RDT Standard

**NEAR-ISOTROPIC PETROLEUM-COKE
BASED GRAPHITES FOR HIGH
TEMPERATURE GAS-COOLED REACTOR
CORE COMPONENTS**

OCTOBER 1977

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**DIVISION OF NUCLEAR POWER DEVELOPMENT,
DEPARTMENT OF ENERGY**

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RDT STANDARD

DEPARTMENT OF ENERGY
DIVISION OF NUCLEAR POWER DEVELOPMENT

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NEAR-ISOTROPIC PETROLEUM-COKE BASED GRAPHITES FOR HIGH TEMPERATURE GAS-COOLED REACTOR CORE COMPONENTS

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NEAR-ISOTROPIC PETROLEUM-COKE BASED GRAPHITES FOR HIGH TEMPERATURE GAS-COOLED REACTOR CORE COMPONENTS

1. SCOPE

This standard covers procurement requirements for extruded graphite logs, 15 in. (381 mm) or greater in diameter, manufactured with near-isotropic petroleum cokes and coal-tar pitch binders which are candidates or reference materials for replaceable fuel and reflector blocks for High-Temperature Gas-Cooled Reactors (HTGRs). The requirements are designed to produce the degree of lot-to-lot reproducibility which is required to ensure consistent and predictable properties and irradiation performance for specific graphite grades and to ensure traceability of the graphite logs to production processes and raw materials that affect performance.

1.1 Applicability. This standard is intended for use in the procurement of developmental and commercial grades of nuclear graphite which are to be evaluated on Department of Energy (DOE) funded programs for use as core components in HTGRs.

2. APPLICABLE DOCUMENTS

2.1 American National Standards (ANSI).

ANSI N45.2 Quality Assurance Requirements for Nuclear Power Plants

2.2 American Society for Testing and Materials (ASTM) Publications.

ASTM C 559	Density in Air of Manufactured Carbon and Graphite Articles by Physical Measurements
ASTM C 560	Chemical Analysis of Carbon and Graphite
ASTM C 561	Ash in Graphite
ASTM C 611	Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature
ASTM E 4	Verification of Test Machines
ASTM E 6	Terms Relating to Methods of Mechanical Testing
ASTM E 50	Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals
ASTM E 83	Verification and Classification of Extensometers

ASTM E 111	Young's Modulus at Room Temperature
ASTM E 115	Photographic Processing in Spectrochemical Analysis
ASTM E 116	Photographic Photometry in Spectrochemical Analysis
ASTM E 177	Use of the Terms Precision and Accuracy as Applied to Measurements of a Property of a Material
ASTM E 228	Linear Thermal Expansion with a Vitreous Silica Dilatometer

3. DEFINITION OF TERMS

3.1 Near-Isotropic Graphite. Near-isotropic graphites are defined as those meeting the "material specifications in 4.1.1, 4.2, 4.3 and 4.4; process specifications in 5.2 and 5.3.2; and the chemical and physical property requirements described in 9.3 and 9.4."

3.2 Graphite Log. A graphite log is defined as an extruded right cylinder of 15 in. (381 mm) or greater in diameter by 32 in. (813 mm) or greater in length.

3.3 Mix Formulation. Mix formulation is defined as the percentages of each raw material in the green mix used to manufacture a graphite grade.

3.4 Graphite Grade. A graphite grade is defined as the graphite product of a given supplier that is manufactured by a standard process from the same generic type of raw materials.

3.5 Raw Material and Processing Lots.

3.5.1 Calcined Coke or Recycle Graphite Lot.

3.5.1.1 Developmental Graphites. A calcined coke or recycle graphite lot is defined as either of the following:

1. The weight of coke or recycle graphite entering the comminution equipment in a continuous operation that is required to produce 100 logs of finished product.
2. The weight of coke or recycle graphite used to manufacture a small, but complete development batch of logs if it is less than the weight defined in 1 above.

3.5.1.2 Commercial Graphites. A calcined coke or recycle graphite lot is defined as the weight of coke or recycle graphite entering the comminution equipment in a continuous operation that is required to produce 20% of the logs of the order with the limitation that the coke lot represent a minimum of 100 or a maximum of 500 logs.

3.5.2 Binder or Additive Lot.

3.5.2.1 Developmental Graphites. A binder or additive lot is defined as either of the following:

1. The weight of binder or additive entering the mixing equipment in a continuous operation that is required to produce 100 logs of finished product.
2. The weight of binder or additive used to manufacture a small, but complete developmental batch of logs if it is less than the weight defined in 1 above.

3.5.2.2 Commercial Graphites. A binder or additive lot is defined as the weight of binder or additive entering the mixing equipment in a continuous operation that is required to produce 20% of the logs of the order with the limitation that the binder or additive lot represent a minimum of 100 or a maximum of 500 logs.

3.5.3 Extrusion Lot.

3.5.3.1 Developmental Graphites. An extrusion lot is defined as either of the following:

1. One hundred logs extruded in sequence.
2. The number of logs extruded to manufacture a small, but complete developmental batch of logs if less than 100 logs.

3.5.3.2 Commercial Graphites. An extrusion lot is defined as the number of logs extruded in sequence to produce 20% of the logs of the order with the limitation that the extrusion lot represent a minimum of 100 or a maximum of 500 logs.

