Designation: C 781 – 96

An American National Standard

Standard Practice for Testing Graphite and Boronated Graphite Components for High-Temperature Gas-Cooled Nuclear Reactors[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 those properties of graphite and boronated graphite materials that may be used for the design and evaluation of high-temperature gas-cooled reactors.

1.2 The test methods referenced herein are applicable to materials used for replaceable and permanent components as defined in Section 7 and include fuel and removable reflector elements; target elements and insulators; permanent side reflector elements; 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 Table 1 and Table 2.

1.4 *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:*

C 559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles[2]

C 560 Test Methods for Chemical Analysis of Graphite[2]

C 561 Test Method for Ash in a Graphite Sample[2]

C 577 Test Method for Permeability of Refractories[3]

C 625 Practice for Reporting Irradiation Results on Graphite[2]

C 626 Methods for Estimating the Thermal Neutron Absorption Cross Section of Nuclear Graphite[4]

C 651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature[2]

C 695 Test Method for Compressive Strength of Carbon and Graphite[2]

C 709 Terminology Relating to Manufactured Carbon and Graphite[2]

C 747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance[2]

C 749 Test Method for Tensile Stress-Strain of Carbon and Graphite[2]

C 816 Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method[2]

C 838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes[2]

C 1179 Test Method for Oxidative Mass Loss of Manufactured Carbon and Graphite Materials in Air[2]

C 1251 Test Method for Determination of Specific Surface Areas of Advanced Ceramic Materials by Gas Adsorption[2]

D 2854 Test Method for Apparent Density of Activated Carbon[2]

D 2862 Test Method for Particle Size Distribution of Granular Activated Carbon[2]

D 4292 Test Method for Vibrated Bulk Density of Calcined Petroleum Coke[5]

E 132 Test Method for Poisson's Ratio at Room Temperature[6]

E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer[7]

E 261 Practice for Determining Neutron Fluence Rate, Fluence, and Spectra by Radioactivation Techniques[8]

E 1461 Test Method for Thermal Diffusivity of Solids by the Flash Method[7]

3. Terminology

3.1 *Definition:*

[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.F on Manufactured Carbon and Graphite Products .

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 15.01.

[4] Discontinued; see 1985 *Annual Book of ASTM Standards*, Vol 15.01.

[5] *Annual Book of ASTM Standards*, Vol 05.02.

[6] *Annual Book of ASTM Standards*, Vol 03.01.

[7] *Annual Book of ASTM Standards*, Vol 14.02.

[8] *Annual Book of ASTM Standards*, Vol 12.02.

TABLE 1 Summary of Test Methods for Graphite Components

NOTE 1—Designations under preparation will be added as editorial changes when approved.

	Fuel, Removable Reflector, and Core Support Elements; Target Assemblies and Insulators; and Dowel Pins	Permanent Side Reflector Elements and Dowel Pins	Core Support Pedestals and Dowel Pins
Bulk Density:			
As-Manufactured Shapes	C 838	C 838	C 838
Machined Specimens	C 559	C 559	C 559
Thermal Properties:			
Linear Thermal Expansion	E 228[A]	E 228[A]	E 228[A]
Thermal Conductivity	E 1461[A]	E 1461[A]	E 1461[A]
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[A]	E 132[A]	E 132[A]
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
Oxidation Related Properties:			
Relative Oxidation Rate	C 1179[B]	C 1179[B]	C 1179[B]
Surface Area	C 1251	C 1251	C 1251
Permeability	C 577[AB]	C 577[AB]	C 577[AB]
Catalytic Impurities	C 560[B]	C 560[B]	C 560[B]
Sulfur Concentration	C 816	C 816	C 816
Porosity	[B]	[B]	[B]
Neutronic Impurities:			
Ash	C 561[A]	C 561[A]	C 561[A]
Spectroscopic Analysis	[B]	[B]	[B]
Thermal Absorption Cross Section	C 626[A]	C 626[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 as editorial changes 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:				
Catalytic Impurities	[C]	[C]	[C]	[C]
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 Section10 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 Section10 for details.

