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# Standard Specification for Isotropic and Near-isotropic Nuclear Graphites<sup>1</sup>

This standard is issued under the fixed designation D7219; 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 the classification, processing, and properties of nuclear grade graphite billets with dimensions sufficient to meet the designer's requirements for fuel elements, moderator or reflector blocks, in a high temperature gas cooled reactor. The graphite classes specified here would be suitable for reactor core applications where neutron irradiation induced dimensional changes are a significant design consideration.
- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. (See IEEE/ASTM SI 10.)
- 1.3 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:<sup>2</sup>
- C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles
- C709 Terminology Relating to Manufactured Carbon and Graphite
- C781 Practice for Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components
- C838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes
- C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
- D2638 Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer

IEEE/ASTM SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

2.2 ASME Standard:

NQA-1 Quality Assurance Program Requirements for Nuclear Facilities<sup>3</sup>

## 3. Terminology

- 3.1 *Definitions*—Definitions relating to this specification are given in Terminology C709.
  - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 baking/re-baking charge—number of billets in a baking/re-baking furnace run.
- 3.2.2 *bulk density*—mass of a unit volume of material including both permeable and impermeable voids.
- 3.2.3 *extrusion forming lot*—number of billets of the same size extruded in an uninterrupted sequence.
- 3.2.4 *green batch*—mass of coke, recycle green mix, recycle graphite, and pitch that is required to produce a forming lot.
- 3.2.5 *green mix*—percentage of mix formulation, pitch and additives required for the forming lot, which is processed and ready to be formed.
- 3.2.6 *graphite billet*—extruded, molded, or iso-molded graphite artifact with dimensions sufficient to meet the designer's requirements for reactor components.
- 3.2.7 graphite grade—designation given to a material by a manufacturer such that it is always reproduced to the same specification and from the same raw materials and mix formulation.
- 3.2.8 *graphitization charge*—number of billets of the same grade in a graphitizing furnace run.
- 3.2.9 *graphitizing furnace run*—total number of billets graphitized together in one graphitization furnace.
- 3.2.10 *high purity nuclear graphite*—nuclear graphite with an Equivalent Boron Content less than 2 ppm.
- 3.2.11 *impregnation charge*—number of billets in an autoclave cycle.
- 3.2.12 *isotropic nuclear graphite*—graphite in which the isotropy ratio based on the coefficient of thermal expansion is 1.00 to 1.10.

<sup>&</sup>lt;sup>1</sup> This specification is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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<sup>&</sup>lt;sup>2</sup> 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.

<sup>&</sup>lt;sup>3</sup> Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Three Park Ave., New York, NY 10016-5990.

**TABLE 1 ASTM Standard Classes of Nuclear Graphite** 

	OTE leature Datie B		Purity	Dulla Damaita C	Class Designation
Class <sup>A</sup>	CTE Isotropy Ratio <sup>B</sup> $(\alpha_{AG}/\alpha_{WG})$	Ash Content, <sup>B</sup> ppm (max)	Boron Equivalent, <sup>D</sup> ppm (max)	Bulk Density, C g/cm <sup>3</sup> (min)	
Isomolded, isotropic—High Purity	1.0-1.1	300	2	1.7	IIHP
Isomolded, isotropic—Low Purity	1.0-1.1	1000	10	1.7	IILP
Isomolded, near-isotropic—High Purity	1.1-1.15	300	2	1.7	INHP
Isomolded, near-isotropic—Low Purity	1.1-1.15	1000	10	1.7	INLP
Extruded, isotropic—High Purity	1.0-1.1	300	2	1.7	EIHP
Extruded, isotropic—Low Purity	1.0-1.1	1000	10	1.7	EILP
Extruded, near-isotropic—High Purity	1.1-1.15	300	2	1.7	ENHP
Extruded, near-isotropic—Low Purity	1.1-1.15	1000	10	1.7	ENLP
Molded, isotropic—High Purity	1.0-1.1	300	2	1.7	MIHP
Molded, isotropic—Low Purity	1.0-1.1	1000	10	1.7	MILP
Molded, near-isotropic—High Purity	1.1-1.15	300	2	1.7	MNHP
Molded, near-isotropic—Low Purity	1.1-1.15	1000	10	1.7	MNLP

<sup>&</sup>lt;sup>A</sup> These classes may be further modified by the grain size as defined in Terminology C709 (see Table 2).