3.5.4 Baking Lot.

3.5.4.1 Developmental Graphites. A baking lot is defined as either of the following:

1. The number of logs in a batch type baking furnace run.
2. One hundred logs baked in sequence if the baking process is continuous or semi-continuous.

3.5.4.2 Commercial Graphites. A baking lot is defined as either of the following:

1. The number of logs in a batch type baking furnace run.
2. Twenty % of the logs of the order with the limitation that the baking lot represent a minimum of 100 or a maximum of 500 logs baked in sequence if the baking process is continuous or semi-continuous.

3.5.5 Impregnant Lot.

3.5.5.1 Developmental Graphites. An impregnant lot is defined as either of the following:

1. The weight of impregnant required to impregnate 100 logs in sequence.
2. The weight of impregnant required to impregnate in sequence a small but complete developmental batch of logs if it is less than 100 logs.

3.5.5.2 Commercial Graphites. An impregnant lot is defined as the weight of impregnant required to impregnate in sequence 20% of the logs of the order with the limitation that the impregnant lot represent a minimum of 100 or a maximum of 500 logs.

3.5.6 Graphitizing Lot. A graphitizing lot is defined as the number of logs in a graphitizing furnace run.

4. RAW MATERIALS

4.1 Fillers.

4.1.1 Type, Size, and Sampling. The filler shall consist of calcined near-isotropic petroleum coke. Recycle graphite from near-isotropic graphite manufactured in compliance with this standard (requirements of Sections 8 and 9 excepted) may also be used. The proportion of recycle graphite in the raw material formulation shall not exceed 25 wt % of the filler. All filler particles shall pass through a number 12 U.S. Standard sieve [0.066 in. (1.68 mm)]. The calcined coke shall be sampled and distributed as described in Table 1.

4.1.2 Test for Reproducibility of Calcined Coke. Calcined coke samples obtained as described in Table 1 shall be fabricated into graphite specimens, which shall be tested for coefficient of thermal expansion (CTE) as described in Appendix A. The mean CTE value resulting from these tests shall represent the reference CTE value,¹ against which results of the same tests on samples from subsequent coke lots will be compared to evaluate reproducibility.

4.2 Binder. The binder shall consist of coal-tar pitch.

4.3 Impregnant. The impregnant shall consist of either a coal-tar pitch or a petroleum pitch. The type selected shall be identified to the purchaser.

4.4 Manufacturing Additives. Additives may be used during manufacture to improve the product. The supplier shall certify to the purchaser that the additives are the same and in the same proportions as those used to manufacture prototype, preproduction or production logs of the grade in question that has been qualified by characterization and irradiation tests and found to have satisfactory properties and irradiation behavior.

5. MANUFACTURE

5.1 Formulation. The weight percent of recycle graphite in the filler shall be specified to the purchaser.

5.2 Forming. The green carbon mix shall be formed by extrusion.

5.3 Degree of Graphitization. The degree of graphitization shall be determined on each log as follows:

5.3.1 Laboratory Correlation of Specific Electrical Resistivity and Graphitization Temperature. Samples cut from a full-size baked log or full size baked logs of the graphite grade being procured shall be graphitized under controlled laboratory conditions at 2700°C (2973 K). The specific electrical resistivity (SER) of the graphitized specimens or logs shall be measured at room temperature using ASTM C 611 or an equivalent supplier's standard test method. A SER value shall be established that represents a graphitization temperature of 2700°C (2973 K) for the grade in question. A new SER value shall be established if significant changes are made in raw materials or processing procedures. The supplier's procedure shall be approved by the purchaser.

¹Reference CTE values shall be established for only those coke lots used in graphite logs that have been evaluated and found to have acceptable as-received properties and irradiation performance. A reference CTE value shall be established for the coke used in each graphite grade qualified for potential HTGR application.

Table 1. Inspection, Sampling, and Testing of Raw Materials

Raw Material	Inspection Plan	Sampling Procedure	Method of Test
Calcined coke filler	The coke shall be sampled as it is transferred from the storage facility to the comminution equipment. One composite sample shall be collected for each lot. Each lot shall be given a number that can be traced to the extruded logs manufactured from that lot.	<p>Three 11 lb (5 kg) samples shall be collected from each lot using the same sampling interval. These samples shall be combined and reduced by cone and quartering or riffing to the following:</p> <ul style="list-style-type: none"> a. A 4.4 lb (2 kg) sample for storage in sealed plastic or glass container. This sample shall be retained for a minimum of 5 years. b. A 6.6 lb (3 kg) sample for preparation of CTE test specimens. c. A 6.6 lb (3 kg) sample shall be sent to the purchaser for the exclusive purpose of performing tests that satisfy the requirements of this standard, if required. This material shall not be transmitted by the purchaser to a third party without the approval of the supplier. 	The procedure in Appendix A shall be used to prepare test specimens and for measurement of CTE.

5.3.2 Measurement of Logs. The SER of each graphite log shall be measured at room temperature using a standard supplier developed procedure. The procedure shall be approved by the purchaser. The measured value shall be used to identify a graphitization temperature for each log using the SER versus graphitization temperature correlation established as described in 5.3.1 above. Each log shall have a SER that corresponds to a graphitization temperature of 2700°C (2973 K) or higher.

