Designation: D8501 – 23

Standard Classification System for and Basis for Specification for Unfilled Poly(Ether Ketone Ketone) (PEKK) Materials for Molding, Extrusion, Composites, Powder Coating and Additive Manufacturing[1]

This standard is issued under the fixed designation D8501; 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 poly(ether ketone ketone) materials, commonly referred to as PEKK, which are suitable for molding, extrusion, composites, powder coating and additive manufacturing. Only materials in this Class 6-8 are covered by this specification. This classification system provides requirements for the use of regrind or reprocessed materials.

1.2 This specification covers thermoplastic resin materials supplied in pellet as well as powder form.

1.3 This specification applies only to PEKK copolymers, without any additional fillers or inorganic additives, alloys, or treatments for modification of attributes.

1.4 This classification system and subsequent line callout (specification) are intended to provide means of calling out poly(ether ketone ketone) materials used in the fabrication of end items or parts.

1.5 Poly(ether ketone ketone) (PEKK) is a member of the poly (aryl ether ketone) or PAEK polymer family. PEKK has a broad range of repeat unit combinations of Isophthaloyl and Terephthaloyl repeat units. This standard classifies the polymer options.

1.6 The values stated in SI units, as detailed in IEEE/ASTM S-10, are to be regarded as the standard. The values given in parentheses are for information only.

1.7 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

NOTE 2—PEKK is a thermoplastic polymer. Testing conditions can affect the technical results. Specimens prepared by techniques different than prescribed in this specification can have properties that vary from the values specified.

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulation

D257 Test Methods for DC Resistance or Conductance of Insulating Materials

D618 Practice for Conditioning Plastics for Testing

D638 Test Method for Tensile Properties of Plastics

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

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

D1600 Terminology for Abbreviated Terms Relating to Plastics

D2863 Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index)

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

D3892 Practice for Packaging/Packing of Plastics

D4000 Classification System for Specifying Plastic Materials

D6247 Test Method for Determination of Elemental Content

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials.

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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.

of Polyolefins by Wavelength Dispersive X-ray Fluorescence Spectrometry
D8371 Test Method for Trace Metal Content Analysis in Carbon Black

2.2 *Underwriters' Laboratories Standards (UL):*[3]
UL94 Standard for Tests for Flammability of Plastic Materials

2.3 *ISO Standards:*[4]
ISO 179/1eU Determination of Charpy Impact Properties
ISO 527-1 Determination of tensile properties
ISO 1133 Determination of the Melt Mass-Flow Rate (MFR) and Volume-Flow Rate (MVR) of Thermoplastics
ISO 1183 Methods for determining the density of non-cellular plastics

2.4 *International Electrochemical Commission (IEC):*[5]
IEC 60243-1 Electric strength of insulation materials
IEC 60205 Calculation of the effective parameters of magnetic piece parts

3. Terminology

3.1 *Definitions of Terms*—Except for the terms defined in 3.2, the terminology used in this classification system is in accordance with Terminologies D883 and D1600.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *copolymer, n*—a polymer made by the reaction of two or more different monomers with units of more than one kind.

3.2.2 *isophthaloyl repeat unit, n*—refers to the chemical structure where ketone linkages are located in the meta position. See Fig. 1.

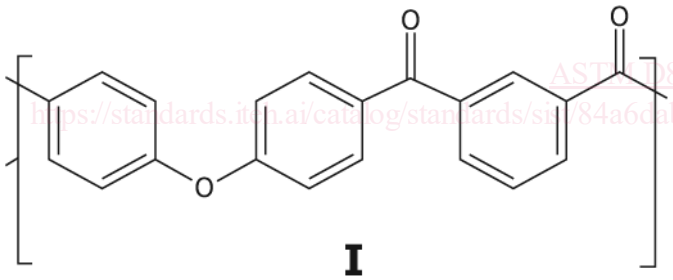

FIG. 1 Meta-linkage within Backbone of Poly(ether ketone ketone) (isophthaloyl repeat unit)

3.2.3 *poly(ether ketone ketone), n*—a highly customizable semicrystalline polymer in the poly(aryl ether ketone) (PAEK) family, consisting of two polymer repeat units consisting of either terephthaloyl or isophthaloyl repeat units.

3.2.3.1 *Discussion*—Poly(ether ketone ketone) specifically has a repeating structural unit containing one ether and two ketone linkages.

3.2.4 *regrind (plastic), n*—a product or scrap such as sprues, runners, sheet, film, rod and melted strands that have been reclaimed by shredding and granulating for use in-house.

