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

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1. Scope

1.1 This specification covers polyaryletherketone (PAEK) resins in virgin forms as supplied by a vendor (flakes, pellets, blocks, etc.). 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, etc.) required for the production of a specific part or device. Therefore, properties of fabricated forms of these resins 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 resins 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 resins 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:*

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

D 256 Test Methods for Impact Resistance of Plastics and Electrical Insulating Materials[2]

D 570 Test Method for Water Absorption of Plastics[2]

D 621 Test Methods for Deformation of Plastics Under Load[2]

D 638 Test Method for Tensile Properties of Plastics[2]

D 648 Test Method for Deflection Temperature of Plastics Under Flexural Load[2]

D 695 Test Method for Compressive Properties of Rigid Plastics[2]

D 696 Test Method for Coefficient of Linear Thermal Expansion of Plastics[2]

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[2]

D 792 Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement[2]

D 955 Test Method for Measuring Shrinkage from Mold Dimensions of Molded Plastics[2]

D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[2]

D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[2]

D 1898 Practice for Sampling of Plastics[3]

D 3417 Test Method for Heats of Fusion and Crystallization of Polymers by Thermal Analysis[4]

D 3418 Test Methods for Transition Temperatures of Polymers by Thermal Analysis[4]

D 4000 Classification System for Specifying Plastic Materials[4]

F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[5]

2.2 *ISO Documents:*

ISO 1133 Plastics—Determination of the Melt Mass-Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics[6]

ISO 1628/1 Plastics, Guidelines for the Standardization of Methods for Determination of Viscosity Number and Limiting Viscosity Number of Polymers in Dilute

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

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

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

[4] *Annual Book of ASTM Standards,* Vol 08.03.

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

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

TABLE 1 Required Properties of Some PAEK Virgin Resins

PAEK Type	T_B (°C)	T_m (°C)	Voscosity Number, (ml/g)	Melt Flow, (cm^3/10 min)	Heavy Netaksm (%)
	ASTM D 3418, 20°C/min	ASTM D 3418, 20°C/min	ISO 1628, Sulfuric Acid 0.5 %, 25°C	ISO 1133, 400°C, 10 kg	U.S. Pharmacopeia Test 231
PEKEKK	160–200	360–400	≥55	25–120	≤0.1
PEEK	125–165	320–360	≥77		≤0.1
PEKK	135–175	310–350		40–100	≤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/m^3) (Minimum)	Tensile Strength, (MPA) (Minimum)		Percent Elongation, (%) (Minimum)	Izod Impact Strength, (ft-lf/in) (Minimum)	Deformation Under Load, (%) (Maximum)
	ASTM D 1505	ASTM D 638, Type IV, 5.08 cm/min		ASTM D 638, Type IV, 5.08 cm/min	ASTM D 256, d = 0.254 cm, r = 0.025 cm	ASTM D 621 (A), 7 MPA, 24 hr, 23°C, 90 min Rec.
PEKEKK	1.2	90	70	10	0.7	2
PEEK	1.3	90	70	10	1.0	2
PEKK	1.3			10		2
PEEKK	1.3	90		10		2
PEK				10		2

Solution—Part 1: General Conditions[6]

ISO 10993 Biological Evaluation of Medical Devices, Parts 1–12[6]

2.3 *Other Documents:*

United States Pharmacopeia, Vol. XXI, or latest edition[7]

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 resin as obtained by the synthesizer after removal of residual monomers, solvents, catalysts, etc. 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 PAEK resins 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, etc.) (see Appendix X2).

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

5. General Properties

5.1 PAEK resins used in medical applications may comply with the Food and Drug Administration (FDA) regulation 21 CFR 177.1580[8] that covers both wet and dry food contact applications.

6. Chemical Properties

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

6.2 The infrared spectrum[9] of these materials is characteristic of their molecular repeating units. Representative spectra are listed in Appendix X3. The PAEK resin 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 resins.

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

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

[8] Available from the Food and Drug Administration, Pittsburgh, PA.

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

7.4 Tests and test procedures shall be such as to assure 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 Method D 790, Test Method D 792, Test Method D 955, Test Method D 1238, Test Method D 1505, Test Method D 3417, Test Method D 3418, and Test Method D 4000.

8. Sampling

8.1 The material should be sampled in accordance with standard sampling procedures, such as those described in Practice D 1898, or other sampling techniques unless otherwise agreed upon between consumer and supplier.

9. Biocompatibility

9.1 Biocompatibility of PAEK 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 by packager and consumer, and regulating bodies.[10]

10. Keywords

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

[10] 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 resins may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of PAEK 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 crosslinking) 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.