6. HANDLING AND STORAGE

Graphitized logs shall be brushed clean after removal from the graphitization furnace. During subsequent handling and storage, precautions shall be taken to protect the logs from contaminants other than ambient air.

7. IDENTIFICATION

Each graphite log shall be marked with a unique log identification number. Each log shall be traceable through these identifying numbers to each of the following:

1. Formulation designation.
2. Calcined coke lot.
3. Recycle graphite lot.
4. Binder lot.
5. Additive lot.
6. Extrusion lot.
7. Baking lot.
8. Impregnant lot.
9. Graphitizing lot.
10. Position of log in graphitization furnace.

8. IMPURITIES

Maximum impurity contents shall be equal to or less than the following in ppm by weight. The graphite logs shall be sampled for impurity content as described in Table 2 and analyses shall be made as described in ASTM C 560, ASTM C 561, or Appendix B.

Ash	1000
Iron (Fe)	100
Vanadium (V) + Titanium (Ti)	100
Calcium (Ca)	5
Boron (B)	5

9. PHYSICAL PROPERTIES

9.1 Density. The bulk density of each graphite log shall be 1.70 g/cm^3 (Mg/m^3) or greater. The density shall be measured according to the procedure described in Appendix C.

9.2 Tensile Strength. The mean axial tensile strength of four replicate specimens shall equal or exceed 1250 psi (8.6 MPa). Each graphite log shall be sampled as described in Table 2.

9.3 Thermal Expansivity. The coefficient of linear thermal expansion α shall be measured from 22° to 500°C (295 to 733 K) on logs selected at random from each lot. The mean values of α , measured in the axial and radial directions, shall be between 3 and $6 \times 10^{-6} \text{ }^\circ\text{C}^{-1} (\text{K}^{-1})$. The coefficient of volume thermal expansion $\alpha(\text{axial}) + 2\alpha(\text{radial})$ shall be equal to or greater than $10 \times 10^{-6} \text{ }^\circ\text{C}^{-1} (\text{K}^{-1})$. The graphite logs shall be sampled as described in Table 2.

9.4 Anisotropy Ratio. The anisotropy ratio, $\alpha(\text{radial})/\alpha(\text{axial})$ shall be calculated from values measured as described in 9.3 above. The ratio shall be between 0.9 and 1.4.

10. INSPECTION, SAMPLING, AND TESTING

Inspection, sampling, and testing necessary to show compliance with the requirements of this standard are given in Tables 1 and 2, and Appendices A, B, and C. Unless otherwise stated, the confidence level on all property requirements shall be 90/90 (90% of the material exceeds the specified value 90% of the time).

11. VERIFICATION OF COMPLIANCE WITH REQUIREMENTS

11.1 Compliance. The supplier shall submit a certification of compliance with each graphite purchase verifying that the requirements of this standard have been met.

11.2 Access. A purchaser's representative shall have access to those areas of the supplier's plant necessary to inspect all test equipment, testing operations and test records used to satisfy the requirements of this standard.

12. REJECTION

Failure to meet any of the requirements of this standard shall be cause for rejection of the involved graphite logs. Nonconforming items shall be processed in accordance with Section 14 (Section 16 of ANSI N45.2).

Table 2. Inspection, Sampling and Testing of Manufactured Graphite Logs

Property	Inspection Plan	Sampling Procedure	Method of Test
1. Impurities	Each lot of graphitized logs shall be qualified based on test results on samples from logs randomly selected from the lot. A log selection plan shall be included in the purchase specification.	A sample shall be taken along the longitudinal axis of the log between 1 to 16 in. (25 to 406 mm) from either end of the log. A log sampling plan shall be included in the purchase specification.	Ash: ASTM C 651 shall be used. B, Fe, Ti, V or Ca: ASTM C 650 or Appendix B shall be used.
2. Tensile Strength	Each log shall be tested.	A minimum of four specimens shall be taken with the long dimension of the specimen parallel to the longitudinal axis of the log. The specimens shall be taken from a cylindrical core of approximately 0.625 in. (16 mm) diameter whose longitudinal axis lies along the longitudinal axis of the log. The specimens shall be taken within 4 in. (101 mm) of the center of the log. A sampling procedure shall be included in the purchase specification.	Appendix D shall be used.

(continued)

Table 2. Inspection, Sampling and Testing of Manufactured Graphite Logs (continued)

Property	Inspection Plan	Sampling Procedure	Method of Test
3. Thermal Expansivity	Each lot of graphitized logs shall be qualified based on test results on samples from logs randomly selected from the lot. A minimum of two logs shall be selected. A log selection plan shall be included in the purchase specification.	A minimum of four specimens shall be taken adjacent to each other. Two specimens shall have their long dimensions parallel to the longitudinal axis of the log (axial) and two shall have their long dimensions parallel to the radial axis of the log (radial). The location of the specimens in the log and a sampling procedure shall be included in the purchase specification.	ASTM E 228 shall be used.
4. Bulk Density	Each whole log shall be measured.	Each whole log shall be measured.	Appendix C shall be used.
5. Degree of Graphitization	Each log shall be measured for SER and the value shall be converted to a corresponding graphitization temperature.	Each whole log shall be measured.	Standard supplier developed and approved procedures shall be used to measure the SER of the logs, and to establish and use the correlation between SER and graphitization temperature.

