, or specific parts and devices are fabricated.

4. Classification

4.1 The PEEK polymer in the scope of this specification is a pure semicrystalline homopolymer consisting of phenylene rings connected by ether (E) and carbonyl (or ketone, K) groups along the polymer chain (see Appendix X1). Its polymeric structure is defined by the repeating unit EEK.

4.2 Types of PEEK plastics, molding, and extrusion grades are described in Classification System D4000.

5. Properties

5.1 The properties listed below shall be considered in selecting material(s) in accordance with the specific end-use requirements.

5.2 The infrared spectrum[5] of these materials is characteristic of their molecular repeating units. A representative spectrum is listed in Appendix X3. The PEEK polymer shall yield an infrared 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 polymer are listed in Table 1. If additional characteristics are necessary because of a specific application, the procedures referenced in Section 2 are recommended, or as agreed upon between the vendor and the purchaser.

5.4 The viscosity requirements will vary depending upon the grade and test method. The method and requirements shall be 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 fabricated forms.

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

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

5.7 Tests and test procedures shall be such as to ensure a high level of control and characterization of the virgin polymer as received from the supplier. The test methods referenced in Section 2 may be appropriate (Test Methods D648 and D695).

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

TABLE 1 Required Properties of Virgin Resin

Parameter	Method	Requirement
Glass transition temperature, T_g (°C)	DSC,[A] 20°K/min, sealed sample, T_g taken on second reheat, D3418	125 - 165
Melt temperature, T_m (°C)	DSC, 20°K/min, sealed sample, T_m taken as max point on reheat endotherm, D3418	320 - 360
Recrystallization temperature, T_c (°C)	DSC, 20°K/min, sealed sample, T_c taken as max point on cooling exotherm, D3418	260 - 320
Viscosity	As agreed per 5.4	As agreed per 5.4
Infrared spectrum	As agreed per 5.2	As agreed per 5.2
Total heavy metals (Ag, As, Bi, Cd, Cu, Hg, Mo, Pb, Sb, and Sn), max, ppm	US Pharmacopeia, Test 233	<100

[A] Differential Scanning Calorimetry (DSC).

TABLE 2 Required Properties of Fabricated Forms

Parameter	ISO Methods and Requirements		ASTM Methods and Requirements	
Density, kg/m^3	ISO 1183	1280 - 1320	ASTM D792 or ASTM D1505	1280 - 1320
Tensile Strength:	ISO 527, Type 1B, 50 mm/min		ASTM D638, Type IV, 5.08 cm/min	
at yield (zero slope), min, MPa		90		90
at break, min, MPa		70		70
Elongation at break,[A] min, %	ISO 527, Type 1B, 50 mm/min	5	ASTM D638, Type IV, 5.08 cm/min	5
Flexural strength, min, MPa	ISO 178	110	ASTM D790	110
Flexural modulus, min, GPa	ISO 178	3	ASTM D790	3
Impact strength, notched Izod, min	ISO 180	4 (kJ/m^2)	ASTM D256, 0.254 cm depth, 0.025 cm radius	50 (J/m)

[A] Use an extensometer for measuring strain and calculating percent elongation.

5.7.1 With reduced crystallinity, certain polymers have been shown to be more susceptible to environmental stress cracking.[6,7] Depending upon the implant application, the end user should evaluate the material for environmental stress cracking resistance.[6,7]

6. Sampling

6.1 The material should be sampled in accordance with standard sampling procedures or other sampling techniques unless otherwise agreed upon between the consumer and the supplier.

7. General Requirements

7.1 *Quality System Requirements*—The PEEK polymer and fabricated forms as described in the scope of this specification should be produced in accordance with an ISO 13485-certified quality management system.

7.2 *Biocompatibility*—PEEK has been shown to produce a well-characterized level of biological response following long term clinical use.[8] The results of these studies and the clinical history indicate an acceptable level of biological response in the applications in which the material has been utilized.[8] When new applications, or modification to the material or physical forms of the materials are being contemplated, biocompatibility shall be determined in accordance with Practice F748 or the ISO 10993 series, unless otherwise agreed upon between the packager and the consumer and regulating bodies. A recent review article[8] includes an extensive bibliography regarding the biocompatibility of PEEK biomaterials.