- 3.2.13 *low purity nuclear graphite*—nuclear graphite with an Equivalent Boron Content greater than 2 ppm but less than 10 ppm.
- 3.2.14 *mix formulation*—percentages of each specifically sized filler used to manufacture a graphite grade.
- 3.2.15 *molding forming lot*—number of billets molded from a molding powder lot.
- 3.2.16 *molding powder lot*—sufficient quantity of re-milled and blended green batch produced from an uninterrupted flow of raw materials, or produced in a sequence of identical materials batches, to produce a molding forming lot.
- 3.2.17 *near isotropic nuclear graphite*—graphite in which the isotropy ratio based on the coefficient of thermal expansion is 1.10 to 1.15.
- 3.2.18 *nuclear graphite class*—designation of a nuclear graphite based upon its forming method, isotropy, purity and density (see Table 1).
- 3.2.19 *production lot*—specified number of billets made in accordance with this specification and additional requirements determined by the purchaser.
- 3.2.20 *purification charge*—number of billets in a purification run.
- 3.2.21 *recycle green mix*—ground non-baked billets or non used green mix manufactured in compliance with the mix formulation specified here.

## 4. Significance and Use

4.1 The purpose of this specification is to document the minimum acceptable properties and levels of quality assurance and traceability for isotropic and near-isotropic nuclear grade graphites.

# 5. Materials and Manufacture

- 5.1 Nuclear Graphite Classes—See Table 1.
- 5.2 Raw Materials:
- 5.2.1 Fillers:
- 5.2.1.1 The filler shall consist of a near-isotropic or isotropic coke derived from a petroleum oil or coal tar.
- 5.2.1.2 The coke shall have a coefficient of linear thermal expansion (CTE), determined in accordance with Practice

TABLE 2 ASTM Graphite Grain Size Definitions from Terminology C709

Graphite Designation	Definition of Grains in the Starting Mix that are: <sup>A</sup>			
Medium Grained Fine Grained Superfine Grained Ultrafine Grained Microfine Grained	Generally < 4 mm <sup>B</sup> Generally < 100 µm Generally < 50 µm Generally < 10 µm Generally < 2 µm			

<sup>&</sup>lt;sup>A</sup> Grain size as defined in Terminology C709.

C781 and measured over the temperature range 25 to 500°C, of between  $3.5 \times 10^{-6}$  and  $5.5 \times 10^{-6}$  °C<sup>-1</sup>.

- 5.2.1.3 The coke shall be sampled and distributed as described in Table 3.
- 5.2.1.4 Graphite manufactured in compliance with this specification but failing to meet the property requirements of Sections 6 and 7 may be used as recycle material in the mix formulation.
- 5.2.1.5 Recycle green mix manufactured from raw materials in compliance with this specification may be used in the mix formulation.
- 5.2.1.6 The maximum filler particle size used in the mix formulation shall be 1.68 mm.
- 5.3 *Binder*—The binder(s) shall consist of coal tar pitch of the same grade from the same manufacturer. The specific binder(s) used shall be identified to the purchaser and be traceable through the forming lot.
- 5.4 *Impregnant*—The impregnant(s) shall consist of a petroleum or coal tar pitch of the same grade from the same manufacturer. The specific impregnant used shall be identified to the purchaser and be traceable through the impregnation steps.
- 5.5 Manufacturing or Processing Additives—Additives (for example, extrusion aids) may be used to improve the processing, quality and properties of the product, but only with the consent and approval of the purchaser, and they must be traceable through the forming lot.

<sup>&</sup>lt;sup>B</sup> Determined in accordance with Practice C781.

<sup>&</sup>lt;sup>C</sup> Determined in accordance with Test Method C559.

<sup>&</sup>lt;sup>D</sup> Determined in accordance with Practice C1233.

<sup>&</sup>lt;sup>B</sup> For nuclear graphite, the maximum grain size is 1.68 mm in accordance with 5.2.1.6.

#### TABLE 3 Inspection Sampling and Testing of Filler Cokes

Inspection Plan	Sampling Procedure	Tests and Test Methods
A representative sample of the coke shall be taken prior to the mixing step of manufacture	Sample in accordance with Practice D346  1. A sufficient sample for preparation of CTE test specimens  2. A sufficient sample will be taken for additional testing. This sample shall be retained for a period specified by the graphite purchaser	The procedure in Practice C781 shall be used to prepare test specimens for the measurement of coke CTE.  Measure the coke real density in accordance with Test Method D2638.

# 5.6 Manufacture:

- 5.6.1 *Formulation*—The mix formulation (as defined in 3.2.14) and recycle green mix fraction (as defined in 3.2.21) in the filler shall be recorded. This information shall be reported to the purchaser if requested.
- 5.6.2 *Forming*—The green mix may be formed by extrusion, molding (including vibrationally molding), or isomolding.
- 5.6.3 *Graphitization Temperature*—The graphitization temperature shall be determined on each billet using the procedure described in Practice C781. Each billet tested in accordance with Practice C781 shall have a Specific Electrical Resistivity (SER) corresponding to a graphitization temperature of at least 2700°C.