13. SHIPPING CONTAINER MARKING

Each shipping container shall be clearly marked with the following:

1. Manufacturer's name.
2. Manufacturer's grade number.
3. Manufacturer's log number or numbers.
4. Purchaser's purchase order number.

14. QUALITY ASSURANCE

The supplier shall maintain a quality assurance program in accordance with ANSI N45.2 with the following clarifications and exceptions.

<u>Section</u>	<u>Clarification or Exception</u>
1.1, 1.2 and 1.3, Purpose, Scope and Responsibility	ANSI N45.2 states the standard is for quality assurance program for <u>nuclear power plants</u> . ANSI N45.2 is being applied to this graphite standard because the graphite is a key component in an HTGR.
2. Quality Assurance Program	This section applies in its entirety and the program shall be described in a Quality Assurance Manual or equivalent document, which shall be submitted to the purchaser for approval.
3. Organization	Applies in its entirety.
4. Design Control	This entire section is <u>NOT</u> applicable because no reactor component design work is to be accomplished by the supplier.
5. Procurement Document Control	Applies in its entirety.
6. Instructions, Procedures and Drawings	Applies in its entirety.
7. Document Control	Applies in its entirety.
8. Control of Purchase Material Equipment and Services	Applies in its entirety.

<u>Section</u>	<u>Clarification or Exception</u>
9. Identification and Control of Materials, Parts and Components	Applies in its entirety.
10. Control of Special Processes	Applies in its entirety.
11. Inspection	Applies in its entirety.
12. Test Control	This entire section is <u>NOT</u> applicable because the purchaser is responsible for the design and the tests to demonstrate the item will perform satisfactorily in-service.
13. Control of Measuring and Test Equipment	Applies in its entirety.
14. Handling, Storage and Shipping	Applies in its entirety.
15. Inspection, Test and Operating Status	Applies except for the last paragraph regarding operating status of nuclear power plant valves and switches.
16. Nonconforming Items	Applies in its entirety.
17. Corrective Action	Applies in its entirety.
18. Quality Assurance Records	<p>The first two paragraphs of Section 18 apply. In addition the following records are required to be transmitted to the purchaser for approval at the time of shipment of material under this purchase order.</p> <ul style="list-style-type: none">a. Statement that the requirements of this standard have been met.b. Copy of correlation data for electrical resistivity and graphitization temperature in accordance with 5.3.

SectionClarification or Exception

- c. Test results for electrical resistivity, impurity content, density, tensile strength, thermal expansivity and anisotropy ratio which were obtained on graphitized logs. Test results shall be identified to individual log numbers. Test results for coke CTE which were obtained on test specimens fabricated from the coke used to produce graphitized logs. Test results shall be identified to individual log numbers.
- d. Copies of any inspection reports or other deviation or nonconforming reports applicable to the material being shipped.

Other quality related records (such as audit reports, process or personnel qualifications, manufacturing procedures, furnace run sheets and final dimensional inspection data) shall be maintained by the supplier for a period of ten years after completion of shipment. These records shall be available to the purchaser upon request during this period.

19. Audits

Applies in its entirety.

APPENDIX A

QUALIFICATION TEST FOR CALCINED NEAR ISOTROPIC PETROLEUM COKE

A.1 SCOPE

This method is applicable to calcined petroleum cokes of any origin.

A.2 SAMPLING

Approximately 1.1 lb (0.5 kg) of calcined coke are obtained from the 6.6 lb (3 kg) samples described in Table 1 by cone and quartering or riffing while progressively crushing to smaller size particles as the sample is split down.

A.3 PROCEDURE

A.3.1 Preparation of Green Test Specimens. The sample of calcined coke is split; one-half is retained for possible recheck. The other half is milled to flour so that 47 to 57% passes a U.S. Series 200 mesh screen. Then, 0.44 lb (0.200 kg) of the flour and 0.19 lb (0.085 kg) medium coal-tar pitch binder [110°C (383 K), SP, crushed to pass a 10 mesh screen] are slowly heated in a suitable laboratory mixer with occasional stirring to 150°C (423 K). At this point, 0.013 lb (0.005 kg) of extrusion oil, such as Shell #79 or Mobil Promor 102, or their equivalents, are added to the mix and stirred in. The mixture is mixed for a minimum of 15 min at 150°C (423 K). The container is removed from the heat and allowed to cool with stirring to 130°C (403 K).

A suitable laboratory press with a 1.5 in. (38 mm) diameter mud chamber is preheated to about 115°C (388 K); the green mix is tamped into the mud chamber and extruded. This amount of mix is enough for three test specimens approximately 0.60 in. (15.4 mm) in diameter by 6 in. (152 mm) in length.

A.3.2 Baking. The green specimens are packed (not touching) in a suitable sagger on a bed of petroleum coke/sand mixture (10 to 65 mesh) then covered with about 2 in. (50.8 mm) of the same packing media. The sagger is placed into a furnace at 100°C (373 K), and heated at 110° to 120°C/h to 850°C (1123 K) and held for 1 to 2 h. Before opening, the sagger should cool to 300°C (573 K). After unpacking the rods are cleaned using coarse sandpaper if necessary.