8. Keywords

8.1 PEEK; polyetheretherketone

[6] Hay, J. N., and Kemmish, D. J., "Environmental Stress Crack Resistance and Absorption of Low-Molecular-Weight Penetrants by Poly(Aryl Ether Ether Ketone)," *Polymer*, Vol 29, April 1988, pp. 613–618.

[7] Srivastava, A. P., Depke, N., and Wolf, C. J., "Environmental Stress Deformation of Poly(ether ether ketone)," *J. Applied Polymer Science*, Vol 66, 1997, pp. 725–731.

[8] Kurtz, S.M. and Devine, J.N., "PEEK Biomaterials in Trauma, Orthopedic, and Spinal Implants," *Biomaterials*, Vol 28, No. 32, 2007, pp. 4845-4869.

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

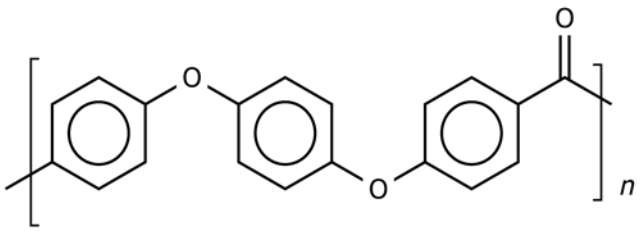
X1.1 The PEEK polymers may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of PEEK polymers may be sterilized. Sterilization methods successfully used include steam, ethylene oxide, and irradiation. Repeated sterilization may 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 the polymer 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.

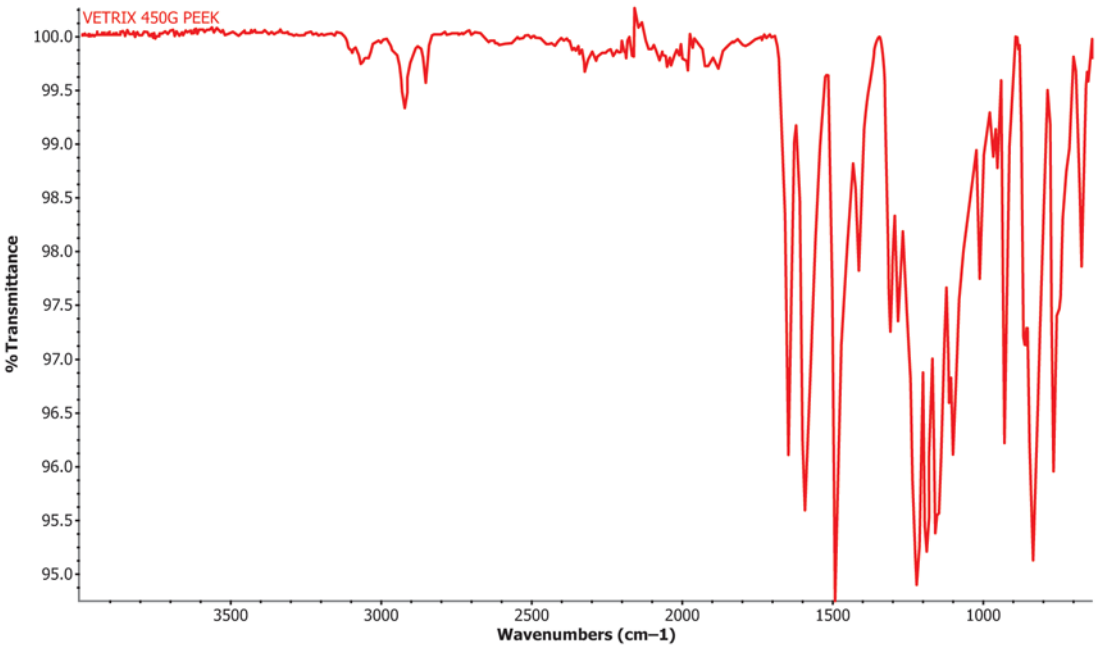
Therefore, this feature of the polymer and its fabricated form should be evaluated, using appropriate test methods, to ensure efficacy.

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 F748 or the ISO 10993 series.

X2. CHEMICAL STRUCTURE OF PEEK



X3. REPRESENTATIVE INFRARED SPECTRA OF PEEK



RELATED MATERIAL

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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.