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An American National Standard

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

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

- 1.1This 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. *
- 1.1 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 Table 1 and Table 2 Tables 1 and 2.

1.4

- 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
- C560Test Methods for Chemical Analysis of Graphite² 561 Test Method for Ash in a Graphite Sample 111-0781-08
- C561Test Method for Ash in a Graphite Sample²
- € 577 Test Method for Permeability of Refractories
- C 611 Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature
- C 625Practice for Reporting Irradiation Results on Graphite²
- C626Methods for Estimating the Thermal Neutron Absorption Cross Section of Nuclear Graphite 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
- C816Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method² 769 Test Method for Sonic Velocity in

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, Vol 05.05.volume information, refer to the standard's Document Summary page on the ASTM website.



TABLE 1 Summary of Test Methods for Graphite Components

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

	Fuel, Removable Reflector, and Gore Support Elements; PBMR Reflector, Keys and Sleeves; and Dowel Pins	Permanent Side Re- flector Elements and Dowel Pins	Core Support Pedes- tals-and Dowel Pins
Bulk Density:			
- As-Manufactured Shapes			
Machined Speci-	C 838	C 838	
mensC 838	C 559	C 559	
C 559			
As-Manufactured Shapes			
and Core Support Elements;	Permanent Side	Core Support Pedes-	
Pebble Bed Reflector,	Reflector Elements	tals	
Key and Sleeves;	and Dowel Pins	and Dowels	
and Dowel Pins			
Thermal Properties:			
Fabrication			
Linear Thermal Expansion	E 228A	E 228A	E 228A
- Thermal Conductivity	E 1461 ^A	E 1461 ^A	E 1461 ^A
Linear Thermal Expansion	C 838	C 838	C 838
Mechanical Properties:			
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 ⁴
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	В	В	В
Modulus of Elasticity	C 747	C 747	C 747
Oxidation Related Proper-			
ties:			
Physical Properties			
Relative Oxidation Rate	C 1179B	C 1179B	C 1179B
Relative Oxidation Rate	C 559	C 559	<u>C 559</u>
—Surface Area	C 1251	C 1251	C 1251
Surface Area	<u>C 1274</u>	<u>C 1274</u>	<u>C 1274</u>
- Permeability	C 577 ^{AB}	C 577 ^{AB}	C 577^{AB}
Permeability	<u>C 577^{A,B}</u>	<u>C 577^{A,B}</u>	<u>C 577^{A,B}</u>
Catalytic Impurities	C 1039	C 1039	C 1039
C 560 Spectroscopic	ASTM C	781-08 C 560^B	C 560 ^B
Analysis	<u> 110 1111 0</u>	<u>701 00</u>	
Spectroscopic Analysis	talog/standardg/sist/4672acl		14a0e74/astm ² c781-08
— Sulfur Concentration	C 816	C 816	C 816
Sulfur Concentration	<u>C 611</u>	<u>C 611</u>	<u>C 611</u>
Porosity		₽	
Thermal Properties			
Linear Thermal Expansion	E 228 ^B Neutronic		
	Impurities:		
Linear Thermal Expansion	E 228 ^A		
— Ash	C-561 ^{-A}	C-561 ^A	C 561 ^A
Ash	<u>E 1461^A</u>	<u>E 1461^A</u>	E 1461 ^A
— Spectroscopic Analysis			
Chemical Properties	-	_	
- Oxidative Mass Loss	C 1179 ⁸	8	₽
Oxidative Mass Loss	<u>C 1179</u> ^B	C 1179 ^B	C 1179 ^B
Thermal Absorption Cross	<u>C 816</u>	<u>C 816</u>	<u>C 816</u> €
Content	C 626 ^A	C 626 ^A	
Ash Content	<u>C</u> 561 ^A	C 561 ^A	C 561 ^A
Section	A		
Section	C 1233 ^A	C 1233 ^A	С

^A Modification of this test method is required. See Section 8 for details.

Manufactured Carbon and Graphite Materials for Use in Obtaining an Approximate Young's Modulus

C838Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes² 816 Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method

C1179Test Method for Oxidative Mass Loss of Manufactured Carbon and Graphite Materials in Air²

C1251Test Method for Determination of Specific Surface Areas of Advanced Ceramic Materials by Gas Adsorption² 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

^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 as editorial changes when approved.

