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Standard Specification for Polyaryletherketone (PAEK) Polymers for Surgical Implant Applications[1]

This standard is issued under the fixed designation F1579; 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.

ε^1 NOTE—Editorial changes were made throughout in June 2003.

1. Scope

1.1 This specification covers polyaryletherketone (PAEK) polymers in virgin forms as supplied by a vendor (pellets, powder, 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 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 polymers should be evaluated using test methods that 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 PAEK polymers only. Fabricated forms, material or forms containing colorants, fillers, processing aids, or other additives, as well as polymer blends that contain PAEK, 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 unfilled PAEK polymers for use in medical devices. The properties listed should be considered in selecting material according to the specific end-use requirements.

1.5 When evaluating material to 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]

D149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies

D256 Test Methods for Determining the Izod Pendulum Impact Resistance of Plastics

D570 Test Method for Water Absorption of Plastics

D621 Test Methods for Deformation of Plastics Under Load

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

D696 Test Method for Coefficient of Linear Thermal Expansion of Plastics Between -30°C and 30°C with a Vitreous Silica Dilatometer

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

D955 Test Method of Measuring Shrinkage from Mold Dimensions of Thermoplastics

D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

D1505 Test Method for Density of Plastics by the Density-Gradient Technique

D1898 Practice for Sampling of Plastics

D3417 Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC)[3]

D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by

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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.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

Differential Scanning Calorimetry
D4000 Classification System for Specifying Plastic Materials
F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices
F1876 Specification for Polyetherketoneetherketoneketone (PEKEKK) Resins for Surgical Implant Applications
F2026 Specification for Polyetheretherketone (PEEK) Polymers for Surgical Implant Applications

2.2 *ISO Documents:*

ISO 1133 Plastics—Determination of the Melt Mass-Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics[4]

ISO 1628/1 Plastics, Guidelines for the Standardization of Methods for Determination of Viscosity Number and Limiting Viscosity Number of Polymers in Dilute Solution—Part 1: General Conditions[4]

ISO 10993 Biological Evaluation of Medical Devices, Parts 1–12[4]

2.3 *Other Documents:*

United States Pharmacopeia, Vol. XXI, or latest edition[5]

Food and Drug Administration Regulation 21 CFR 177.2415[6]

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, complex shaped parts and devices).

3.1.2 *formulated compound*—PAEK 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 PAEK polymer as obtained by the synthesizer after removal of residual monomers, solvents, catalysts, and so forth. It typically will be in the form of pellets or powder. It is the material from which fibers, tubes, rods, slabs, sheets, films, or specific parts and devices are fabricated.

4. Classification

4.1 PAEK polymers in the scope of this specification are pure semicrystalline homopolymers consisting of phenylene rings connected by ether and carbonyl groups. Their structures are commonly identified by the sequence of ether (E) and carbonyl (or ketone, K) groups along the polymer chain (for example, PEKEKK, PEEK, and so forth) (see Specification F1876, Specification F2026, and Appendix X2).

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

5. General Properties

5.1 PAEK polymers used in medical applications may comply with the Food and Drug Administration (FDA) regulation 21 CFR 177.2415, which covers both wet and dry food contact applications.

6. Chemical Properties

6.1 The required physical and chemical properties of some virgin PAEK polymers are listed in Table 1.

6.2 The infrared spectrum[7] of these materials is characteristic of their molecular repeating units. Representative spectra are listed in Appendix X3. The PAEK polymer shall yield an infrared transmittance spectrum which exhibits major bands only at the wavelengths listed for the standard reference spectrum for the material.

6.2.1 The infrared spectrum, as used herein, is to identify the specific type of PAEK present and does not necessarily indicate an acceptable degree of material purity.

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

7. Mechanical Properties

7.1 The mechanical properties of consolidated forms of these materials are dependent on the consolidation process. Additionally, the necessary mechanical properties of consolidated forms will vary from one application to another. Table 2 lists some typical mechanical properties expected for some of these consolidated forms.

7.2 The viscosity requirements will vary depending upon grade and test method. The method and requirements shall be agreed upon between vendor and purchaser.

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

[5] Available from U.S. Pharmacopeial Convention, 12601 Twinbrook Pkwy., Rockville, MD 20852.

[6] Available from the Food and Drug Administration, 5600 Fishers Ln., Rockville, MD 20857.

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

TABLE 1 Required Properties of Some PAEK Virgin Polymers

PAEK Type	T_B (°C)	T_m (°C)	Heavy Metals (%)
	ASTM D3418, 20°C/min	ASTM D3418, 20°C/min	U.S. Pharmacopeia Test 231
PEKEKK	160–200	360–400	≤0.1
PEEK	125–165	320–360	≤0.1
PEKK	135–175	305–365	≤0.1
PEEKK	140–195	350–390	≤0.1
PEK	140–180	350–395	≤0.1

TABLE 2 Typical Mechanical Properties of Some PAEK Consolidated Forms

PAEK Type	Density, (kg/m³) (Minimum)	Tensile Strength, (MPa) (Minimum)		Percent Elongation, (%) (Minimum)	Izod Impact Strength, (J/m) (Minimum)
	ASTM D1505	ASTM D638, Type IV, 5.08 cm/min		ASTM D638, Type IV, 5.08 cm/min	ASTM D256, d = 0.254 cm, r = 0.025 cm
		Yield	Break		
PEKEKK	1200	90	70	10	37
PEEK	1280	90	70	5	50
PEKK	1300			10	
PEEKK	1300	90		10	
PEK				10	

7.3 The mechanical properties of consolidated forms of PAEKs shall be determined on finished parts or test specimens processed similarly to finished parts.

7.4 Tests and test procedures shall be such as to assure a high level of control and characterization of the virgin polymer as received from the supplier. The following are some test methods that may be appropriate: Test Method D149, Test Method D256, Test Method D570, Test Method D638, Test Method D648, Test Method D695, Test Method D696, Test Method D790, Test Method D792, Test Method D955, Test Method D1238, Test Method D1505, Test Method D3417, Test Method D3418, and Test Method D4000.

8. Sampling

8.1 The material should be sampled in accordance with standard sampling procedures, such as those described in Practice D1898, or other sampling techniques unless otherwise agreed upon between consumer and supplier.

9. Biocompatibility

9.1 Biocompatibility of PAEK polymers and implant devices made using these materials shall be determined in accordance with Practice F748 or the ISO 10993 series, unless otherwise agreed upon by packager and consumer, and regulating bodies.[8]

10. Keywords

10.1 PAEK; PEEK; PEEKK; PEKEKK; PEKK; PEK; polyaryletherketone

[8] 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.

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 PAEK polymers may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of PAEK 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 crosslinking) 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.