3.1.1 Terminology C 709 shall be considered as applying to the terms used in this practice.

4. Significance and Use

4.1 Properly 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 unirradiated and unoxidized 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, 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, graphite target assemblies and insulators, and nuclear instrumentation.

7.1.2 The fuel elements serve multiple functions, including (a) 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, target assemblies and insulators, and nuclear instrumentation within them, (b) moderation of fast neutrons within the core region, (c) a thermal reservoir and conductor for nuclear heat generated in the fuel, (d) a physical constraint for the flow of coolant gases, and (e) a guide for and containment of fuel material, reactivity control materials, target assemblies and insulators, 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), target assemblies and insulators, 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 *Target Assemblies and Insulators*:

7.2.1 A target assembly is a removable graphite canister consisting of an inner and outer annular sleeve and bottom and top end caps, that contains an annular channel for target materials and a central channel for the passage of cooling gas. A target assembly insulator is an annular sleeve containing a recessed area around its outer circumference that provides a thermal insulating gap between the insulating sleeve and the adjoining element channel. The inner circumference of the insulating sleeve provides a channel for the passage of coolant gas between the insulator and the target assembly.

7.2.2 The primary function of the target asemblies is to provide support, guide, and containment of target materials used for the production of special radioisotopes and a physical constraint for the flow of coolant gas. The primary functions of the target assembly insulators are to provide thermal insulation of the target assemblies from the fuel or removable reflector elements and a physical constraint for the flow of coolant gas.

7.3 *Permanent Side Reflector Elements*:

7.3.1 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.

7.3.2 The permanent side reflector elements encircle the active (fuel) elements and passive (removable reflector) elements of the reactor core and serve multiple functions, including (a) vertical and lateral mechanical support for the permanent side reflector elements above and beside them, (b) lateral mechanical support for the fuel, removable reflector, and core support elements, (c) moderation of fast neutrons within the reflector region, (d) reflection of thermal neutrons back into the core region, and (e) 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.

7.4 *Core Support Pedestals and Elements*:

7.4.1 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.

7.4.2 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.

8. Test Methods

8.1 *Bulk Density*:

8.1.1 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 A1 to provide for the measurement of bulk density of nonuniform specimens.

8.2 *Thermal Properties*:

8.2.1 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 ensure the reliability of measurements for coarse-grained graphites and to permit more convenient sizes for irradiation test specimens and manufacturing control.

8.2.2 Calculate the thermal conductivity from the thermal diffusivity as determined by Test Method E 1461. The required calculation is described in Annex Annex A2.

8.3 *Mechanical Properties*:

8.3.1 Determine Properties:

8.3.2 Determine compressive strength in accordance with Test Method C 695.

8.3.3 A new tension test method is under preparation which will provide for testing both unirradiated and irradiated specimens. 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 Annex A3 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 unirradiated (control) graphite specimens. The procedures of Test Method E 132 are modified in Annex A4 to provide specimen geometries and measurements specifically adapted for measuring the Poisson's ratio of graphite.

8.3.4 Determine flexural strength in accordance with Test Method C 651.

8.3.5 A test method for determining fracture toughness is in preparation.

8.4 *Oxidation Related Properties*:

8.4.1 A test method for determining relative rates of oxidation by water vapor is in preparation.

8.4.2 *Gaseous Permeability*—Test Method C 577 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 permeabilities than those covered by Test Method C 577.

8.4.3 *Catalytic Impurities*—Determine the concentration of iron, vanadium, and calcium in accordance with Test Methods C 560. New test methods for determining the concentrations of other catalytic impurities are in preparation.

8.4.4 *Sulfur*—Determine sulfur concentration in accordance with Test Method C 816.

8.4.5 A test method for determining porosity is in preparation.

8.5 *Neutronic Impurities*:

8.5.1 Determination of ash shall be in accordance with Test Method C 561. New test methods for determining the ash content of graphites with impurities which are lost during conventional ashing are in preparation.

8.5.2 A test method for determining impurity concentrations by spectroscopic techniques is in preparation.

8.5.3 Test Method C 626 is being revised to provide for calculation of both nonburnable and burnable boron-equivalent 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

10.1 *Particle Size*:

10.1.1 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*:

10.2.1 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.