	Compacts			Neutron	
	Control Rod	Burnable Poison	Reserve Shutdowr	Shield Material	
Bulk Density	C 838	C 838	C 838	D 4292	
Linear Thermal Expansion	A	E 228 ^A	E 228 ^A	В	
Particle Size	C	C	С	D 2862	
Mechanical Strength:					
Compressive Strength	C 695 ^A	C 695 ^A	C 695 ^A	₽	
Impact Performance	₽	₿	₿	€	
Compressive Strength	C 695 ^A	C 695 ^A	C 695 ^A	В	
Chemical Properties:				_	
Catalytic Impurities	6	6	6	6	
Catalytic Impurities	В	В	В	C	
Sulfur Concentration	_	_	_	_	
Chemical Properties:					
<u> </u>	6	6	6	6	
Sulfur Concentration	С	C	C	С	
Hafnium Concentration	c -	c	<u></u>	c	
Relative Oxidation Rate	C	C	C	C	
Boron Analysis:					
Total Boron	C	C	C	С	
	C	C	C	C	
Boron as Oxide		-	-		
B ₄ C 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.

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 2862Test Method for Particle Size Distribution of Granular Activated Carbon²
- D4292Test Method for Vibrated Bulk Density of Calcined Petroleum Coke
- 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 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 With a Vitreous Silica-Push-Rod Dilatometer
- E 261 Practice for Determining Neutron Fluence, Fluence Rate, Fluence, 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 of Solids by the Flash Method

3. Terminology

- 3.1Definition:
- 3.1.1Terminology C709
- 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 unon-irradiated and unon-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.

^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.



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 E261 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 Elements 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 (PBMR) Reflector Blocks:
- 7.4.1 The PBMR-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 sub-systems, namely, the bottom, side, and top reflector. The side reflector is-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.
- 7.4.2The 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.
- 7.4.34 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

- 8.1Bulk Density
- 8.1 Fabrication:
- 8.1.1Determine bulk density on as-manufactured or machined specimens in accordance with Test Methods C838 and C559, respectively. Test Method C838
- 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 A1 to provide for the measurement of bulk density of nonuniform specimens.
 - 8.2Thermal Properties:
- 8.2.1Determine 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 ensure the reliability of measurements for eoarse-grained graphite and to permit more convenient sizes for irradiation test specimens and manufacturing control.
- 8.2.2Calculate the thermal conductivity from the thermal diffusivity as determined by Test Method E1461. The required calculation is described in Annex are modified in Annex A2 to provide for the measurement of bulk density of non-uniform specimens.
- <u>8.1.3 Graphitization Temperature</u>—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.

8.3

- <u>8.2</u> *Mechanical Properties*:
- 8.3.1Determine compressive strength in accordance with Test Method C695
- 8.2.1 Compressive Strength—Determine compressive strength in accordance with Test Method C 695.
- 8.3.2A 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 C749 and E132
- 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 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 E132 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.
 - 8.3.3Determine flexural strength in accordance with Test Method C651
 - 8.2.3 Flexural Strength—Determine flexural strength in accordance with Test Method C 651.
 - 8.3.4A test method for determining fracture toughness is in preparation.
 - 8.3.5Determine modulus of elasticity in accordance with Test Method C747.
 - **8.4**Oxidation Related Properties
 - 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:
- 8.4.1Determine the relative rate of oxidation by mass loss in air in accordance with Test Method C1179
- 8.3.1 Bulk Density—See 8.1.2.
- 8.4.2A test method for determining surface area is in preparation.
- 8.4.3
- 8.3.2 Surface Area—The determination of the specific surface area (BET) shall be in accordance with Test Method C 1274.
- <u>8.3.3</u> 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 permeabilities permeability than those covered by Test Method C 577.
- 8.4.4*Catalytic Impurities*—Determine the concentration of iron, vanadium, and calcium in accordance with Test Methods C560. New test methods for determining the concentrations of other catalytic impurities are in preparation.
 - 8.4.5Sulfur—Determine sulfur concentration in accordance with Test Method C816
 - 8.3.4 Apparent Porosity—The determination of the apparent porosity shall be in accordance with Test Method C 1039.
 - 8.4.6A test method for determining porosity is in preparation.
 - 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 *Neutronic Impurities* Chemical Properties:
- 8.5.1Determination of ash shall be in accordance with Test Method C561. New test methods for determining the ash content of graphites with impurities that are lost during conventional ashing are in preparation.
 - 8.5.2A test method for determining impurity concentrations by spectroscopic techniques is in preparation.
- 8.5.3Test Method C626 is being revised to provide for calculation of both nonburnable and burnable boron-equivalent content.
- 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. -8329-17c514a0e74/astm-c781-08
 - 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 (B₄C) 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: