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An American National Standard

Standard Specification for Isotropic and Near-isotropic Nuclear Graphites¹

This standard is issued under the fixed designation D 7219; 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

<u>1.2</u> 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: ASTM Standards: ²

C 559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles

C 709 Terminology Relating to Manufactured Carbon and Graphite

C 781 Practice for Testing Graphite and Boronated Graphite <u>Components</u> 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 2638Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer<u>Test Method for Real Density of</u> 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 Facilities³

https://standards.iten.arcatalog/standards/sist/e18/bcco-8be-49ec-80a2-7a7c227be142/astm-d7219-08

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.

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

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

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

3.2.5<u>3.2.4</u> green batch—the mass of coke, recycle green mix, recycle graphite, and pitch that is required to produce a forming lot. —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—an extruded, extruded, molded, or iso-molded graphite artifact with dimensions sufficient to meet the designer's requirements for reactor components.

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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.

3.2.7 graphite grade—the designation_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.

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3.2.8 graphitization charge—the number of billets in a graphitizing furnace run. ____number of billets of the same grade in a graphitizing furnace run.

3.2.9 *high purity nuclear graphite*—nuclear graphite whose Boron Equivalent content is less that 2 ppm. graphitizing furnace run—total number of billets graphitized together in one graphitization furnace.

3.2.10 *impregnation charge*—the number of billets in an autoclave cycle. high purity nuclear graphite—nuclear graphite with an Equivalent Boron Content less than 2 ppm.

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. 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.

<u>3.2.13</u> low purity nuclear graphite—nuclear graphite whose Boron Equivalent content is greater than 2 ppm but less that 10 ppm. —nuclear graphite with an Equivalent Boron Content greater than 2 ppm but less than 10 ppm.

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

3.2.14*molding forming lot*—the number of billets molded from a molding powder lot. _____percentages of each specifically sized filler used to manufacture a graphite grade.

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. <u>molding forming lot</u>_number of billets molded from a molding powder 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. 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 <u>near isotropic nuclear graphite</u>—graphite in which the isotropy ratio based on the coefficient of thermal expansion is 1.10 to 1.15.

<u>3.2.18</u> nuclear graphite class—the designation 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 *production lot*—specified number of billets made in accordance with this specification and additional requirements determined by the purchaser.

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

3.2.203.2.21 recycle green mix—ground non-baked billets or non-formed formulationnon used green mix manufactured in compliance with the mix formulation specified here.

4. Significance and Use

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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.

TABLE	1 ASTM	Standard	Classes	of	Nuclear	Graphite	

	OTE lastrony Datia ^B		Purity	Bully Density C		
Class ^A	CTE Isotropy Ratio ^B - $(\alpha_{AG}/\alpha_{WG})$	Ash Content, ^B ppm (max)	Boron Equivalent, ^D ppm (max)	Bulk Density, ^C g/cm ³ (min)	Class Designation	
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(see Table 2).

^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 2 ASTM Graphite Grain Size Definitions from Terminology C 709

Graphite Designation	Definition of Grains in the Starting Mix that are: ^A
Medium Grained Medium Grained Fine Grained Superfine Grained Ultrafine Grained Microfine Grained	Generally < 4 mm Generally < 4 mm Generally < 100 μm Generally < 50 μm Generally < 10 μm Generally < 2 μm
A Croin size as defined in Term	inglam, C 700

^A Grain size as defined in Terminology C 709.

^B For nuclear graphite, the maximum grain size is 1.68 mm in accordance with 5.2.1.6.

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 5.5×10^{-6} °C ⁻¹.

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 shall consist of coal tar pitch. The specific binder used shall be identified to the purchaser and be traceable through the forming lot. ____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 shall consist of a petroleum or coal tar pitch and be traceable through the impregnation step. —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.

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https://standards.iteh.ai/catalog/standards/sist/c187bcc6-88be-49ec-80a2-7a7c227be142/astm-d7219-08

TABLE 3 Inspection Sampling and Testing of Filler Cokes

Raw Material	Inspection Plan	Sampling Procedure	
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 Filler coke	The proc test sp
1. A sufficient sample for preparation of CTE test specimens			
		 A sufficient sample will be taken for additional testing. This sample shall be retained for a period specified by the graphite purchaser 	Measure Metho
	2. A sufficient sample will be taken for additional testing. This sample shall be retained for a period specified by the graphite purchaser	2. A sufficient sample will be taken for additional testing. This sample shall be retained for a period specified by the graphite purchaser	Measure Metho

5.6 Manufacture:

5.6.1 *Formulation*—The mix formulation (as defined in 3.2.133.2.14) and recycle green mix fraction (as defined in 3.2.203.2.21) in the filler shall be recorded. This information shall be reported to the purchaser if requested.

