



Standard Specification for Isotropic and Near-isotropic Nuclear Graphites¹

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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.

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:²

- C 709 Terminology Relating to Manufactured Carbon and Graphite
- C 781 Practice for Testing Graphite and Boronated Graphite for High-Temperature Gas-Cooled Nuclear Reactors
- C 838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes
- C 1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
- D 2638 Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer

2.2 ASME Standard:

NQA-1 Quality Assurance Program Requirements for Nuclear Facilities³

3. Terminology

3.1 *Definitions*—Definitions relating to this specification are given in Terminology C 709.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *apparent porosity*—ratio of the volume of open pores to the exterior volume expressed as a percentage.

3.2.2 *baking/re-baking charge*—the number of billets in a baking/re-baking furnace run.

3.2.3 *bulk density*—the mass of a unit volume of material including both permeable and impermeable voids.

3.2.4 *extrusion forming lot*—the number of billets of the same size extruded in an uninterrupted sequence.

3.2.5 *green batch*—the mass of coke, recycle green mix, recycle graphite, and pitch that is required to produce a forming lot.

3.2.6 *graphite billet*—an extruded, molded, or iso-molded graphite artifact with dimensions sufficient to meet the designer's requirements for reactor components.

3.2.7 *graphite grade*—the 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*—the number of billets in a graphitizing furnace run.

3.2.9 *high purity nuclear graphite*—nuclear graphite whose Boron Equivalent content is less than 2 ppm.

3.2.10 *impregnation charge*—the number of billets in an autoclave cycle.

3.2.11 *isotropic nuclear graphite*—a graphite in which the isotropy ratio based on the coefficient of thermal expansion is 1.00 to 1.10.

3.2.12 *low purity nuclear graphite*—nuclear graphite whose Boron Equivalent content is greater than 2 ppm but less than 10 ppm.

3.2.13 *mix formulation*—the percentages of each specifically sized filler used to manufacture a graphite grade.

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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 Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Three Park Ave., New York, NY 10016-5990.

3.2.14 *molding forming lot*—the number of billets molded from a molding powder lot.

3.2.15 *molding powder lot*—a 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.

3.2.16 *near isotropic nuclear graphite*—a graphite in which the isotropy ratio based on the coefficient of thermal expansion is 1.10 to 1.15.

3.2.17 *nuclear graphite class*—the designation of a nuclear graphite based upon its forming method, isotropy, purity and density (see Table 1).

3.2.18 *production lot*—a specified number of billets made in accordance with this specification as determined by the purchaser.

3.2.19 *purification charge*—the number of billets in a purification run.

3.2.20 *recycle green mix*—ground non-baked billets or non-formed formulation 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 C 781 and measured over the temperature range 25 to 500°C, of between 3.5×10^{-6} and $5.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$.

5.2.1.3 The coke shall be sampled and distributed as described in Table 3.

TABLE 2 ASTM Graphite Grain Size Definitions from Terminology C 709

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

^A Grain size as defined in Terminology C 709.

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 shall consist of coal tar pitch. The specific binder used shall be identified to the purchaser and be traceable through the forming lot.

5.4 *Impregnant*—The impregnant shall consist of a petroleum or coal tar pitch and be traceable through the impregnation step.

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.

5.6 *Manufacture:*

5.6.1 *Formulation*—The mix formulation (as defined in 3.2.13) and recycle green mix fraction (as defined in 3.2.20) in the filler shall be recorded. This information shall be reported to the purchaser if requested.

5.6.2 *Forming*—The green carbon mix may be formed by extrusion, molding (including vibrationally molding), or isomolding.

TABLE 1 ASTM Standard Classes of Nuclear Graphite

Class ^A	CTE Isotropy Ratio ^B (α_{AG}/α_{WG})	Purity		Bulk Density, ^C g/cm ³ (min)	Class Designation
		Ash Content, ^B ppm (max)	Boron Equivalent, ^D ppm (max)		
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

^A These classes may be further modified by the grain size as defined in Terminology C 709.

^B Determined in accordance with Practice C 781.

^C Determined in accordance with Test Method C 838.

^D Determined in accordance with Practice C 1233.

TABLE 3 Inspection Sampling and Testing of Filler Cokes

Raw Material	Inspection Plan	Sampling Procedure	Tests and Test Methods
Filler coke	A representative sample of the coke shall be taken prior to the mixing step of manufacture	Sample in accordance with Practice D 346 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 C 781 shall be used to prepare test specimens for the measurement of coke CTE Measure the coke real density in accordance with Test Method D 2638

5.6.3 *Graphitization Temperature*—The graphitization temperature shall be determined on each billet using the procedure described in Practice **C 781**. Each billet tested in accordance with Practice **C 781** shall have a Specific Electrical Resistivity (SER) corresponding to a graphitization temperature of at least 2700°C.

