Designation: C 781 – 08

An American National Standard

Standard Practice for Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components[1]

This standard is issued under the fixed designation C 781; 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 practice covers the test methods for measuring the properties of graphite and boronated graphite materials. These properties may be used for the design and evaluation of high-temperature gas-cooled reactor components.

1.2 The test methods referenced herein are applicable to materials used for replaceable and permanent components as defined in Section 7 and Section 9, and includes fuel elements; removable reflector elements and blocks; permanent side reflector elements and blocks; core support pedestals and elements; control rod, reserve shutdown, and burnable poison compacts; and neutron shield material.

1.3 This practice includes test methods that have been selected from existing ASTM standards, ASTM standards that have been modified, and new ASTM standards that are specific to the testing of materials listed in 1.2. Comments on individual test methods for graphite and boronated graphite components are given in Sections 8 and 10, respectively. The test methods are summarized in Tables 1 and 2.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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:*[2]

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

C 561 Test Method for Ash in a Graphite Sample

C 577 Test Method for Permeability of Refractories

C 611 Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature

C 625 Practice for Reporting Irradiation Results on Graphite

C 651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature

C 695 Test Method for Compressive Strength of Carbon and Graphite

C 709 Terminology Relating to Manufactured Carbon and Graphite

C 747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance

C 749 Test Method for Tensile Stress-Strain of Carbon and Graphite

C 769 Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining an Approximate Young's Modulus

C 816 Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method

C 838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes

C 1039 Test Methods for Apparent Porosity, Apparent Specific Gravity, and Bulk Density of Graphite Electrodes

C 1179 Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air

C 1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials

C 1274 Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption

D 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D 1193 Specification for Reagent Water

D 2854 Test Method for Apparent Density of Activated Carbon

D 2862 Test Method for Particle Size Distribution of Granular Activated Carbon

D 3104 Test Method for Softening Point of Pitches (Mettler Softening Point Method)

D 4292 Test Method for Determination of Vibrated Bulk

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[1] This practice 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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[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.

***A Summary of Changes section appears at the end of this standard.**

TABLE 1 Summary of Test Methods for Graphite Components

NOTE—Designations under preparation will be added when approved.

	Fuel, Removable Reflector, and Core Support Elements; Pebble Bed Reflector, Key and Sleeves; and Dowel Pins	Permanent Side Reflector Elements and Dowel Pins	Core Support Pedestals and Dowels
Fabrication			
As Manufactured Bulk Density	C 838	C 838	C 838
Mechanical Properties			
Compressive Strength	C 695	C 695	C 695
Tensile Properties	C 749^A	C 749^A	C 749^A
Poisson's Ratio	E 132^B	E 132^B	E 132^B
Flexural Strength	C 651^A	C 651^A	C 651^A
Fracture Toughness	^{B}	^{B}	^{B}
Modulus of Elasticity	C 747	C 747	C 747
Physical Properties			
Bulk Density–Machined Specimens	C 559	C 559	C 559
Surface Area (BET)	C 1274	C 1274	C 1274
Permeability	C 577^{A,B}	C 577^{A,B}	C 577^{A,B}
Apparent Porosity	C 1039	C 1039	C 1039
Spectroscopic Analysis	^{B}	^{B}	^{B}
Electrical Resistivity	C 611	C 611	C 611
Thermal Properties			
Linear Thermal Expansion	E 228^A		
Thermal Conductivity	E 1461^A	E 1461^A	E 1461^A
Chemical Properties			
Oxidative Mass Loss	C 1179^B	C 1179^B	C 1179^B
Sulfur Concentration	C 816	C 816	C 816
Ash Content	C 561^A	C 561^A	C 561^A
Equivalent Boron Content	C 1233^A	C 1233^A	^{C}

[A] Modification of this test method is required. See Section 8 for details.
[B] New test methods are required. See Section 8 for details.
[C] There is no identified need for determining this property.

TABLE 2 Summary of Test Methods for Boronated Graphite Components

NOTE—Designations under preparation will be added when approved.

	Compacts			Neutron Shield Material
	Control Rod	Burnable Poison	Reserve Shutdown	
Bulk Density	C 838	C 838	C 838	D 4292
Linear Thermal Expansion	^{A}	E 228^A	E 228^A	^{B}
Particle Size	^{C}	^{C}	^{C}	D 2862
Mechanical Strength:				
Compressive Strength	C 695^A	C 695^A	C 695^A	^{B}
Impact Performance	^{B}	^{B}	^{B}	^{C}
Chemical Properties:				
Sulfur Concentration	^{C}	^{C}	^{C}	^{C}
Hafnium Concentration	^{C}	^{C}	^{C}	^{C}
Relative Oxidation Rate	^{C}	^{C}	^{C}	^{C}
Boron Analysis:				
Total Boron	^{C}	^{C}	^{C}	^{C}
Boron as Oxide	^{C}	^{C}	^{C}	^{C}
B_4C Particle Size	D 2862^D	D 2862^D	D 2862^D	D 2862^D

[A] Modification of this test method is required. See Section 10 for details.
[B] There is no identified need for determining this property.
[C] New test methods are required. See Section 10 for details.
[D] Additional test methods are required. See Section 10 for details.