## 6. Chemical Properties

- 6.1 Each graphite production lot shall be sampled in accordance with Section 11. The chemical impurities to be measured shall be as agreed between the supplier and the purchaser. The minimum list of elements to be measured and used for the EBC calculation shall be B, Cd, Dy, Eu, Gd, and Sm.
- 6.2 The boron equivalent shall be calculated in accordance with Practice C1233. The acceptance limits for the boron equivalent, as well as for ash content, are given in Table 1.
- 6.3 Table X1.1 contains a list of chemical impurities that are typically measured depending on end-use requirements. The impurities are categorized as neutron absorbing impurities, oxidation promoting catalysts, activation relevant impurities, metallic corrosion relevant impurities, and fissile/fissionable elements.

# 7. Physical and Mechanical Properties

- 7.1 Each graphite production lot shall be sampled in accordance with Section 11 and shall conform to the requirements for physical properties prescribed in Table 1 and Table 4 for the appropriate nuclear graphite class, and to the additional requirements of the purchaser.
- 7.2 The bulk density of each graphite billet shall be measured as described in Test Method C838.

# 8. Other Requirements

- 8.1 The graphitized billets shall be handled and stored such that they are protected from contaminants other than ambient air.
- 8.2 Each graphite billet shall be marked with a unique billet identification number. Each billet shall be traceable through these identifying numbers to each of the following:
  - 8.2.1 Mix formulation,
  - 8.2.2 Coke batch,
  - 8.2.3 Recycle graphite batch,

- 8.2.4 Forming lot,
- 8.2.5 Molding powder lot,
- 8.2.6 Baking charge,
- 8.2.7 Impregnant charge,
- 8.2.8 Graphitization furnace run,
- 8.2.9 Position of billet in graphitization furnace,
- 8.2.10 Purification step (if performed),
- 8.2.11 Binder pitch,
- 8.2.12 Impregnant pitch, and
- 8.2.13 Additives used (if any).

#### 9. Dimensions

9.1 Graphite billet dimensions are typically 0.4 to 0.6 m diameter (extruded) or thickness (molded/extruded) of 0.6 by 0.6 m cross-section (iso-molded) and 0.75 to 3.0 m length.

## 10. Workmanship, Finish and Appearance

10.1 Graphitized billets shall be brushed clean after removal from the graphitization furnace.

# 11. Sampling and Cutting

- 11.1 A statistical sampling plan shall be developed by the supplier and agreed with the purchaser. The plan shall describe the number of graphite billets to be sampled and the frequency of sampling. The following minimum sampling frequencies are recommended per production lot, depending on the number of billets per production lot.
- 11.1.1 Sample four billets for each production lot containing ten or fewer billets.
- 11.1.2 Sample one additional billet for every five additional billets per production lot, up to 50 billets.
- 11.1.3 For production lots exceeding 50 billets the additional sampling requirements should be agreed between the supplier and the purchaser.
- 11.1.4 During production the sampling plan may be reevaluated based on statistical analysis of the production data. Any revised sampling plan must be agreed between the supplier and the purchaser.
- 11.2 A cutting plan shall be agreed between the purchaser and manufacturer. The cutting plan shall describe the type, location, number, orientation of the test specimens, and any required archive specimens needed for property determinations as set forth in Sections 6 and 7 of this specification. The cutting plan shall reflect property gradients and anisotropy introduced by forming and processing. In addition the number of each type of specimen defined by the cutting plan shall be sufficient to yield statistically significant data.

TABLE 4 Physical and Mechanical Properties for Nuclear Graphite

Class <sup>A</sup>	Coefficient of Thermal Expansion (25 to 500°C),	Strength, <sup>B</sup> WG MPa, min			Dynamic Elastic Modulus, WG, GPa	
	WG °C <sup>-1</sup>	AG, Wm <sup>-1</sup> K <sup>-1</sup> min	Tensile	Flexural	Compressive	wodalao, wa, ar a
Practice <sup>C</sup>	C781	C781	C781	C781	C781	C781
IIHP	3.5-5.5 × 10 <sup>-6</sup>	90	22	35	65	8–15
IILP	$3.5-5.5 \times 10^{-6}$	90	22	35	65	8–15
INHP	$3.5-5.5 \times 10^{-6}$	80	20	30	60	8–15
INLP	$3.5 – 5.5 \times 10^{-6}$	80	20	30	60	8–15
EIHP	$3.5 – 5.5 \times 10^{-6}$	100	15	21	45	8–15
EILP	$3.5-5.5 \times 10^{-6}$	100	15	21	45	8-15
ENHP	$3.5-5.5 \times 10^{-6}$	100	15	21	45	8-15
ENLP	$3.5-5.5 \times 10^{-6}$	100	15	21	45	8–15
MIHP	$3.5 – 5.5 \times 10^{-6}$	100	15	21	45	8–15
MILP	$3.5-5.5 \times 10^{-6}$	100	15	21	45	8-15
MNHP	$3.5-5.5 \times 10^{-6}$	100	15	21	45	8-15
MNLP	$3.5-5.5 \times 10^{-6}$	100	15	21	45	8-15

A WG = With Grain; AG = Against Grain.