6. Chemical Properties

6.1 Each graphite billet/production lot sampled in accordance with Section **11** shall conform to the requirements for chemical purity specified in **Table 4** or **Table 5**, and to the requirements of the purchaser.

6.2 The boron equivalent shall be calculated in accordance with Practice **C 1233**. The concentrations of at least the following elements shall be determined and used in the calculation: Boron, Cadmium, Chlorine, Cobalt, Dysprosium, Europium, Gadolinium, Lithium, Manganese, Nickel, Samarium, Silver, Titanium, Tungsten, and Vanadium. Specified boron equivalent limits are given in **Table 1**.

6.3 **Table X1.1** contains a list of chemical impurities typically found in graphite. The impurities are categorized as neutron absorbing impurities, oxidation promoting catalysts, activation relevant impurities, metallic corrosion relevant impurities, and fissile/fissionable elements. The suggested limits represent the reactor designer's preferences for chemical purity.

7. Physical and Mechanical Properties

7.1 Each graphite billet/production lot sampled in accordance with Section **11** shall conform to the requirements for physical properties prescribed in **Table 1** and **Tables 6-11** for the appropriate nuclear graphite class, and to the requirements of the purchaser. **Table X1.2** is a summary table of the properties reported in **Tables 6-11**.

TABLE 4 Chemical Purity Requirements for HP Class Nuclear Graphite

Test	Practice	Specification (ppm)
Ash Content	C 781	300 maximum
Chemical Impurities – Ca	C 781	< 30
Chemical Impurities – Co	C 781	< 0.1
Chemical Impurities – Fe	C 781	< 30
Chemical Impurities – Cs	C 781	< 0.1
Chemical Impurities – V	C 781	< 50
Chemical Impurities – Ti	C 781	< 50
Chemical Impurities – Li	C 781	< 0.2
Chemical Impurities – Sc	C 781	< 0.1
Chemical Impurities – Ta	C 781	< 0.1
Boron Equivalent	C 1233	2 maximum
Chemical Impurities – N	C 781	to be determined ^A
Relative Oxidation Rate in Air	C 781	to be determined ^A

^A Data are not currently available to establish this value.

TABLE 5 Chemical Purity requirements for LP Class Nuclear Graphite

Test	Practice	Specification (ppm)
Ash Content	C 781	1000 maximum
Chemical Impurities – Ca	C 781	< 100
Chemical Impurities – Co	C 781	< 0.3
Chemical Impurities – Fe	C 781	< 100
Chemical Impurities – Cs	C 781	< 0.3
Chemical Impurities – V	C 781	< 250
Chemical Impurities – Ti	C 781	< 150
Chemical Impurities – Li	C 781	< 0.6
Chemical Impurities – Sc	C 781	< 0.3
Chemical Impurities – Ta	C 781	< 0.3
Boron Equivalent	C 1233	10 maximum
Chemical Impurities – N	C 781	to be determined ^A
Relative Oxidation Rate in Air	C 781	to be determined ^A

^A Data are not currently available to establish this value.

TABLE 6 Physical and Mechanical Properties for Nuclear Graphite Classes IIHP and IILP

Test ^A	Practice	Specification
Apparent Porosity	C 781	14 % max
Thermal Conductivity at 25°C, AG	C 781	90 W/m·K min
Coefficient of Thermal Expansion (25-500°C), WG	C 781	3.5-6.0 × 10 ⁻⁶ °C ⁻¹
Tensile Strength, WG	C 781	22 MPa min
Flexural Strength, WG	C 781	35 MPa min
Compressive Strength, WG	C 781	65 MPa min
Dynamic Elastic Modulus, WG	C 781	15 GPa max
Dynamic Elastic Modulus, WG	C 781	8 GPa min
Stress-Strain Response and Modulus of Elasticity, WG	C 781	7 GPa min
Strain to Failure, WG	C 781	0.3 % min
Fracture Toughness, WG	C 781	0.8 MPa·√m min
Weibull Modulus, WG	C 781	15 min
Weibull Characteristic Value, WG	C 781	to be determined ^B

^A WG = With Grain; AG = Against Grain.

^B Data are not currently available to establish this value.

8. Other Requirements

8.1 The graphitized billets shall be handled and stored in such a manner 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 Formulation designation,
- 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 Graphitizing charge,
- 8.2.9 Position of billet in graphitization furnace,
- 8.2.10 Purification step (if performed),