Density of Calcined Petroleum Coke

D 5600 Test Method for Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D 7219 Specification for Isotropic and Near-isotropic Nuclear Graphites

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

E 132 Test Method for Poisson's Ratio at Room Temperature

E 228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

E 261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques

E 639 Test Method for Measuring Total-Radiance Temperature of Heated Surfaces Using a Radiation Pyrometer

E 1461 Test Method for Thermal Diffusivity by the Flash Method

3. Terminology

3.1 *Definitions*—Terminology C 709 shall be considered as applying to the terms used in this practice.

4. Significance and Use

4.1 Property data obtained with the recommended test methods identified herein may be used for research and development, design, manufacturing control, specifications, performance evaluation, and regulatory statutes pertaining to high temperature gas-cooled reactors.

4.2 The test methods are applicable primarily to specimens in the non-irradiated and non-oxidized state. Many are also applicable to specimens in the irradiated or oxidized state, or both, provided the specimens meet all requirements of the test method. The user is cautioned to consider the instructions given in the test methods.

4.3 Additional test methods are in preparation and will be incorporated. The user is cautioned to employ the latest revision.

5. Sample Selection

5.1 All test specimens should be selected from materials that are representative of those to be used in the intended application.

6. Test Reports

6.1 Test results should be reported in accordance with the reporting requirements included in the applicable test method. Where relevant, information on grade designation, lot number, billet number, orientation, and location (position of sample in the original billet) shall be provided.

6.2 Information on specimen irradiation conditions shall be reported in accordance with Practices C 625 and E 261 or referenced to source information of equivalent content.

GRAPHITE COMPONENTS

7. Description and Function

7.1 *Fuel and Removable Reflector Elements*:

7.1.1 A fuel element is a removable graphite element that contains channels for the passage of coolant gas, the fuel material (typically in the form of a compact containing coated particle fuel), the alignment dowel pins, and the insertion of a handling machine pickup head. A fuel element may also contain channels for reactivity control material (control rods), reserve shutdown compacts, and burnable poison compacts, and nuclear instrumentation.

7.1.2 The fuel elements serve multiple functions, including *(1)* vertical and lateral mechanical support for the fuel elements and removable reflector elements above and adjacent to them, and for the fuel, reactivity control materials, and nuclear instrumentation within them, *(2)* moderation of fast neutrons within the core region, *(3)* a thermal reservoir and conductor for nuclear heat generated in the fuel, *(4)* a physical constraint for the flow of coolant gases, and *(5)* a guide for and containment of fuel material, reactivity control materials, and nuclear instrumentation.

7.1.3 A removable reflector element is a removable graphite element that contains channels for the alignment dowel pins and the insertion of a handling machine pickup head. A removable reflector element may also contain channels for the passage of coolant gas, reactivity control materials (control rods), neutron flux control materials (neutron shield materials), and nuclear instrumentation.

7.1.4 The primary function of the removable reflector elements that are located at the boundaries of the active reactor core (fuel elements) is to provide for moderation of fast neutrons escaping from and reflection of thermal neutrons back into the active core region.

7.1.5 Except for support, guide, and containment of fuel material, removable reflector elements may also serve any of the functions listed in 7.1.2.

7.2 *Permanent Side Reflector Element*:

7.2.1 A permanent side reflector element is a graphite block that is designed to remain permanently in the core but may be removed for inspection and replacement, if necessary. A permanent side reflector element contains channels for alignment dowel pins. It may also contain channels for neutron flux control materials (boronated steel pins) and nuclear instrumentation, and recessed areas along its length on its outer periphery to provide channels for the passage of coolant gas between the element and the metallic lateral restraint for the reactor core.

7.2.2 The permanent side reflector elements encircle the active (fuel) elements and passive (removable reflector) elements of the reactor core and serve multiple functions, including *(1)* vertical and lateral mechanical support for the permanent side reflector elements above and beside them, *(2)* lateral mechanical support for the fuel, removable reflector, and core support elements, *(3)* moderation of fast neutrons within the reflector region, *(4)* reflection of thermal neutrons back into the core region, and *(5)* support, guide, and containment of nuclear instrumentation and neutron flux control materials (boronated steel pins) for reducing the neutron flux to metallic structures outside the permanent side reflector boundary.

7.3 *Core Support Pedestals and Elements*:

7.3.1 A core support pedestal is a graphite column that is designed to remain permanently in the core but can be removed for inspection and replacement, if necessary. A core support pedestal has a central reduced cross-section (dog bone shape) that at its upper end contains channels for the passage of coolant gas, alignment dowel pins, and the insertion of a handling machine pickup head, and at its lower end contains a recessed region for locating it with respect to the metallic structure that supports the graphite core support assembly. A core support element is a graphite element that contains channels for alignment dowel pins and the insertion of a handling machine pickup head. The core support elements may also contain channels for the passage of coolant gas, neutron flux control materials, and nuclear instrumentation.

7.3.2 The primary function of the core support pedestals is to provide for vertical mechanical support for core support elements and permanent side reflector elements above them. In addition, core support pedestals provide for lateral mechanical support for adjacent core support pedestals and permanent side reflector elements and physical constraint for the flow of coolant gases. The primary function of the core support elements is to provide for vertical mechanical support for core support, fuel, and removable reflector elements above them. In addition, core support elements provide for lateral mechanical support for adjacent core support and permanent side reflector elements and may provide for the physical constraint of coolant gases and for the support, guide, and containment of neutron flux control materials and nuclear instrumentation.