3.2.5 *reprocessed plastic, n*—thermoplastic prepared from usually melt processed scrap or reject parts by a plastics processor, or from non-standard or non-uniform virgin material.

3.2.6 *terephthaloyl repeat unit, n*—refers to the chemical structure where ketone linkages are located in the para position. See Fig. 2.

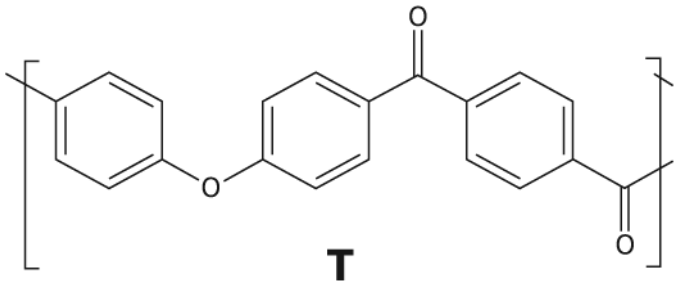

FIG. 2 Para-linkage within Backbone of Poly(ether ketone ketone) (terephthaloyl repeat unit)

3.2.7 *viscosity, n*—the property of resistance to flow in any material with fluid properties.

4. Classification

4.1 Poly(ether ketone ketone) PEKK materials shall be classified into Classes that are subdivided into Grades as shown in Tables 1 and 2.

4.1.1 *Class*—PEKK polymers contain only two types of repeat units and be classified only by the ratio of Terephthaloyl (T) and Isophthaloyl (I) units, as described in Table 1. The Table 1 ratios of repeat units can greatly affect the properties of the polymer. Table 1 describes, also, the melting point ranges for each class.

NOTE 3—The crystallinity and crystallization rate are strongly affected by the structure of the polymer backbone linkages (para or meta).

4.1.1.1 Determine the T/I ratio by NMR analysis. Melt point ranges indicated are representative of the T to I ratio for each class.

4.1.2 *Grade*—The desirable melt volume flow rate (MVR) of PEKK polymers is a function of the requirements of the application or intended use. Thus, the desirable melt flow will depend on whether the polymer is intended to be used as an injection molding grade, powder coating grade, 3D printing grade or extrusion grade. The grades shall be distinguished as

TABLE 1 Class Assignment Based on Ratio of (T) and (I)

Class	Percent of (T)	Melting Point[A] Range, °C
6	57-62	290-305
7	68-73	325-350
8	78-85	355-370

[A]For some medium to very high viscosity materials, it's possible that a melt point will not be detected with a ramp rate of 20°C/min. Since different ramp rates can influence the detection of transition temperatures, other rates can be used as long as the rate is noted, and the DSC is calibrated at that ramp rate.

[3] Available from Underwriters Laboratories (UL), UL Headquarters, 333 Pfingsten Road, Northbrook, IL, 60062, http://www.ul.com.

[4] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

[5] Available from International Electrotechnical Commission (IEC), 3, rue de Varembé, 1st floor, P.O. Box 131, CH-1211, Geneva 20, Switzerland, https://www.iec.ch.

TABLE 2 Grade Designation Based on Melt Volume Flow Rate (MVR) for Unfilled PEKK Materials

MVR (cm^3 /10min) Range @ 380°C and 1 or 5 kg weight[A]	Grade Description Based on Viscosity	Applications
60-120	Ultra Low Viscosity	Intended for composites
16-60	Very Low Viscosity	Intended for reinforced grades
8-16	Low Viscosity	Intended for reinforced grades
4-8	Medium Viscosity	Intended for extrusion or injection molding
11-24	High Viscosity	Intended for extrusion
3-12	Very High Viscosity	Intended for extrusion

[A] 1 kg weight for ultra low, very low, low and medium viscosity grades; 5 kg weight for high and very high viscosity grades.

low, medium or high volume flow rate and the ranges shall be as required by Table 2. The melt volume flow rate shall be measured in accordance with ISO 1133 or Test Method D1238, at a temperature of 380°C, by using a 5-kg weight (for the high and very high viscosity grades) and a 1-kg weight for all the other grades.

4.2 Tables 3 and 4 describe predefined cells to be used to refer to specific aspects of this specification.

Specification
Standard Number Block, Class, Grade, Additional Requirements
Example: ASTM D8501 PEKK Class 6, Grade Low Viscosity

NOTE 4—Additional requirements could be ASTM D4000 suffixes or special notes.

NOTE 5—Ranges for MVR are classification values only.