5.6.2 *Forming*—The green earbon mix may be formed by extrusion, molding (including vibrationally molding), or iso-molding. 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/productionproduction lot shall be sampled in accordance with Section 11shall conform to the requirements for chemical purity specified in Table 4 or Table 5, and to the requirements of the purchaser. 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 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 . 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 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. 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 billet/production production lot shall be sampled in accordance with Section 11 and 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

TABLE 4 Chemical Purity Requirements for HP Class-Nuclear Graphite							
<u>Class^A</u>	Coefficient of Thermal Expansion (25 to 500°C), WG °C ⁻¹	Thermal Conductivity at 25°C, AG, Wm ⁻¹ K ⁻¹ min	Te <u>n</u> stile		^B WG MPa, min <u>SCompreessification (ppm)ve</u>	<u>Dynamic Elastic</u> Modulus, WG, GPa	
Ash ConPracticent ^C	C 781	300 maximum	10.00				
Chemical Impurities - Ca	C 781	< 30					
Chemical Impurities - Co	s.iteh.al/c781log/star	hdards/sist/0.187bcc6-					
Chemical Impurities - Fe	C 781	- < 30					
Chemical Impurities – Cs	C 781	- < 0.1					
Chemical Impurities – V	C 781	-					
HHP HHP ChemicalImpurities — Ti IILP ChemicalImpurities — Li INHP ChemicalImpurities — Se INLP Chemical Impurities — Ta	$\begin{array}{r} < 50^{-6} \\ 3.5-5.5 \times 10^{-6} \\ \hline 6.78.1 \\ \hline \hline 6.781 \end{array}$	90 90 √50 90 € 90 € 80 0 80 01	22 22 22 22 22 20 20 20 20	95 35 95 35 36 30 30 30 30	65 65 65 60 60 60 60	8-15 8-15 8-15 8-15 8-15 8-15 8-1 8-1 8-15	
BoronEquivalent EIHP ChemicalImpurities—N EILP ENHP ENHP Relative Oxidation Rate in Air ENLP	$\begin{array}{r} \hline \textbf{C} \ \textbf{1233} \\ \hline \textbf{3.5-5.5} \times 10^{-6} \\ \hline \textbf{C} \ \textbf{78.1} \\ \hline \textbf{3.5-5.5} \times 10^{-6} \\ \hline \textbf{3.5-5.5} \times 10^{-6} \\ \hline \textbf{3.5-5.5} \times 10^{-6} \\ \hline \textbf{C} \ \textbf{78.1} \\ \hline \textbf{3.5-5.5} \times 10^{-6} \\ \hline \textbf{3.5-5.5} \times 10^{-6} \end{array}$	$\begin{array}{r} \begin{array}{r} 100\\ \underline{100}\\ \hline to \ be \ determined 100\\ \underline{100}\\ \underline{100}\\ \hline to \ be \ determined 100\\ \underline{100}\\ \hline to \ be \ determined 100\\ \underline{100}\\ \end{array}$	15 15 15 15 15 15 15 15 15 15	2 maximum1 21 24 21 21 21 21 21 21 21 21	45 45 45 45 45 45 45 45 45	8-15 8-15 8-15 8-15 8-15 8-15 8-15 8-15	
MIHP MILP MNHP MNLP	$\begin{array}{c} 3.5{-}5.5\times10^{-6}\\ \underline{3.5{-}5.5\times10^{-6}}\\ \underline{3.5{-}5.5\times10^{-6}}\\ \underline{3.5{-}5.5\times10^{-6}}\\ \underline{3.5{-}5.5\times10^{-6}}\end{array}$	$\frac{\frac{100}{100}}{\frac{100}{100}}$	<u>15</u> <u>15</u> <u>15</u> <u>15</u>	21 21 21 21 21	45 45 45 45 45	8–15 8–15 8–15 8–15 8–15	

BLE 4 Chemical Purity Requirements for HP Class Nuclear Graphite

⁴WG = With Grain; AG = Against Grain.

^BAt least one of the three strength measurements should be selected for production lot acceptance in agreement between the supplier and the purchaser. ^{AC}Data are not currently available to establish this value.