A.3.3 Graphitizing. The baked specimens are placed loosely in a graphite capsule and heated at approximately 15°C/min to 2800°C (3073 K) and held for 30 min. Graphitization shall be conducted in nitrogen, argon or helium and the capsule shall be cooled to 300°C (573 K) before removing the rods.

A.4 MEASURING AND REPORTING COEFFICIENT OF THERMAL EXPANSION

CTE of the test specimens shall be measured along the longitudinal axis over a minimum 50°C (50 K) range between 0° to 100°C (273 to 373 K) according to ASTM E 228. The average of single determinations on each of the three specimens shall be reported.

A.5 PRECISION OF MEASUREMENTS

Replicate measurements on a given sample shall not differ by more than $1.7 \times 10^{-7}^{\circ}\text{C}^{-1}$ (K^{-1}) at 90% confidence level and $2.1 \times 10^{-7}^{\circ}\text{C}^{-1}$ (K^{-1}) at 95% confidence level.

APPENDIX B

SPECTROCHEMICAL ANALYSIS OF NUCLEAR GRAPHITE BY THE POWDER D.C. ARC TECHNIQUE

B.1 SCOPE

This method covers the determination of the following elements in nuclear graphite in the concentration ranges¹ in ppm by weight shown:

Iron	0.5 - 1000
Vanadium	0.5 - 500
Titanium	1.0 - 50
Boron	0.05 - 1
Calcium	1.0 - 1000

B.2 SUMMARY OF THE METHOD

Three parts graphite drillings are diluted with one part recrystallized silver chloride to act as a buffer and carrier. The mixture is packed into the crater of a graphite cup. Direct current arc excitation is used. The spectra are recorded photographically and the intensity of selected lines is determined photometrically and the concentration of each element is read from a curve relating log intensity to log concentration.

B.3 APPARATUS

B.3.1 Sample Preparation Equipment. The sample representative of the material shall be obtained by drilling with a carbide tipped drill and shall be reduced in weight using cone and quartering or riffing and comminuted using a plastic vial and plastic cup. Sampling of bulk graphite is discussed in ASTM C 560.

B.3.2 A D.C. arc source capable of providing a current of 25 to 30 A.

B.3.3 Spectrograph having sufficient resolving power and linear dispersion to separate clearly the analytical lines from other lines in the spectral region 2400 Å (240 nm) to 3400 Å (340 nm). These conditions are satisfied by instruments having a reciprocal linear dispersion of 4 Å (0.40 nm) to 8 Å (0.8 nm) per mm at 3000 Å (300 nm).

¹For higher concentrations dilute sample with high purity graphite.

B.3.4 Photographic processing equipment, providing developing, firing, washing and drying operations and conforming to the requirements of ASTM E 115.

B.3.5 Microphotometer having a precision of $\pm 1.0\%$ for transmittance between 5% and 90% loss.

B.4 PURITY AND CONCENTRATION OF REAGENTS

B.4.1 The purity and concentration of chemical reagents shall conform to the requirements prescribed in the recommended practices for apparatus, reagents, and safety precautions for chemical analysis of metals (ASTM E 50).

B.4.2 Reagent grade silver chloride shall be recrystallized from ammonium hydroxide using the method described in ASTM E 2 SM 8 13.¹ The recrystallized silver chloride shall be stored in an opaque bottle and every effort maintained to prevent exposure to ultraviolet light.

B.4.3 Electrodes, High Purity Graphite. The lower electrode shall be similar to ASTM S 14 except that the cavity is deeper by 0.37 in. (9.3 mm) to accept the large charge.² The counter electrode shall be in accordance with ASTM C 1.

B.4.4 Photographic Emulsions. Eastman process, SA No. 1, or SA No. 2, or equivalent. Either films or plates may be used.

B.4.5 Photographic Processing Solutions. The formulas for processing solutions are given in ASTM E 115.

B.5 STANDARDS

Since impurities in nuclear graphite are generally interstitial or lattice substituted atoms, the excitation of impurities can be significantly different from that of standards prepared by addition of metallic salts to spectroscopically pure graphite. Standards used must be synthetic graphite graphitized to at least 2500°C (2773 K) containing in situ impurities. Standard curves must be made using values of impurities determined using wet chemical analysis as required in ASTM C 560.

¹ASTM E2 SM 8 13 Suggested method for spectrochemical analysis of Zirconium by the Silver Chloride Carrier-D-C Arc Technique.

²A suitable electrode is commercially available from the Ultra-Carbon Co., Bay City, Michigan under the designation 715-C. An equivalent electrode may be used.

B.6 PREPARATION OF SAMPLES

A representative sample of 150 mg of the graphite are weighed into 0.5 in. (12.7 mm) diameter polystyrene vials containing a 0.375 in. (9.5 mm) polymethyl methacrylate ball, and 50 mg of recrystallized silver chloride are added to the vial and the vial capped and vibrated 40 s in an amalgamator.¹

B.7 ELECTRODE SYSTEM

Place the lower electrode filled with 60 mg of the sample silver chloride mixture tamped and center vented in the lower water cooled electrode holder (anode) and the counter electrode in the upper clamp. Position the external optics, including the gap, in such a manner that the resulting spectral lines represent radiant energy from the entire arc column; except that both electrode tips should be screened out. These conditions should be satisfied with an analytical gap of about 0.118 in. (3.0 mm) to 0.157 in. (4.0 mm).

