



Standard Practice for Testing Graphite and Boronated Graphite Materials for High- Temperature Gas-Cooled Nuclear Reactor Components¹

This standard is issued under the fixed designation C781; 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^{*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

- C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles
- C561 Test Method for Ash in a Graphite Sample
- C577 Test Method for Permeability of Refractories
- C611 Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature
- C625 Practice for Reporting Irradiation Results on Graphite
- C651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature
- C695 Test Method for Compressive Strength of Carbon and Graphite
- C709 Terminology Relating to Manufactured Carbon and Graphite
- C747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance
- C749 Test Method for Tensile Stress-Strain of Carbon and Graphite
- C769 Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining Young's Modulus
- C816 Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method
- C838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes
- C1039 Test Methods for Apparent Porosity, Apparent Specific Gravity, and Bulk Density of Graphite Electrodes
- C1179 Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air
- C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- C1274 Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption

¹ This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products—Products, Liquid Fuels, 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 1—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 | C838 | C838 | C838 |
| Mechanical Properties | | | |
| Compressive Strength | C695 | C695 | C695 |
| Tensile Properties | C749 ^A | C749 ^A | C749 ^A |
| Poisson's Ratio | E132 ^B | E132 ^B | E132 ^B |
| Flexural Strength | C651 ^A | C651 ^A | C651 ^A |
| Fracture Toughness | _B | _B | _B |
| Modulus of Elasticity | C747 | C747 | C747 |
| Physical Properties | | | |
| Bulk Density—Machined Specimens | C559 | C559 | C559 |
| Surface Area (BET) | C1274 | C1274 | C1274 |
| Permeability | C577 ^{A,B} | C577 ^{A,B} | C577 ^{A,B} |
| Apparent Porosity | C1039 | C1039 | C1039 |
| Spectroscopic Analysis | _B | _B | _B |
| Electrical Resistivity | C611 | C611 | C611 |
| Thermal Properties | | | |
| Linear Thermal Expansion | E228 ^A | | |
| Thermal Conductivity | E1461 ^A | E1461 ^A | E1461 ^A |
| Chemical Properties | | | |
| Oxidative Mass Loss | C1179 ^B | C1179 ^B | C1179 ^B |
| Sulfur Concentration | C816 | C816 | C816 |
| Ash Content | C561 ^A | C561 ^A | C561 ^A |
| Equivalent Boron Content | C1233 ^A | C1233 ^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 1—Designations under preparation will be added when approved.

| | Compacts | | | Neutron Shield Material |
|--------------------------------|--------------------|--------------------|---------------------|-------------------------------|
| | Control Rod | Burnable Poison | Reserve Shutdown | |
| Bulk Density | C838 | C838 | C838 | D4292 _B |
| Linear Thermal Expansion | _A | E228 ^A | E228 ^A | |
| Particle Size | _C | _C | _C | D2862 |
| Mechanical Strength: | | | | |
| Compressive Strength | C695 ^A | C695 ^A | C695 ^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 ₄ C Particle Size | D2862 ^D | D2862 ^D | D2862 ^D | D2862 ^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.

[D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis](#)
[D1193 Specification for Reagent Water](#)
[D2854 Test Method for Apparent Density of Activated Carbon](#)
[D2862 Test Method for Particle Size Distribution of Granular Activated Carbon](#)
[D3104 Test Method for Softening Point of Pitches \(Mettler Softening Point Method\)](#)
[D4292 Test Method for Determination of Vibrated Bulk Density of Calcined Petroleum Coke](#)
[D5600 Test Method for Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry \(ICP-AES\)](#)

- D7219 Specification for Isotropic and Near-isotropic Nuclear Graphites
- E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E132 Test Method for Poisson's Ratio at Room Temperature
- E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer
- E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques
- E639 Test Method for Measuring Total-Radiance Temperature of Heated Surfaces Using a Radiation Pyrometer (Withdrawn 2011)³
- E1461 Test Method for Thermal Diffusivity by the Flash Method

3. Terminology

3.1 *Definitions*—Terminology **C709** 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 **C625** and **E261** 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:*

³ The last approved version of this historical standard is referenced on www.astm.org.

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~~ 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 [C838](#) and [C559](#), respectively. Test Method [C838](#) includes shaped articles other than right circular cylinders and rectangular parallelepipeds. Test Method [C559](#) is used when a higher degree of accuracy is required. The procedures of Test Method [C559](#) 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 [C611](#)) 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 [C695](#).

8.2.2 *Tensile Strength*—Determination of tensile properties may also be made in accordance with Test Methods [C749](#) and [E132](#). The procedures of Test Method [C749](#) 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 E132 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 C651.

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 C747. Sonic velocity (Test Method C769) 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 C1274.

8.3.3 *Gaseous Permeability*—Test Method C577 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 C577.

8.3.4 *Apparent Porosity*—The determination of the apparent porosity shall be in accordance with Test Method C1039.

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 E228. 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~~, 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 E1461. 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 C1179. (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 D5600. 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 C816.

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 C561.

8.5.4 *Equivalent Boron Content*—Test Method C1233 shall be used to calculate equivalent boron content. The elements specified in D7219 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 **D2862**. 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 **C838**. Determine apparent bulk density of neutron shield material in accordance with Test Method **D2854**.

10.3 *Linear Thermal Expansion*—Determine linear thermal expansion in general accordance with Test Method **E228**. Modifications to Test Method **E228**, 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 **C695**. An exception is for control rod compacts, for which Test Method **C695** 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 B₄C particle size prior to manufacture of component shapes in accordance with Test Method **D2862**. A new test method may be required for determining B₄C 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

ASTM C781-08(2014)
(Mandatory Information)

<https://standards.iteh.ai/catalog/standards/sist/9161a112-7824-4523-82f9-8fc4d0a55b80/astm-c781-082014>

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 **D346**.

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