# 12. Finished Inspection

- 12.1 Graphite billets shall be visually inspected for external flaws. The allowable size, type, and number of flaws shall be defined in agreement between the purchaser and the manufacturer and be described in the purchase specification.
- 12.2 It is recommended that all graphite billets are non-destructively tested to screen for internal defects. The allowable size, type, and number of internal flaws should be defined in agreement between the purchaser and the manufacturer and be described in the purchase specification.

## 13. Rejection and Rework

- 13.1 Graphite billets failing on chemical purity (see Section 6) may be purified/re-purified and subjected to retest.
- 13.2 Graphite billets failing on SER (see 5.6.3) may be re-graphitized and subjected to retest.
- 13.3 All other billets failing to meet the requirement of Sections 6 and 7 of this specification may be used as recycle graphite in accordance with 5.2.1.4.

## 14. Certification

14.1 The manufacturer shall certify that the graphite meets the requirements of this specification and the purchase specification.

#### 15. Product Marking

15.1 Each billet shall be marked with a permanent unique number, which shall be traceable to the production history as specified in 8.2.

# 16. Packaging and Storage

- 16.1 Packaging of the finished billet shall be in accordance with the purchaser's specification.
- 16.2 Storage of the finished billets prior to shipping shall be such that no damage is incurred.

#### 17. Quality Assurance

17.1 The manufacturer of nuclear graphite furnished under this specification shall comply with the applicable quality assurance requirements of ASME NQA-1 as identified by the purchaser's specification. The purchaser specification may require application of quality assurance requirements other than ASME NQA-1.

#### 18. Keywords

18.1 chemical properties; mechanical properties; nuclear graphite; physical properties; processing

<sup>&</sup>lt;sup>B</sup> At least one of the three strength measurements should be selected for production lot acceptance in agreement between the supplier and the purchaser.

<sup>&</sup>lt;sup>C</sup> Equivalent practices may be used by manufacturers based outside the United States.

#### **APPENDIX**

(Nonmandatory Information)

#### X1. IMPURITIES

X1.1 The control of impurities in nuclear graphite depends on the specific end-use requirements. Table X1.1 categorizes typical chemical impurities found in nuclear graphite that contribute to specific effects, for example, neutron absorption. The contributing effect of the impurity element depends on both the nature of the impurity and its concentration in the graphite. For example boron and gadolinium have a high neutron absorption cross-section but in the high purity nuclear

graphite the quantities are low. However, an element such as iron that has a low absorption cross-section can typically be an order of magnitude higher in concentration and thus influence the overall Equivalent Boron Content, more so in the low purity nuclear graphite. It is therefore recommended that the supplier provide chemical impurity data on the nuclear graphite to be supplied from which the key impurities to be measured can be agreed upon with the purchaser.

TABLE X1.1 Impurity Categories for Nuclear Graphites<sup>A</sup>

Neutron Absorbing Impurities	Oxidation-promoting Catalysts	Activation Relevant Impurities	Metallic Corrosion Relevant Impurities	Fissile/fissionable elements
Boron	Aluminum	Antimony (Sb)	Chlorine	Thorium
Cadmium	Barium	Cadmium	Mercury	Uranium
Chlorine	Calcium	Cesium	Sulfur	
Dysprosium	Copper	Chlorine <sup>B</sup>		
Éuropium	Iron	Cobalt <sup>A</sup>		
Gadolinium	Lead	Europium		
Gold	Magnesium	Gadolidium		
Indium	Manganese	Iron		
Iron	Nickel <sup>C</sup>	Lithium <sup>D</sup>		
Mercury	Potassium	Molybdenum		
Nickel	Silicon	Nickel		
Rhenium	Silver	Nitrogen <sup>E</sup>		
Rhodium	Sodium	Scandium		
Samarium	Titanium	Silver		
Titanium		Tantalum		
Vanadium		Terbium		
		Thorium		
		Uranium		
		Dysprosium		

<sup>&</sup>lt;sup>A</sup> Problem isotope is <sup>60</sup>Co, strong gamma emitter, long half-life.

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<sup>&</sup>lt;sup>B</sup> Active isotope is <sup>36</sup>Cl (gamma emitter with extremely long half-life).

<sup>&</sup>lt;sup>C</sup> Source of <sup>60</sup>Co.

<sup>&</sup>lt;sup>D</sup> Primary source of tritium <sup>3</sup>H (beta emitter).

<sup>&</sup>lt;sup>E</sup> Primary source of <sup>14</sup>C, beta emitter with long-half life.