B.8 EXCITATION AND EXPOSURE

Produce and record the spectrum in accordance with the following conditions:

1. Electrical Parameters

Voltage, V	250
Current, A	25

2. Exposure Conditions

Spectral Region, Å (nm)	2400 to 3400 (240 to 340)
Slit Width, mm	0.025 to 0.070
Slit Height, mm	1.0 to 7.0
Filter	Three-step spectral filter or equivalent sector wheel, placed at "stigmatic" focal point, transmitting approximately 100%, 20%, and 5%.
Preburn Period, s	None
Exposure Period, s	30

3. Replicate Exposure

Expose samples in duplicate.

¹A dental amalgamator suitable for this purpose is sold under the trade name "Wig-L-Bug." An equivalent amalgamator may be used.

B.9 PHOTOGRAPHIC PROCESSING

Process the emulsion in accordance with ASTM E 115.

B.10 CALIBRATION

B.10.1 Emulsion Calibration. Calibrate emulsions in accordance with ASTM E 116.

B.10.2 Preparation of Analytical Curves. From the data obtained on the standards prepare an analytical curve by plotting, as ordinate, the logarithm of the concentration of the element sought against, as the abscissa the logarithm of the intensity of the spectrum line of the element sought. At least four log intensity ratios shall be averaged for each of at least four points on the analytical curve.

B.11 CALCULATIONS

B.11.1 Frequently, check the analytical curve by exposing a high and a low standard with any series of samples. Compensate, if necessary, for any curve shift by adjusting to the original curve.

B.11.2 Determine the concentration from the average of two determinations of the intensity of the spectrum line of the element sought.

Table 3. Spectral Lines of Quantitative Impurity Elements

Element	Wavelength		Concentration ¹ Range, (ppm)
	Å	nm	
Fe	3020.6	302.06	0.5-25
	3021.1	302.11	1-100
	2999.5	299.95	25-500
	2744.1	274.41	250-1000
V	3183.4	318.34	1-5
	3185.4	318.54	0.5-25
	3184.0	318.40	3-25
	2198.0	219.80	100-250
	2668.0	266.80	25-500
Ti	3088.0	308.80	2-50
B	2497.7	249.77	0.1-2.5
Ca	3179.3	317.93	1-100

¹For higher concentrations dilute sample with high purity graphite.

APPENDIX C

STANDARD METHOD OF TEST FOR BULK DENSITY OF AS-MANUFACTURED CARBON AND GRAPHITE

C.1 SCOPE

This method covers the determination of the density of as-manufactured carbon and graphite from measurements of weight and dimensions in air at room temperature.

C.2 SIGNIFICANCE

C.2.1 This method provides a means of determining bulk density in air on as-manufactured logs.

C.2.2 This method is suitable for manufacturing control and acceptance specifications.

C.3 DEFINITION

Bulk density is the weight in air of a unit volume including both permeable and impermeable voids present in the material at room temperature.

C.4 TEST SPECIMENS

Logs or Blocks. Logs or blocks may be measured and weighed as manufactured, or after rough ends have been squared by machining. The stock shall preferably have been stored in a dry condition under a roof after heat treatment; if there is doubt as to the dryness of the piece, it shall be weighed, dried, and reweighed. Repeat, if necessary, until there is less than 0.1% weight loss. The drying temperature should be 100° to 110° C (273 to 283 K).

C.5 PROCEDURE

Logs or Blocks. Logs or blocks as manufactured, shall be measured on the diameter or cross-section at two different locations, taking care to avoid distortions. Two length measurements shall be taken on cylinders; the second measurement shall be either 90° or 180° from the first. On rectangular shaped pieces, four length measurements shall be made, two each along opposite faces. Additional measurements shall be made, if needed, to determine a valid mean for each dimension. Dimensional and weight measurements shall be made with equipment accurate to 0.5%.

C.6 CALCULATIONS

C.6.1 Calculate the bulk density in accordance with Equation 1:

$$\text{Density} = \frac{W_g}{V_{\text{cm}^3}} = \left(\frac{W_{\text{Mg}}}{V_{\text{m}^3}} \right) \quad (1)$$

where

W_g = weight in grams in air,

V_{cm^3} = volume in cubic centimeters

C.6.2 Calculate to three significant figures.

C.7 REPORT

C.7.1 The report shall include the following.

C.7.1.1 The type, source, grade, geometry and whether the specimen is as-manufactured or machined.

C.7.1.2 Densities of the as-manufactured articles.

C.8 PRECISION

A determination between five plants with two operators performing redundant measurements in each plant yielded a within-plant standard deviation of 0.0052 g/cm³ and a between-plant standard deviation of 0.032 g/cm³, as defined in ASTM E 177.