4.2.1 For the examples shown in Table 4, this line callout would be ASTM D8501 PEKK Class 6, Grade Low Viscosity. This describes a PEKK polymer with 57-62 % Terephthaloyl (T) repeat unit and 38-43 % Isophthaloyl (I) repeat unit with a melting point between 290-305°C, and a melt volume flow rate of between 8 cm^3 and 16 cm^3 per 10 minutes when tested in accordance with ISO 1133 or Test Method D1238 at a 380°C temperature using a 1 kg weight. The second example shows a callout for PEKK, D8501, Class 8, Grade High Viscosity, "high purity product, no use of regrind". This describes a PEKK polymer with 78-85 % Terephthaloyl (T) repeat unit and 15-22 % Isophthaloyl (I) repeat unit with a melting point of between 355 and 370°C, and a melt volume flow rate of between 3 and 12cm^3 per 10 minutes when tested in accordance with ISO 1133 or Test Method D1238 at a 380°C temperature and using a 5 kg weight. A comma shall be used as the separator between the standard number, the class number and the grade type. The cells addressing grade callout and special notes are not mandatory. The special notes are encased in quotations.

NOTE 6—A provision for special notes is included in the table so that other information, such as a preferred property range, can be provided when required.

TABLE 3 Classification for Unfilled PEKK Materials

Standard Number Block	Polymer Family	Class	Grade
Example: Specification ASTM D8501	PEKK	6, 7, 8	Ultra Low Viscosity, Very low viscosity, Low Viscosity, Medium viscosity, High viscosity, Very High Viscosity

TABLE 4 Examples of Designation or Specification Callout

Standard Block	Polymer Family	Class	Grade	Special Notes
Specification ASTM D8501	PEKK	6	Low Viscosity	"..."
Specification ASTM D8501	PEKK	8	High Viscosity	"High purity product, no use of regrind"

4.2.2 When additional requirements are used, they shall be preceded by a comma. If a callout section is not used, it is acceptable to leave it out or to use three dashes to indicate that the section is not required.

4.2.3 *Suffixes*—When additional requirements are needed that are not covered by the basic requirements or cell-table requirements, indicate through the use of suffixes. A list of suffixes can be found in Classification System D4000 (Table 3) and are to be used for additional requirements as appropriate

NOTE 7—With the use of ASTM D4000 suffixes, the example of "tensile strength at yield >= 130 MPa" would be replaced by the suffix "KX130" (for ISO 527) or "KY130" (for ASTM D638).

4.2.4 An example callout for a PEKK polymer requiring 80 % terephthaloyl repeat unit, no specific viscosity, and a tensile yield strength of at least 130 Mpa would be:

ASTM D8501 PEKK, Class 8, "minimum 130 Mpa tensile yield strength required."

5. General Requirements

5.1 The material shall be free of foreign matter and of uniform composition.

5.1.1 Use of regrind or reprocessed PEKK material is permissible with up to a maximum of 20 % as long as it meets specifications of the callout; however, this shall be reported in the additional requirements cell.

6. Detail Requirements

6.1 *General Attributes:*

6.1.1 *Peak Melting Endotherm Grade 8*—The material covered by this specification shall have a peak melting endotherm not exceeding the limits for a Class 8 material, as required by Table 1 when tested in accordance with Test Method D3418. The determination of melting temperature in accordance with 6.1.1 involves heating, cooling and reheating a solid specimen with a weight 5 mg to 10 mg, at a heating rate of 20°C/min,

ranging from 20°C to 400°C, with no upper limit hold. The peak melting temperature during the reheat shall be recorded.

6.1.2 *Peak Melting Endotherm Grade 6 and 7*—The determination of melting temperature in accordance with 6.1.1 for materials in Classes 6 and 7, as required by Table 1, shall also involve heating, cooling and reheating a solid specimen with a weight of 5 mg to 10 mg at a heating rate of 20°C/min, ranging from 20°C to 380°C, with no upper limit hold. The peak temperature shall be determined on the initial heating phase, by analyzing the highest melting endotherm.

6.1.3 *Specific Gravity*—A solid specimen of the material covered by this specification shall have the specific gravity in the range of 1.20-1.40 when tested in accordance with Test Method D792 or ISO 1183.

6.1.4 *Limiting Oxygen Index and Flame Rating*—Materials covered by this specification shall exhibit a minimum limiting oxygen index of not less than 30 when tested in accordance with Test Method D2863 and comply with a UL94 flammability rating of V-0, at a thickness of 0.8 mm.

