



Designation: C791 – 19

# Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Boron Carbide<sup>1</sup>

This standard is issued under the fixed designation C791; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover procedures for the chemical, mass spectrometric, and spectrochemical analysis of nuclear-grade boron carbide powder and pellets to determine compliance with specifications.

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

1.3 The analytical procedures appear in the following order:

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Total Carbon by Combustion in an Inductive Furnace and Infrared Measurement	8 – 17
Total Boron by Titrimetry and ICP OES	18 – 28
Isotopic Composition by Mass Spectrometry	29 – 33
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Chloride and Fluoride by Ion-Selective Electrode	51 – 59
Water by Constant-Voltage Coulometry and Weight Loss on Drying	60 – 63
Metallic Impurities by DCArc OES and wet chemical methods	64 and 65
Soluble Boron by Titrimetry and ICP OES	66 – 80
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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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.03 on Neutron Absorber Materials Specifications.

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## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

- C750 Specification for Nuclear-Grade Boron Carbide Powder
- C751 Specification for Nuclear-Grade Boron Carbide Pellets
- C859 Terminology Relating to Nuclear Materials
- C1128 Guide for Preparation of Working Reference Materials for Use in Analysis of Nuclear Fuel Cycle Materials
- D1193 Specification for Reagent Water
- E969 Specification for Glass Volumetric (Transfer) Pipets

## 3. Terminology

3.1 *Definitions*—For definitions of terms relating to nuclear materials, refer to Terminology C859.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *analyte*—the constituent determined by a chemical measurement process.

3.2.2 *analytical or emission line*—the particular wavelength of electromagnetic radiation used in determining the presence or concentration of an element.

3.2.3 *background*—spectral intensity that would be measured at the wavelength of the emission line if the emission and overlapping lines were not present.

3.2.4 *calibration*—the act, process, or result of establishing the relationship between the response of an instrument and the amount of analyte present.

3.2.5 *calibration function*—the graphical or mathematical representation of the relationship between the response of an instrument and the concentration or mass of the analyte.

3.2.6 *calibration samples or solutions*—samples or solutions with known analyte contents or analyte concentrations, respectively, to establish the relationship between the response of an instrument and the amount of analyte.

<sup>2</sup> 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.

3.2.7 *certified reference material (CRM)*—a reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

3.2.8 *DCArc OES*—optical emission spectrometry (OES) with direct current arc (DCArc) as excitation source.

3.2.9 *drift correction*—the process of adjusting for a shift of an instrument calibration.

3.2.10 *ICP MS*—mass spectrometry (MS) with inductively coupled plasma (ICP) as ionization source.

3.2.11 *ICP OES*—optical emission spectrometry (OES) with inductively coupled plasma (ICP) as excitation source.

3.2.12 *interlaboratory study (ILS)*—a study undertaken to demonstrate the precision and bias of a test method.

3.2.13 *internal standard*—a material present in or added to test samples that serves as an intensity reference for spectral measurements.

3.2.14 *matrix*—all components of a material except the analyte.

3.2.15 *matrix matching*—adjustment of the composition of calibration samples or solutions to match the composition of the matrix.

3.2.16 *method*—instructions used to produce a numerical result, which are detailed in a document referred to as “the method.”

3.2.17 *nebulizer*—a device for converting a sample solution into a gas-liquid aerosol for ICP OES.

3.2.18 *optical emission spectrometry (OES)*—pertaining to emission spectrometry in the ultraviolet, visible, or infrared wavelength regions of the electromagnetic spectrum.

3.2.19 *repeatability,  $S_r$* —the standard deviation of results collected using the same method on the same material in the same laboratory on different days.

3.2.20 *reproducibility,  $S_R$* —the standard deviation of results obtained on the same material using the same method in different laboratories.

3.2.21 *sample*—a portion of a material selected and processed to render its composition representative of the composition of the whole.

3.2.22 *sensitivity*—the change of instrument response with change in analyte concentration.

3.2.23 *standardization*—the experimental establishment of the concentration of a reagent solution.

3.2.24 *TIMS*—thermal ionization mass spectrometry.

#### 4. Significance and Use

4.1 Boron carbide is used as a control material in nuclear reactors. In order to be suitable for this purpose, the material must meet certain criteria for assay, isotopic composition, and impurity content. These methods are designed to show whether

or not a given material meets the specifications for these items as described in Specifications C750 and C751.

4.1.1 An assay is performed to determine whether the material has the specified boron and carbon content.

4.1.2 Determination of the isotopic content of the boron and the free carbon content is made to establish whether the content is in compliance with the purchaser’s specifications.

4.1.3 Impurity content is determined to ensure that the maximum concentration limit of certain impurities (chloride, fluoride, water, metallic impurities, soluble boron) is not exceeded.

#### 5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>3</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193.

#### 6. Safety Precautions

6.1 Many laboratories have established safety regulations governing the use of hazardous chemicals and equipment. The users of these methods should be familiar with such safety practices.