7.4 *Pebble Bed Modular Reactor Reflector Blocks*:

7.4.1 The fuel form of a pebble bed reactor is typically a 60 mm diameter sphere (pebble) containing graphite-carbon matrix and coated particle fuel.

7.4.2 The Pebble Bed reactor core structure consists of a graphite reflector supported and surrounded by a metallic core barrel. The graphite reflector is comprised of a large number of graphite blocks arranged in circular rings of separate columns. The graphite reflector can be subdivided into three subsystems, namely, the bottom, side, and top reflector. The side reflector may be split into an inner replaceable reflector and an outer permanent reflector. The graphite reflector blocks are interlinked within each circular ring by graphite keys set in machined channels in the reflector blocks. Certain Pebble Bed reactors designs have annular fuelled cores, and thus the reactor contains a central graphite column.

7.4.3 The primary function of the reflector blocks that are located at the boundary of the active reactor core (fuelled region) is to provide for moderation of fast neutrons escaping from, and reflection of thermal neutrons back into, the active core region.

7.4.4 Replaceable reflector blocks contain vertical channels for the reactivity control rods and reserve shutdown system. These channels contain graphite sleeves to eliminate cross flow of reactor coolant gas.

8. Test Methods

8.1 *Fabrication*:

8.1.1 *Coefficient of Thermal Expansion of Coke*—The method known as the flour-based graphitized rod CTE test is described in Annex A1.

8.1.2 *Bulk Density*—Determine bulk density on as-manufactured or machined specimens in accordance with Test Methods C 838 and C 559, respectively. Test Method C 838 includes shaped articles other than right circular cylinders and rectangular parallelepipeds. Test Method C 559 is used when a higher degree of accuracy is required. The procedures of Test Method C 559 are modified in Annex A2 to provide for the measurement of bulk density of non-uniform specimens.

8.1.3 *Graphitization Temperature*—The graphitization temperature of a full-size billet is estimated from a laboratory correlation between Specific Electrical Resistivity (SER) (Test Method C 611) and heat treatment temperature. The method is described in Annex A3.

8.2 *Mechanical Properties*:

8.2.1 *Compressive Strength*—Determine compressive strength in accordance with Test Method C 695.

8.2.2 *Tensile Strength*—Determination of tensile properties may also be made in accordance with Test Methods C 749 and E 132. The procedures of Test Method C 749 are modified in Annex A4 to provide for the measurement of the tensile stress-strain properties of specimens with glued ends, a convenient method that has been used in the past and verified for the testing of irradiated and non-irradiated (control) graphite specimens. The procedures of Test Method E 132 are modified in Annex A5 to provide specimen geometries and measurements specifically adapted for measuring the Poisson's ratio of graphite.

8.2.3 *Flexural Strength*—Determine flexural strength in accordance with Test Method C 651.

8.2.4 *Fracture Toughness*—A test method for determining fracture toughness is in preparation.

8.2.5 *Modulus of Elasticity*—Determine modulus of elasticity in accordance with Test Method C 747. Sonic velocity (Test Method C 769) may be used to give an approximate Young's Modulus.

8.3 *Physical Properties*:

8.3.1 *Bulk Density*—See 8.1.2.

8.3.2 *Surface Area*—The determination of the specific surface area (BET) shall be in accordance with Test Method C 1274.

8.3.3 *Gaseous Permeability*—Test Method C 577 for measuring gaseous permeability must be modified to permit the additional use of helium as the permeating medium and the use of alternative geometries for specimens and specimen holders. A second method is also in preparation to provide for materials with lower permeability than those covered by Test Method C 577.

8.3.4 *Apparent Porosity*—The determination of the apparent porosity shall be in accordance with Test Method C 1039.

8.4 *Thermal Properties*:

8.4.1 *Coefficient of Thermal Expansion of Graphite*—Determine the linear coefficient of thermal expansion (CTE) of graphite of all grain sizes in (general) accordance with Test Method E 228. Test specimens of cylindrical or prismatic geometry shall be used. The diameter or transverse-edge length, respectively, shall be no less than five times the maximum grain size of the graphite, and in no case smaller than 4 mm. The length of the test specimen shall be at least 25 mm, preferably 50 mm to 125 mm. The report shall include the temperature range over which the CTE was measured.

8.4.2 *Thermal Conductivity*—Calculate the thermal conductivity from the thermal diffusivity as determined by Test Method E 1461. The required calculation is described in Annex A6.

8.5 *Chemical Properties*:

8.5.1 *Oxidation*—Determine the oxidative mass loss in air in accordance with Test Method C 1179. (A test method for the determination of oxidation rate in air is in preparation.)

8.5.2 *Chemical Impurities*:

8.5.2.1 The chemical impurities shall be measured in accordance with D 5600. An alternate test method for determining impurity concentrations in nuclear graphite by spectroscopic methods is in preparation.

8.5.2.2 Determine sulfur concentration in accordance with Test Method C 816.

8.5.2.3 A method for determining boron levels is described in Annex A7.

8.5.3 *Ash Content*—Determination of ash shall be in accordance with Test Method C 561.

