Designation: F 1876 – 98 (Reapproved 2003)ε1

Standard Specification for Polyetherketoneetherketoneketone (PEKEKK) Resins for Surgical Implant Applications[1]

This standard is issued under the fixed designation F 1876; 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—Table 2 was editorially corrected in June 2003.

1. Scope

1.1 This specification covers polyetherketoneetherketoneketone (PEKEKK) resins in virgin forms as supplied by a vendor, such as flakes, pellets, blocks, and so forth. It provides requirements and associated test methods for these thermoplastics when they are to be used in the manufacturing 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, such as 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 that are appropriate to assure 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 PEKEKK resins only. Fabricated forms, material or forms containing colorants, fillers, processing aids, or other additives, as well as polymer blends that contain PEKEKK, 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 PEKEKK resins for use in medical devices. The properties listed should be considered in selecting material according to the specific end-use requirements.

1.5 *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 Methods for Determining the Izod Pendulum Impact Resistance of Plastics[3]

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

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

D 648 Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position[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 –30°C and 30°C with a Vitreous Silica Dilatometer[3]

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

D 792 Test Methods for Relative Density, Density of Plastics and Specific Gravity 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[4]

D 3417 Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC)[5]

D 3418 Test Methods for Transition Temperatures of Polymers by Differential Scanning Calorimetry (DSC)[5]

D 4000 Classification System for Specifying Plastic Materials[5]

F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[6]

[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.

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

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

[4] Discontinued; See 1997 *Annual Book of ASTM Standards*, Vol 08.01.

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

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

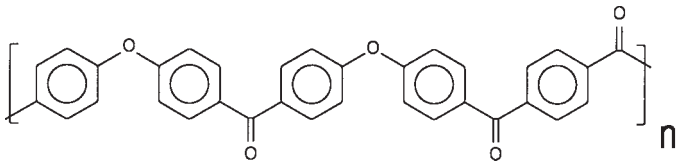

FIG. 1 Chemical Structure of PEKEKK

2.2 *ISO Standards:*

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[6]

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

ISO 10993 Biological Evaluation of Medical Devices, Parts 1–12[7]

2.3 *Other Documents:*

FDA Regulation CFR 177.1580[8]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *fabricated forms*, *n*—those items into which the virgin forms may be converted. These forms 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*, *n*—PEKEKK 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*—that form of the PEKEKK resin as obtained by the synthesizer after removal of residual monomers, solvents, catalysts, and so forth. 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 PEKEKK resins in the scope of this specification are pure semicrystalline homopolymers consisting of phenylene rings connected by either (E) and carbonyl (or ketone, K) groups along the polymer chain (see Fig. 1). Their polymeric structures are defined by the repeating unit EKEKK.

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

5. Properties

5.1 PEKEKK 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.

5.2 The infrared spectrum **(1)**[10] of these materials is characteristic of their molecular repeating units. A representative spectrum is provided in Fig. 2. The PEKEKK resin shall yield an infrared transmittance spectrum that exhibits major bands only at the wavelengths listed for a standard reference spectrum of that material.

5.2.1 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.

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, 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 by vendor and purchaser.

5.4 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.

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 nonsterilized 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 nonsterilized finished part.

5.7 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 (see Test Methods D 149, D 256, D 570, D 638, D 648, D 695, D 696, D 790, D 792, D 955, D 1238, D 1505, D 3417, D 3418, and D 4000).

6. Sampling

6.1 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.

7. Biocompatibility

7.1 Biocompatibility of PEKEKK 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 **(2-5)**.

[7] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[8] Available from Food and Drug Administration (FDA), 5600 Fishers Ln., Rockville, MD 20857.

[9] Available from U.S. Pharmacopeia (USP), 12601 Twinbrook Pkwy., Rockville, MD 20852.

[10] The boldface numbers in parentheses refer to the list of references at the end of this standard.