#### 7. Sampling

7.1 Criteria for sampling this material are given in Specifications C750 and C751.

### TOTAL CARBON BY COMBUSTION IN AN INDUCTIVE FURNACE AND INFRARED MEASUREMENT

#### 8. Scope

8.1 This method covers the determination of total carbon in nuclear-grade boron carbide in either powder or pellet form.

#### 9. Summary of Test Method

9.1 The sample and added combustion accelerators (mostly tungsten-and iron-granules) are heated in an inductive furnace under oxygen atmosphere. The high-frequency field of the furnace couples with electrically conductive components of sample and combustion accelerators. The sample is heated to temperatures not lower than 1400°C and the total carbon content of the sample is released as carbon dioxide and, partially, as carbon monoxide. The reaction gas is passed through a gas-treatment train to ensure that any carbon

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

monoxide formed is converted to carbon dioxide and to remove dust and moisture. The reaction gas is then transferred to the infrared absorption cell of the analyzer. The molecular absorption of carbon dioxide is measured by using a narrow-band optical filter which is translucent for the wavelength of the characteristic infrared absorption of carbon dioxide. The mass fraction of carbon dioxide in the reaction gas is proportional to area of the transient absorption signal. The mass fraction of carbon in the sample is calculated by using a calibration function established by suitable calibration standards measured under comparable conditions.

## 10. Interferences

10.1 At the specification limits usually established for nuclear-grade boron carbide, interferences are insignificant.

## 11. Apparatus

11.1 Commonly used laboratory equipment and special equipment according to the following:

11.1.1 *Carbon analyzer*, with induction furnace and infrared absorption cell, suitable to correctly determine the mass fraction of carbon within the concentration range given by boron carbide.

NOTE 1—The correctness of the analysis result can be proved by using matrix analogous reference materials or by comparing with an independent alternative test method.

11.1.2 *Analytical balance*, capable of reading to the nearest 0.01 mg.

11.1.3 *Ceramic crucible*, for example, mullite or alumina.

11.1.4 *Crucible lid with hole*, for example, mullite or alumina.

## 12. Reagents

12.1 Reagents of known analytical grade shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

12.1.1 *Tungsten granules*.

NOTE 2—Depending on the particle size of the material the decomposition of the sample in the furnace may be improved by partially replacing tungsten granules by tin granules. Tungsten/tin-mixtures are commercially available.

12.1.2 *Iron granules*.

12.1.3 *Calibration and control samples*, with defined carbon content, preferably certified reference materials with composition and carbon content similar to the analyzed material. Also suitable are primary substances preferably carbonates (for example, calcium carbonate,  $\text{CaCO}_3$ ). When certified reference materials are not available, consider Guide C1128 for preparation of working reference materials.

12.1.4 *Oxygen*, purity  $\geq 99.998\%$  v/v.

12.1.5 *Pneumatic gas*, for example, nitrogen, purity  $\geq 99.9\%$  v/v.

## 13. Sampling and Sample Preparation

13.1 Sampling has to be performed in a way that the sample to be analyzed is representative for the total amount of material. In an unknown drying state the sample has to be dried

at  $(110 \pm 5)^\circ\text{C}$  to constant weight. The sample is cooled down to ambient temperature in a desiccator and stored therein.

NOTE 3—Drying for 2 h is normally sufficient.

13.2 The sample material must have a particle size of  $\leq 150\ \mu\text{m}$  (No. 100 sieve). Inhomogeneous sample material has to be homogenized. Standard apparatus and procedures for crushing, milling and homogenization may be used provided that no contamination occurs which lessens the accuracy of the determination.

## 14. Calibration

14.1 The calibration has to be performed daily according to the manufacturer's instructions. It has to be ensured that the mass of carbon in the calibration sample (12.1.3) and test sample are within the same order of magnitude.

NOTE 4—This is achieved by choosing a suitable calibration substance and adapted weights.

14.2 The calibration has to be done according to Section 15.

## 15. Procedure

15.1 *Preparation of Analysis*—Ceramic crucibles (11.1.3) and crucible lids (11.1.4) have to be cleaned prior to use by firing in a muffle furnace at  $1200^\circ\text{C}$  for 1 h. After that, they have to be stored in a desiccator.

15.2 *Determination of Blank Value (Method Blank)*—The same procedure according to 15.4 has to be applied, however without addition of boron carbide. At least three blanks should be determined at least once in each 8-h shift in which total carbon analyses are made.

15.3 *Conditioning of Carbon Analyzer*—Prior to making the initial analysis, condition the carbon analyzer by performing at least two analysis runs. The same procedure according to section 15.4 has to be applied, however only adding a small amount of boron carbide (that is, a spatula-tip of boron carbide).

15.4 *Determination of Carbon Content*—A sub-sample of 20 to 30 mg of boron carbide powder prepared in accordance to Section 12 is weighed to the nearest 0.01 mg into the ceramic crucibles (11.1.3) cleaned according to section 15.1.

NOTE 5—Using modern carbon analysis devices with an automated calculation of the mass fraction of carbon in the sub sample the sample mass has to be entered using the internal or external keyboard. Using carbon analysis devices which measure the absolute mass of carbon of the sample the sample mass has to be noted for later calculations.

The sample in the crucible is covered with approximately 0.9 g and 1.8 g of iron and tungsten granules (12.1.1 and 12.1.2), respectively. For mixing, the crucible has to be shaken carefully. Afterwards, the crucible is closed with the lid (11.1.4), placed into the induction furnace and the combustion cycle is started. Using modern carbon analysis devices the carbon content is calculated and displayed on the internal or external screen or printed out.

To ensure correctness of results and to detect measurement drift a control sample (12.1.3) shall be measured within and at the end of the measurement series.