8.5.4 *Equivalent Boron Content*—Test Method C 1233 shall be used to calculate equivalent boron content. The elements specified in D 7219 shall be measured for the determination of the equivalent boron content.

BORONATED GRAPHITE COMPONENTS

9. Description and Function

9.1 *Control Rod Compacts*:

9.1.1 The control rod compacts are dispersions of approximately 40–weight % boron as boron carbide (B_4C) in a graphite matrix. The compacts are in the form of short, thick-walled tubular elements and are enclosed within the annuli of thin-walled metallic containers. These assemblies are connected to form sections of control rods.

9.1.2 The function of the control rod compacts is to absorb neutrons when inserted within the core, thereby providing a means for controlling the nuclear reactions.

9.2 *Burnable Poison Compacts*:

9.2.1 The burnable poison compacts are dispersions of approximately 1-weight % boron as boron carbide (B_4C) in a graphite matrix. The compacts are in the form of solid cylinders and are enclosed within channels in fuel elements.

9.2.2 The function of the burnable poison is to reduce the magnitude of the long-term reactivity changes that accompany fuel burnup.

9.3 *Neutron Shield Material*:

9.3.1 Neutron shield material consists of granules containing dispersions of approximately 25–weight % boron as boron carbide (B_4C) in a graphite matrix. These granules are enclosed within metallic containers located above the core.

9.3.2 The function of the neutron shield material is to reduce the neutron flux to adjacent metallic components.

9.4 *Reserve Shutdown Compacts*:

9.4.1 The reserve shutdown compacts are dispersions of approximately 40–weight % boron as boron carbide (B_4C) in a graphite matrix. These compacts are in the form of spherical elements or short cylindrical elements with rounded ends and are gravity fed from storage hoppers above the core into channels within fuel elements when an emergency shutdown of the reactor is required.

9.4.2 The function of the reserve shutdown compacts is to absorb neutrons thereby providing a means for rapidly stopping the nuclear reactions.

10. Test Methods for Boronated Graphite

10.1 *Particle Size*—Determine particle size of neutron shield material in accordance with Test Method D 2862. A new test method may be required for determining particle size in as-manufactured compacts.

10.2 *Bulk Density*—Determine bulk density on as-manufactured or machined specimens in accordance with Test Method C 838. Determine apparent bulk density of neutron shield material in accordance with Test Method D 2854.

10.3 *Linear Thermal Expansion*—Determine linear thermal expansion in general accordance with Test Method E 228. Modifications to Test Method E 228, which are in preparation and will be presented as an annex, are required to permit specimen geometries consistent with as-manufactured shapes.

10.4 *Mechanical Properties*:

10.4.1 Determine compressive strength in general accordance with Test Method C 695. An exception is for control rod compacts, for which Test Method C 695 is modified in Annex A8 to conform to specimen machining requirements for boron carbide-containing composite materials.

10.4.2 A test method for determining the impact performance of reserve shutdown compacts is in preparation.

10.5 *Chemical Properties*:

10.5.1 A test method for determining the concentrations of catalytic impurities is in preparation.

10.5.2 A test method for determining the sulfur concentration is in preparation.

10.5.3 A test method for determining the hafnium concentration is in preparation.

10.5.4 A test method for determining the relative rates of oxidation by primary coolant impurities is in preparation.

10.6 *Boron Analyses*:

10.6.1 A test method for determining the total boron content is in preparation.

10.6.2 A test method for determining boron as boron oxide (moisture-leachable boron compound) is in preparation.

10.6.3 Determine B_4C particle size prior to manufacture of component shapes in accordance with Test Method D 2862. A new test method may be required for determining B_4C particle size in as-manufactured components.

11. Keywords

11.1 boronated graphite; chemical properties; graphite; high temperature gas-cooled nuclear reactor; mechanical properties; neutronic properties; physical properties; thermal properties

ANNEXES

(Mandatory Information)

A1. QUALIFICATION CTE TEST FOR CALCINED COKE

A1.1 *Scope*—This method is applicable to the manufacture of graphite test rods from calcined petroleum or coal tar pitch coke of any origin.

A1.2 Sampling

A1.2.1 Coke samples that are submitted for testing shall properly represent those lots, barges, railcars, or trucks which are received by the manufacturing locations.

A1.2.2 The coke sample shall be collected in accordance with Practice D 346.

A1.2.3 Approximately 0.5 kg of calcined coke shall be riffled from a larger sample.

A1.3 Procedure

A1.3.1 *Preparation of Green Test Specimen*—The sample of calcined coke shall be split into equal parts and one half retained for possible recheck. The other half is dried at 110°C for 2 h and then crushed in one cycle to pass through a U.S. Standard 6.35- mm screen. The crushed sample is milled to flour so that at least 95 % passes a U.S. Standard No. 40 screen, and 40 to 60 % passes a U.S. Standard No. 200 screen. Then appropriate quantities of the flour and a suitable medium coal tar pitch binder (nominal softening point 110°C according to Test Method D 3104, crushed to pass a U.S. Standard No. 10 screen) are heated to about 150°C in a suitable laboratory scale mixer with occasional stirring. An extrusion aid may be added and mixed thoroughly. The mixture is then cooled or placed directly into a suitable heated laboratory scale extrusion press and tamped prior to extrusion. The internal diameter of the laboratory press may be 38 to 50 mm. The quantity of mix is sufficient when extruded to produce three test specimens 12 to 20 mm in diameter and 100 to 150 mm in length. The first test specimen extruded is discarded.