6.1.5 *Glass Transition Temperature (Tg)*—Materials covered by this specification shall exhibit a minimum reheat glass transition temperature of greater than 150°C when tested in accordance with Test Method D3418 using the test method parameters given in 6.1.1.

6.1.6 *Iron Content*—The iron content shall be less than 20 ppm when measured in accordance with 7.4.

6.2 *Mechanical Properties:*

6.2.1 *Rheological Properties*—Test the apparent melt volume flow rate of these materials in accordance with Test Method ISO 1133 or Test Method D1238 at 380 ± 1°C (716°F). See Table 2.

6.2.2 *Tensile Properties*—The materials covered by this specification shall have minimum tensile properties as indicated in 6.2.2.1 and 6.2.2.2, when tested in accordance with Test Method D638 or ISO 527-1 at a rate of 50.8 mm (2 in.)/min, using Type I specimens 3.2-mm (0.125-in.) thick or ISO tensile specimens. Test specimens shall yield and rupture in the gage region and not near the heel. The test specimens shall be prepared under conditions specified by the material manufacturer.

6.2.2.1 The tensile modulus shall exceed 2500 Mpa (362, 600 psi) at 23°C (73°F).

6.2.2.2 The minimum elongation at break shall be 1.0 %.

6.2.3 *Impact Resistance*—The materials covered by this specification, other than ultra low viscosity material, shall have a minimum notched charpy impact strength of 4 kJ/m^2. The impact strength shall be determined in accordance with by ISO 179/1eU, using specimens that are either 3.2-mm (0.125-in.) or 6.4-mm (0.25-in.) thick, prepared by injection molding under conditions specified by the material manufacturer. This requirement does not apply for ultra low viscosity material.

6.3 *Electrical Properties:*

6.3.1 *D-C Resistivity*—The materials covered by this specification shall have a d-c volume and surface resistivity greater than 10^{14} Ω·cm when tested as a 0.76-mm (0.030-in.) in accordance with Test Methods D257.

6.3.2 *Dielectric Strength*—The materials covered by this specification shall have a dielectric strength, in air, of no less than 80 kV/mm (2032 kV/ in.) by the "short-time" method of Test Methods IEC 60243-1, using specimens that are 0.1-mm (0.004-in.) thick, as tested in air using 25.4-mm (1-in.) Type 3 electrodes.

6.3.3 *Dielectric Constant*—The materials covered by this specification shall have a dielectric constant less than 3.5 at 1 MHz when tested as a 3.2-mm (0.125-in.) thick specimen (see Section 8) in accordance with Test Methods D150 or in accordance with IEC 60205, at a temperature of 23°C (73°F).

7. Test Methods

7.1 *General*—The inspection and test procedures contained in this section shall be used to determine the conformance of products to the requirements of this specification. Each material manufacturer who represents its materials as conforming to this specification shall be permitted to use statistically-based sampling plans that are appropriate for each manufacturing process. However, the manufacture shall be required to keep the essential records necessary to document, with a high degree of assurance, the claim that all of the requirements of this specification have been met. Additional sampling and testing of the product, as agreed upon between the purchaser and the manufacturer, are not precluded by this section.

7.2 *Moisture*—The material used for test specimen preparation shall have a moisture content of less than 0.05 %.

7.2.1 Table 5 describes the material drying conditions.

NOTE 8—Typical tests for moisture include Karl Fischer and moisture balance.

7.3 *Conditioning and Test Conditions*—Condition the test specimen in accordance with Procedure A of Practice D618 and test under those conditions, unless otherwise specified herein.

7.4 *Iron Determination*—Determine iron content by using either Wavelength Dispersive X-ray Fluorescence (WDXRF) spectroscopy or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-MS or ICP-OES).

NOTE 9—Both methods have been shown to give equivalent results.

7.4.1 The use of Wavelength Dispersive X-ray Fluorescence for determining iron content in PEKK will require a calibrated method that properly brackets the iron content.

NOTE 10—WDXRF methods are described in Test Method D6247.

7.4.2 Test specimen preparation for assessing iron content using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) or Inductively Coupled Plasma Mass Spectrometry (ICP-MS) requires digestion. The digestion process shall result in a clear solution. The digested solutions must be sufficiently diluted for the iron intensity to be bracketed within the ICP calibration curve.

TABLE 5 Drying Temperature Recommendations for PEKK

Class	Temperature °C (°F)	Time (hours)
6	120-140 (248-284)	4-8
7	150-170 (302-338)	4-8
8	150-170 (302-338)	4-8