APPENDIX D

TENSILE PROPERTIES OF NUCLEAR GRAPHITES

D.1 SCOPE

This method of test shall apply to the determination of tensile stress-strain characteristics of nuclear graphite at room temperature. The fracture strength may be measured separately by this technique without measuring and recording the load-elongation curve.

D.2 SUMMARY

The tensile stress-strain characteristics of a cylindrical specimen shall be determined by measuring its response to a continuously increasing applied load. The specimen shall be loaded to failure in uniaxial tension at a constant cross-head speed while recording the applied load. When required, the elongation in a specimen shall be measured and recorded as a function of load by means of a parasitic extensometer. The tensile stress-strain properties shall be determined from the load-elongation behavior of the specimen.

D.3 SIGNIFICANCE AND USE

This test is suitable for product acceptance and for obtaining design, development and research data.

D.4 TERMINOLOGY

The definitions and terms indicated in ASTM E 6 shall be considered as applying to the terms in this method of test. Selected definitions are repeated below.

D.4.1 Tensile Strength. The ratio of the load at fracture to the original cross-sectional area of the specimen.

D.4.2 Strain-at-Fracture. The axial strain when fracture occurs in the gage length.

D.4.3 Modulus of Elasticity.

D.4.3.1 Initial Tangent Modulus. The slope of the stress-strain curve at the origin.

D.4.3.2 Chord Modulus. The slope of the chord drawn between any two specified points on the stress-strain curve.

D.5 APPARATUS

D.5.1 Testing Machine. The machine used for this method of test shall conform to the requirements indicated in ASTM E 4. The testing machine shall have a capacity such that the breaking load of the test specimen shall lie within the maximum capacity range indicated by the manufacturer. In addition, the testing machine shall be used in conjunction with a load cell or other appropriate load measuring device which has been calibrated in a range such that the breaking load of the test specimens falls between 10% and 90% of that range.

D.5.2 Specimen Connectors. Specimen connectors such as shown in Fig. 1 shall be used for connecting the test specimens to the load-train connectors shown in Fig. 2. The figures show sketches and dimensions of the connectors and load-train which have been found to be satisfactory for use with the suggested specimen sizes shown.

D.5.3 Load-Train Connectors. Load-train connectors such as shown in Fig. 2 shall be used to connect the specimen connectors, with the test specimens attached to the load train shown in Fig. 3.

D.5.4 Extensometers.

D.5.4.1 Class A or Class B-1 extensometers, as described in ASTM E 83, shall be used, depending on the degree of precision required. The maximum error of the indicated strain for Class A is 0.00001 and for B-1 is 0.0001.

D.5.4.2 The extensometer shall be calibrated in accordance with the manufacturer's instruction or with instructions in ASTM E 83.

D.6 SAMPLING

Care must be taken to maintain the identification required for the report at the time specimens are removed from the parent log (see D.10.1.1). The extent of the sampling will vary according to the use for which the data is being generated.

D.7 TEST SPECIMENS

D.7.1 The size and configuration of test specimens illustrated in Fig. 4 are recommended. The minimum diameter shall be 3X the maximum particle size. The length shall be a minimum of 3X the diameter.

D.7.2 A centerless grinder is recommended for preparing the specimens. If a lathe is used extreme care should be exercised during machining in order to exert minimal force on the test specimen and provide a smooth, groove-free surface. Axially loads in excess of the compressive yield stress may result in erroneously high fracture strains. Free cutting diamond- or tungsten carbide-tipped tools are recommended.

SI EQUIVALENTS

IN.	0.0002	0.0005	0.001	0.005	0.01	0.0625	0.125	0.375	0.50	0.745	3.25
MM	0.005	0.013	0.025	0.127	0.25	1.59	3.18	9.52	12.7	18.92	82.55

Fig. 1. Specimen Connector

(6061-T6 ALUMINUM RECOMMENDED)

SI EQUIVALENTS

IN.	0.0002	0.005	0.01	1.16	0.125	0.1875	0.250	0.2813	0.310
MM	0.005	0.127	0.25	1.59	3.18	4.76	6.35	7.145	7.87
IN.	0.312	0.625	0.626	0.750	0.752	1.000	1.250	1.375	2.500
MM	7.92	15.88	15.90	19.05	19.10	25.4	31.75	34.925	63.5

Fig. 2. Load-Train Connectors

Fig. 3. Tensile Test Load-Train Set-Up

"A" SPECIMEN

"B" SPECIMEN

SI EQUIVALENTS

IN.	0.001	0.250	0.505	0.75	1.0	1.75	3.00
MM	0.025	6.35	12.83	19.05	25.4	44.45	76.2

Fig. 4. Specimen Size "A" and "B"

D.7.3 Irradiation, oxidation or both may change the original dimensions or shape of a specimen. Care should be taken to use only specimens which meet the requirements of this test procedure.

D.8 PROCEDURE

D.8.1 Preparation of Specimens. Specimen ends shall be taped and retaped with cellophane tape until tape shows no evidence of dust.¹

D.8.2 Measurement of Specimens. The diameter of the test specimens shall be determined at three axial positions, specifically the center and 0.2 in. (5 mm) from each end. Two measurements shall be made at each of these points separated circumferentially by 90°. These measurements shall be accurate to within 0.001 in. (25µm) and averaged to determine the effective sample diameter (see D.10.1.2).