A1.3.2 *Baking*—The duplicate green specimens are packed without touching in a suitable sagger in bed of graphite particles or bed of coke and sand mixture (the pack passes a U.S. Standard No. 10 screen) then covered with about 50 mm of the same packing media. The sagger is placed into a furnace at 100°C and heated at about 90 to 120°C/h to 850 to 900°C and held for 1 to 3 h. The sagger shall be furnace cooled to less than 300°C before opening and unpacking the rods. The rods may be cleaned using coarse sandpaper if required.

A1.3.3 *Graphitizing*—The baked specimens are placed loosely in a graphite capsule and heated at approximately 15°C/min to a temperature above 2700° and held for 30 min. Graphitization shall be conducted under flowing argon and the capsule shall be cooled to less than 300°C before removing the rods.

A1.4 *Measuring the Coefficient of Thermal Expansion*—The rods will be cleaned and machined so that the end faces are parallel and normal to the longitudinal axis. The bulk density shall be measured according to Test Method C 559, and the resistivity measured according to Test Method C 611, and the values recorded. The density and resistivity shall be evaluated to ensure proper processing was achieved. The coefficient of thermal expansion (CTE) of the test specimens shall be measured along the longitudinal axis of the rod between room temperature and 500°C in accordance with Test Method E 228 and 8.4.1. The average of a single determination on each of the two specimens shall be recorded. A variation of the method involves measurement of the CTE at the baked stage and the use of an empirical relationship to estimate the CTE of the graphite.

A1.5 *Report*—The average CTE of the individual rods shall be reported in µm/m·°C (25 to 500°C). The averages of rod bulk density and resistivity shall also be reported.

A1.6 *Precision and Bias*—No precision statement has been determined for this test method.

A2. BULK DENSITY OF NON-UNIFORM TEST SPECIMENS

A2.1 The bulk density of test specimens other than right circular cylinders or rectangular parallelepipeds may be determined using Test Method C 559 provided the specimen volume can be determined within 0.15 %. (See 6.2 of Test Method C 559.)

A2.2 The net volume of a nonuniform, axisymmetric test specimen can be calculated if the shape can be broken down into simple geometric elements. Element volumes can be calculated with the aid of mensuration tables generally found in math and engineering handbooks. Sum the element volumes to obtain the net volume of the test specimen. Calculate the bulk density as in 7.3 of Test Method C 559.

A3. ESTIMATION OF THE GRAPHITIZATION TEMPERATURE OF FULL-SIZE BILLETS FROM THE MEASURED SPECIFIC ELECTRICAL RESISTIVITY

A3.1 The estimate of the graphitization temperature of a full-size billet is determined from a laboratory correlation between heat treatment temperature and the specific electrical resistivity (SER) of small specimens.

A3.2 Procedure

A3.2.1 Samples of appropriate dimensions as described in Test Method C 611 shall be cut from a full sized billet that has completed all processing steps prior to graphitization. The SER and bulk density of the cut samples shall be measured, using Test Methods C 611 and C 559, respectively, and reported along with the sampling diagram. At least three samples at each temperature shall be heat treated under controlled laboratory conditions at a minimum of five temperatures covering the range 2400 to 3100°C. A heating rate of 15°C/min shall be used. The samples shall be held at the heat treatment temperature for a minimum of 30 min. The SER of the heat treated specimens shall be determined at room temperature in accordance with Test Method C 611. An SER versus temperature correlation curve shall be developed and a value shall be established that represents the required full-size billet graphitization temperature, typically 2700°C. Errors in measurement will be determined from the scatter in the SER versus temperature plot. Typically, the temperature is monitored using an optical pyrometer calibrated by Test Method E 639. Note, a new SER value must be established if changes are made in raw materials, mix formulation, or processing procedures.

A3.2.2 The SER of each full-size graphite billet shall be measured at room temperature using a standard procedure developed by the supplier and approved by the purchaser. The value of SER determined on each billet shall be used to establish the graphitization temperature of that billet using the laboratory correlation.

A3.2.3 The SER derived graphitization temperature of each billet shall be reported to the purchaser.

A3.3 *Precision and Bias*—No precision statement has been determined for this test method.

A4. MODIFICATIONS TO TEST METHOD C 749 FOR GLUED-END SPECIMENS

A4.1 The test specimen configurations referred to in Test Method C 749, Fig. 9, incorporate integral grooved heads for mounting the specimens in the gripping devices and reduced gage sections to control fracture location. However, test parameters for some studies (irradiation and oxidation studies and quality assurance tests for many manufactured carbon and graphite articles) may impose such stringent requirements on volume, diameter, and geometry that the resultant test specimen may be simply a right circular cylinder. This annex deals with bonding connectors to cylindrical specimens to conduct tensile tests employing the load train and gripping devices detailed in Test Method C 749.

A4.2 *Test Specimen*—The test specimen shall be cylindrical with ends machined perpendicular to the longitudinal axis.

A4.2.1 The recommended test specimen size is 6.5 mm. (0.256 in.) diameter.

A4.2.2 The recommended height to diameter ratio for the specimen gage section is 4.

A4.2.3 The cylindrical surface shall be flat within 0.05 mm (0.002 in.), and the minimum diameter must not occur at either end of the specimen. The end faces of the specimen shall be perpendicular to the cylindrical surface to within 0.025 mm/mm. (0.001 in./in.) of diameter total indicator reading. Reasonable care shall be exercised to assure that all edges are sharp and without chips or other flaws.

A4.3 *Specimen Connectors*—The specimen connectors that are bonded to the specimen ends shall be sized to fit the gripping devices. The recommended material for the specimen connectors is 6061-T6 aluminum alloy. The end (bond) face of the connector shall be flat within 0.025 mm (0.001 in.) and perpendicular to the cylindrical axis of the connector within 0.001 in./in. (0.02 mm/mm) of diameter total indicator reading.

A4.4 *Attachment of Test Specimens to Specimen Connectors*—Specimen connectors shall be bonded to the test specimen with an epoxy or cyanoacrylate adhesive.

A4.4.1 The axial center line of the test specimen and specimen connectors shall be aligned during bonding using an appropriate alignment fixture. The run out tolerance for the finished assembly shall be within 0.025 mm. (0.001 in.) total indicator reading.

A4.4.2 An adhesive with a tensile shear strength (aluminum alloy to aluminum alloy) greater that 17 MPa (2500 psi) is recommended.

A4.4.3 The bond face of the specimen connector shall be etched or grit blasted, washed, dried, and degreased to promote a strong adhesive bond.

A4.4.4 The ends of the specimen shall be dust-, grease-, and moisture-free.

A4.5 *Test Procedures*—Follow the test procedures given in Test Method C 749 sections 8.1 through 8.4.

A4.5.1 If the fracture occurs within a distance less than 10 times the measured thickness of the adhesive joint at either end of the specimen, the strength results shall be reported but not included in the calculation of the average strength value. Experience has shown that when testing high strength graphite or graphites that have a large Poisson's ratio mismatch with that of the adhesive, specimens may fail at or very near the adhesive joint and yield invalid measurements. Under these circumstances, consideration should be given to the use of a specimen with a reduced gage section.

A4.6 *Tensile Property Calculations*—Calculate the strength, modulus of elasticity, and strain-to-failure as indicated in Section 9 of Test Method C 749.

A4.7 *Precision and Bias*—A round-robin test is being planned to develop precision and bias statements for this test method.

A5. MODIFICATIONS TO TEST METHOD E 132

A5.1 The following modifications to Test Method E 132 are required to more clearly define the method for measuring Poisson's ratio of graphite. The comments are arranged to apply to the relevant sections by number as designated in Test Method E 132.

A5.2 *Note 3*—This discussion is to be disregarded. This annex does not require the measurement of the shear modulus G. The three independent Poisson's ratios will be determined by the requirements following below.

A5.2.1 It is recommended that at least three pairs of extensometers be employed. The third pair shall be transverse to the direction of load and perpendicular to the first set of transverse extensometers (that is, perpendicular to the plane of Fig. 1). Note 5 applies with the additional transverse pair added to the configurations.

A5.3 The following considerations shall be added to this paragraph: The parent block of material shall be assumed to have cylindrical symmetry and the same shall be assumed to apply to all samples abstracted from the block. The samples shall be classified into two groups—axial (wherein the axis of symmetry is assumed to be parallel to the direction of loading), and radial (wherein the axis of symmetry is assumed to be perpendicular to the direction of loading). This classification is recognized to be a simplification, but symmetries of lower order are beyond the scope of this method.

A5.4 This paragraph applies with the following two exceptions: first, the width and thickness shall be equal within practical limitations; second, these two dimensions should exceed the maximum particle size by a factor of 3, or the results may be atypical. It should be emphasized that the specimen size should be as large as possible to reduce experimental difficulty and uncertainty in obtaining representative measurements.

A5.5 The considerations of this paragraph must be modified for the assumed cylindrical symmetry of the specimens. Let the *z*-coordinate be the axis of symmetry and the *x*- and *y*-coordinates be two other mutually orthogonal coordinates. In general, the *z*-axis will correspond to either the molding or the extrusion direction. The approximation of uniform, cylindrical symmetry may not be valid and, if necessary, should be confirmed by appropriate sampling. (**Warning—**The failure of an extensometer pair to separately yield the same value for Poisson's ratio may not be an indication of misalignment, but rather of failure of the material to meet perfect cylindrical symmetry.)

The following two new subsections are added to 7.1:

A5.5.1 Specimens machined such that a uniform load, *p*, is applied along the *z*-axis yield the following Poisson's ratio:

$$\mu_1 = -\left(\frac{\partial \varepsilon_x}{\partial p}\frac{\partial p}{\partial \varepsilon_z}\right)_p = -\left(\frac{\partial \varepsilon_y}{\partial p}\frac{\partial p}{\partial \varepsilon_z}\right)_p$$

where ε_x and ε_y are the transverse strains, and ε_z is the longitudinal strain. Both of these ratios shall be measured to assure that the approximation of cylindrical symmetry is valid.

A5.5.2 Specimens machined such that a uniform load can be applied in a direction perpendicular to the *z*-axis of symmetry (call this the *x*-axis) yield the following Poisson's ratios:

$$\mu_2 = -\left(\frac{\partial \varepsilon_y}{\partial p}\frac{\partial p}{\partial \varepsilon_x}\right)_p$$

$$\mu_3 = -\left(\frac{\partial \varepsilon_z}{\partial p}\frac{\partial p}{\partial \varepsilon_x}\right)_p$$

where ε_y and ε_z are the two transverse strains oriented in the *y* and *z* directions, and ε_x is the longitudinal strain. While the testing in only two directions is a minimum requirement, it is desirable that additional test samples be oriented so that the uniform load applied along the *y*-axis also be evaluated.

A5.6 This paragraph will not apply. A normal regression analysis may be used to determine the instantaneous slope dε/d*p* as appropriate, but no true proportional limit will usually exist. Each of the three μ_i can be calculated by the formula given in the method, except that the "lines" in general will not be linear and each μ_i will be a function of applied load *p*.

A6. DETERMINATION OF THERMAL CONDUCTIVITY FROM THERMAL DIFFUSIVITY

A6.1 The thermal conductivity may be calculated from the thermal diffusivity as determined by Test Method E 1461 from the following equation:

$$\lambda = \alpha C_p \rho$$

where:

λ = thermal conductivity, W/m·K,
α = diffusivity, m^2/s,
C_p = specific heat, J/kg·K, and
ρ = density, kg/m^3.

The required values of C_p are given in Table A6.1. The following equation may be used in place of the table for temperatures (*T*) between 300 and 3000 K:

$$C_p = \frac{1}{11.07\,T^{-1.644} + 0.0003688\,T^{0.02191}} \text{ J/Kg.K}$$

This equation reproduces the tabulated values within 2.0 % for the indicated range of temperatures.

TABLE A6.1 Recommended Values of the Specific Heat of Graphite

T (K)	C_p (J/kg·K)	T (K)	C_p (J/kg·K)	T (K)	C_p (J/kg·K)
300	713	1300	1940	2300	2142
400	961	1400	1972	2400	2154
500	1187	1500	2000	2500	2165
600	1370	1600	2024	2600	2174
700	1516	1700	2046	2700	2184
800	1632	1800	2066	2800	2193
900	1723	1900	2083	2900	2202
1000	1796	2000	2100	3000	2213
1100	1854	2100	2115	...	...
1200	1901	2200	2129	...	...

A6.2 (**Warning—**Calculation of thermal conductivity from thermal diffusivity is valid for most bulk graphites, but can lead to significant error for highly porous carbons and graphites where the initial heat pulse penetrates appreciably beyond the sample front face.)

A7. PREPARATION OF SOLUTIONS FOR DETERMINATION OF BORON CONTENT BY CALCINATION AND ICP-OES

A7.1 *Scope*—This test method is applicable to nuclear grade graphite.

A7.2 *Significance and Use*—This test method is for the determination of the boron content in both low-purity, and high-purity, nuclear graphite grades.

A7.3 *Summary of Test Method*—A test sample of the nuclear graphite is ashed at 700°C. In order to avoid boron losses, calcium hydroxide is added to the test sample before ashing. The residue is extracted with hydrochloric acid, and the boron concentration in the resulting solution is determined by Inductively Coupled Plasma–Optical Emission Spectrometry (ICP-OES) using simultaneous, or sequential, multi-elemental determination of elements. The solution is introduced to the ICP instrument by free aspiration or by an optional peristaltic pump. The concentration of the trace level of boron is then calculated by comparing the emission intensity from the sample with the emission intensities of the standards used in calibration.

A7.4 Apparatus

A7.4.1 *Balance*—Top loading, with automatic tare, capable of weighing to 0.0001 g, 150 g capacity.

A7.4.2 *Furnace*—Electric, capable of regulation of temperature at 700 ± 10°C with allowance for an air purge.

A7.4.3 *Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES)*—Either a sequential or simultaneous spectrometer is suitable, equipped with a quartz ICP torch and Radio Frequency (RF) generator to form and sustain the plasma and used with a calibration boron solution issued from certified standard.

A7.4.4 *Nebulizer*—A high-solids nebulizer is strongly recommended as this type of nebulizer reduces the possibility of clogging and minimizes aerosol particle effects.

A7.4.5 *Peristaltic Pump*—A peristaltic pump is strongly recommended to provide a constant flow of solution.

A7.4.6 *Crucibles*—Approximately 50 mm in diameter and 30 mm in height. Use boron free materials.

A7.4.7 *Sieves*—U.S. Standard No. 60 (250-micron) and No. 200 (74-micron) conforming to Specification E 11.

A7.4.8 *Boron–free Mill*—Laboratory size.

A7.4.9 *Magnetic Stirrer*.

A7.4.10 *Magnetic Stirring Bars*—Polytetrafluoroethylene (PTFE) coated, approximately 30 mm in length.

A7.4.11 *Volumetric Flasks*—25 mL and 1000 mL. Use boron free materials.

A7.4.12 *Plastic Bottles*—With screw cap, 25 mL and 1000 mL. Use boron free materials.