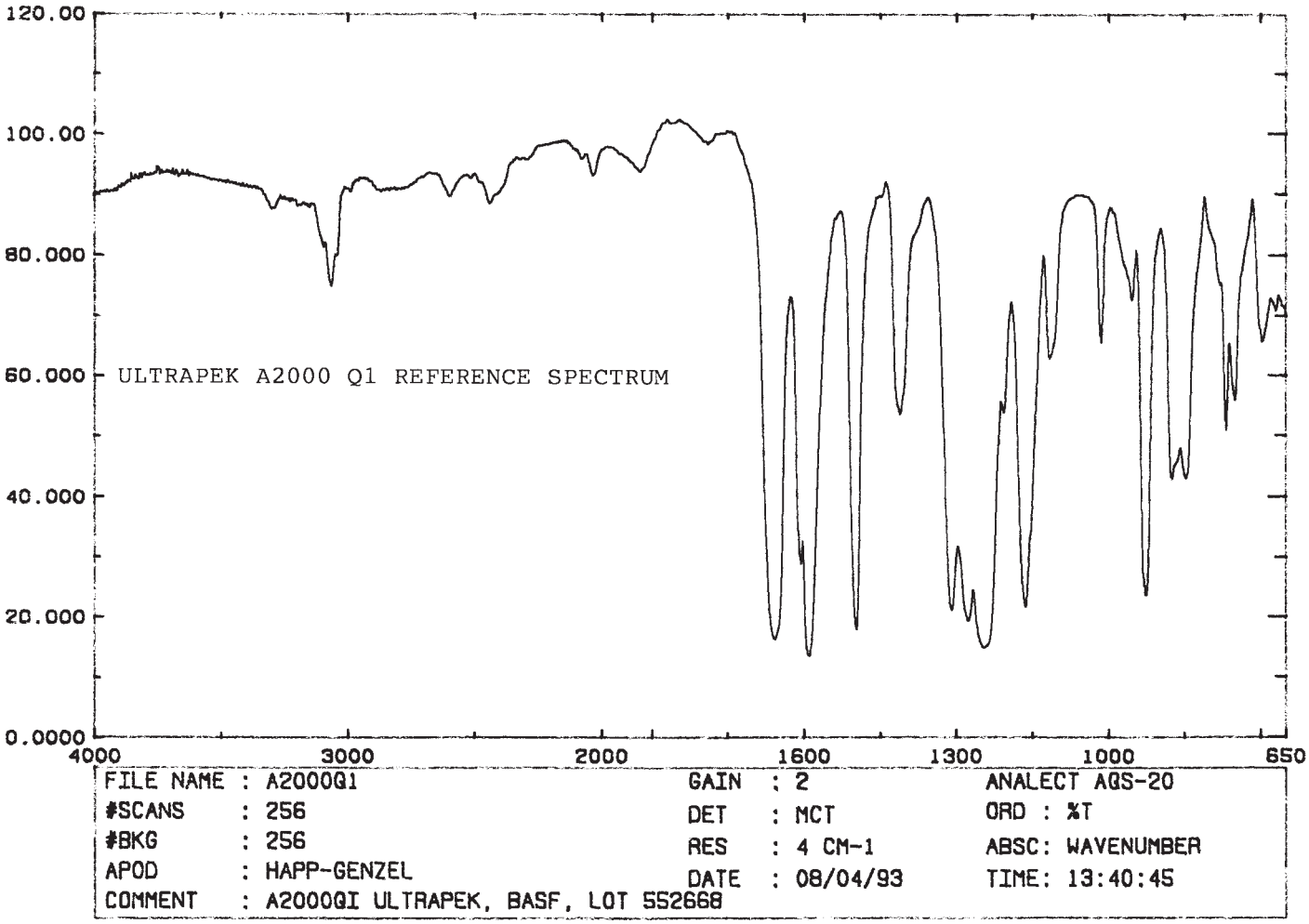

FIG. 2 Representative Infrared Spectra of PEKEKK

TABLE 1 Required Properties of Virgin Resin

Parameter	Method	Requirement
T_g, (°C)	DSC, 20 °C/min, sealed sample	160-200
T_m, (°C)	DSC, 20 °C/min, sealed sample	360-400
Viscosity number, (min) (mL/g)	ISO 1628, conc. sulfuric acid, 0.5 % w/v, 25 °C	55
Melt volume flow rate, (cm^3/10 min)	ISO 1133, 400° C, 10 Kg	25-120
Total heavy metals as Pb, (max) (%)	U.S. Phamacopeia, Test 231	0.1

TABLE 2 Typical Properties of Fabricated Forms

Parameter	Method	Requirement
Density, (min) (kg/m^3)	ISO D 1505	†1200
Tensile strength, (MPa) (min) Yield	D 638, Type IV, 5.08 cm/min	90
break		70
Percent elongation, (%) (min)	D 638, Type IV, 5.08 cm/min	10
Izod impact strength, (J/m), (min)	D 256, 0.254 cm depth, 0.025 cm radius	†37
Deformation under load, (%) (max)	D 621 (A), 7 MPa for 24 h, 23 °C, after 90 min recovery	2

† editorially corrected.

8. Keywords

8.1 PEKEKK; polyetherketoneetherketoneketone

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 PEKEKK resins may be processed by most techniques available for thermoplastic polymers. Medical devices and components of medical devices made of PEKEKK 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. It is imperative, therefore, 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. This feature of the resin and its fabricated form, therefore, should be evaluated using appropriate test methods to assure 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 10933 series.

REFERENCES

(**1**) Silverstein, R. M., Bassler, G. C., and Morrill, T. C., "Spectroscopic Identification of Organic Compounds," Fifth Edition, John Wiley and Sons, New York, NY.

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

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

(**4**) 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.

(**5**) 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.