D.8.3 Preparation of Specimen Connector. Abrade each specimen connector end using #3 0.002 in. (50µm) aluminum oxide. Wash in warm water and dry connectors for 15 minutes at 65°C (338 K).

D.8.4 Assembly of Specimens to Specimen Connectors. Degrease specimen connector end² and apply the epoxy³ to the end of each specimen connector. Referring to Fig. 5 or 6, place the specimen in the split sleeve holder between the connector and press firmly together. Install the mating section of the holder and place a retaining ring over each end of the fixture. Follow the manufacturer's instructions for curing the epoxy.

D.8.5 Self Alignment of the Loading Fixture Shall be Determined. A recommended method uses an aluminum or steel specimen of the same dimensions indicated in Fig. 4. Four strain gages are attached to the specimen at 90° increments around the circumference. The specimen is loaded in uniform steps to a load greater than anticipated for the tests to be performed. At each load, each individual strain measurement should not differ from the average of all four by more than 6%.

D.8.6 Test Conditions.

D.8.6.1 Speed of Testing. The cross-head speed shall be approximately 0.01 in./min (0.004 mm/s).

D.8.6.2 Temperature. The test temperature shall be $25 \pm 10^{\circ}\text{C}$.

¹Scotch magic tape or equivalent.

²Sprajon Freon T.F. Number 2002 or equivalent.

³CONAP AD-3 or equivalent.

		SI EQUIVALENTS								
IN.	0.0005	0.001	0.506	0.745	1.0	1.25	1.5	2.375	6.0	
MM	0.013	0.025	12.85	18.92	25.4	44.45	38.1	60.33	152.4	

Fig. 5. Alignment Fixture of "A" Specimens

SI EQUIVALENTS										
IN.	0.0005	0.001	0.251	0.50	0.745	1.0	1.5	2.00	2.25	4.75
MM	0.013	0.025	6.375	12.7	18.92	25.4	38.1	50.8	57.2	120.7

Fig. 6. Alignment Fixture for Size "B" Specimens

D.8.7 Load-Elongation Measurements.

D.8.7.1 Extensometer Attachment. Attach one or more calibrated extensometers to the specimen. It is recommended that a thin coating approximately 0.0002 in. (5 μ m) thick of a quick setting, room temperature-cured cement¹ with a hard smooth surface, be applied to the specimen surfaces in the area of contact with the extensometer probes prior to attaching the extensometer.

The extensometer shall not contact the specimen surface within 0.2 in. (5 mm) of the specimen ends.

D.8.7.2 Simultaneous Measurements. Simultaneous load and elongation measurements shall be made and recorded during a test.

D.9 CALCULATIONS

D.9.1 Engineering Stress. The engineering stress for a given load shall be calculated by dividing the load by the original cross-sectional area of the specimen.

D.9.2 Strength and Strain at Fracture.

D.9.2.1 Tensile Strength. The tensile strength shall be calculated by dividing the load at fracture by the original cross-sectional area of the specimen.

D.9.2.2 Strain at Fracture. The strain at fracture shall be calculated by dividing the elongation at fracture from the stress-strain curve by the gage length.

D.9.2.3 Specimen Gage Length. If any part of the fracture occurs within a distance less than 10 times the epoxy joint thickness from either end of the specimen, the test results shall be recorded but not used to determine the average.

D.9.3 Modulus of Elasticity.

D.9.3.1 Initial Tangent Modulus. The initial tangent modulus for each specimen shall be calculated from the stress-strain curve as the initial tangent, i.e., the slope of the stress-strain curve at the origin (tangent line OS, Fig. 7A).

D.9.3.2 Chord Modulus. The chord modulus for each specimen shall be calculated from the stress-strain curve as the slope of the chord drawn between any two points on the stress-strain curve (chord RQ, Fig. 7B).

¹Eastman 910 adhesive or equivalent.

D.10 REPORT

D.10.1 The report for each specimen tested shall include the following:

D.10.1.1 Identification and manufacturer's grade and log number, parent material size, specimen location and orientation within the parent material.

D.10.1.2 Specimen dimensions, including diameter, length, cross-sectional area.

D.10.1.3 Cross-head speed.

D.10.1.4 Load-elongation curve (omit if only tensile strength measured).

D.10.1.5 Stress-strain curve (optional).

D.10.1.6 Location of fracture (distance from designated ends and location relative to extensometer, if used).

D.10.1.7 Tensile strength.

D.10.1.8 Initial tangent modulus (omit if only tensile strength measured).

D.10.1.9 Chord modulus including the end points (omit if only tensile strength measured).

D.11 PRECISION

Employing the method of this standard, an inter-comparison between two laboratories involving over 100 samples each yielded a within laboratory standard deviation of from 7.7% to 26%, depending on position of the replicate samples within the parent log. Differences in the mean between the two laboratories was 2.7%, statistically insignificant at a confidence level of 99% as determined by F-test on the ratio of the means.

(A) INITIAL TANGENT MODULUS

(B) CHORD MODULUS BETWEEN
ANY TWO STRESSES Q AND R

Fig. 7. Stress-Strain Diagram Showing Straight Lines Corresponding to (A) Initial Tangent Modulus and (B) Chord Modulus