A7.5 Reagents

A7.5.1 *Purity of Reagents*—Use only reagents of recognized analytical grade, unless otherwise specified. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society[3] where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A7.5.2 *Water*—Complying with grade 2 of Specification D 1193.

A7.5.3 *Ethanol*—Absolute.

A7.5.4 *Hydrochloric Acid*—32 % by volume.

A7.5.5 *Argon*—Welding grade.

A7.5.6 *Calcium Metal*—Granules.

A7.5.7 *Solution No. 1*—Weigh 10 g calcium metal granules into 1000 mL of grade 2 water in a 1000 mL volumetric flask. Stir the solution using a magnetic stirrer until all calcium has dissolved. Let the solid residue deposit and decant the clear solution into a 1000 mL plastic bottle.

A7.5.8 *Solution No. 2*—Pour 400 mL hydrochloric acid and 400 mL of solution No. 1 into a 1000 mL volumetric flask. Dilute to volume with grade 2 water and mix thoroughly.

A7.5.9 *Standard Stock Solutions*—Use certified ready to use single-element Standard Stock Solutions.

A7.5.10 *Calibration Standards*—Use solution No. 2 for dilution of Stock Standard Solutions.

A7.6 *Sample Preparation*—Crush and divide the gross sample to obtain a laboratory analysis sample. Crush the analysis sample with a boron–free mill to pass a No. 60 sieve using Practice D 346. Approximately a 30 g representative portion of the minus No. 60 sieve shall be taken and dried to constant weight at 110°C and stored in a desiccator until cool and needed for the analysis.

A7.7 Preparation of Apparatus

A7.7.1 *ICP-OES Instrument*—Consult the manufacturer's instructions for operation of the inductively coupled plasma emission spectrometer.

A7.7.2 *Peristaltic Pump*—When a peristaltic pump is used, inspect the pump tubing and replace it, as necessary, before starting each day. Verify the solution uptake rate and adjust it to the desired rate.

A7.7.3 *ICP Excitation Source*—Initiate the plasma source at least 30 min before performing the analysis. Some manufacturers recommend even longer warm-up periods.

A7.7.4 *Wavelength Profiling*—Perform any wavelength profiling that is required in the normal operation of the instrument.

A7.7.5 *Operating Parameters*—Assign the appropriate operating parameter to the instrument task file so that the desired

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[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

element can be determined. Parameters to be included are element, wavelength, background correction points (optional), inter-element correction factors (optional), integration time, and internal standard correction (optional).

A7.8 Procedure

A7.8.1 Weigh 5.000 g (±1 mg) of dried nuclear graphite sample prepared in A7.6 into a labeled crucible (for high purity graphite a sample mass of 20.000 g (±1 mg) is recommended). Add a few drops of ethanol for better wetability and add 10 mL of solution No. 1. Dry the mixture at 150°C for 3 h. Place the crucible in a cold muffle furnace and heat directly to 700 ± 10°C until all carbonaceous matter is removed (approximately 12 to 16 h, or longer for larger samples). Let the crucible cool down, moisten the residue with a few drops of water and add 10 mL of hydrochloric acid. After waiting 1 h, pour the suspension into a 25 mL volumetric flask. Rinse the ceramic crucible with water into the volumetric flask, fill up to volume and mix thoroughly. Let the solid residue deposit and decant the clear solution into a 25 mL plastic bottle. The resulting solution is the analysis solution.

A7.8.2 Calibrate the ICP-OES instrument using matrix matched calibration standards (see A7.5) and determine the boron content in the analysis solution following Test Method D 5600.

A8. MODIFICATIONS TO TEST METHOD C 695

A8.1 The following modifications to Test Method C 695 are designed to permit measurements of compressive strength of boron carbide/graphite composite materials for which, due to differences in hardness of the two components, normal machining tolerances cannot be maintained. Paragraph numbering below corresponds to pertinent section numbers of Test Method C 695.

A8.1.1 The cushion pad thickness shall be 0.4 to 0.6 mm (0.015 to 0.025 in.).

A8.1.2 *Section 7*—This section shall apply up to the requirement on surface finish. It is recognized that tool drag will cause pullout of boron carbide particles; hence, all surfaces of the sample shall have a finish no rougher than a rating of 12.7 µm rms (500 µin.). Furthermore, these surfaces shall show no obvious regions of segregation between carbide and graphite (carbon) and shall not exhibit visible spalling. Reasonable care should be exercised to assure that edges are sharp and without flaws and chipping.

A8.1.3 *Section 7*—This section shall apply in its entirety with the exception that the minimum diameter shall be no less than five times the maximum particle size in the carbide or the graphite (carbon), whichever is larger. In those cases where the formed part does not permit abstracting samples meeting this requirement, it may be waived; but in no case shall the diameter be less than 7.6 mm (0.3 in.).

SUMMARY OF CHANGES

Subcommittee D02.F0 has identified the location of selected changes to this standard since the last issue (C 781–02) that may impact the use of this standard.

(*1*) Extensively revised and expanded this practice with the addition of test article manufacturing and test procedures for coke coefficient of thermal expansion determination, the procedure for establishing the graphitization temperature—specific electrical resistance calibration curve used in graphite production, and the boron content determination.

(*2*) Corrected the procedure for calculating specific heat.

(*3*) Added several standards to Section 2, Referenced Documents.