



Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Boron Carbide¹

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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 analytical procedures appear in the following order:

	Sections
Total Carbon by Combustion and Gravimetry	7-17
Total Boron by Titrimetry	18-28
Isotopic Composition by Mass Spectrometry	29-38
Chloride and Fluoride Separation by Pyrohydrolysis	39-45
Chloride by Constant-Current Coulometry	46-54
Fluoride by Ion-Selective Electrode	55-63
Water by Constant-Voltage Coulometry	64-72
Impurities by Spectrochemical Analysis	73-81
Soluble Boron by Titrimetry	82-95
Soluble Carbon by a Manometric Measurement	96-105
Metallic Impurities by a Direct Reader Spectrometric Method	106-114

2. Referenced Documents

2.1 ASTM Standards:

C 750 Specification for Nuclear-Grade Boron Carbide Powder²

C 751 Specification for Nuclear-Grade Boron Carbide Pellets²

D 1193 Specification for Reagent Water³

E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis⁴

E 116 Practice for Photographic Photometry in Spectrochemical Analysis⁴

E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes⁴

3. Significance and Use

3.1 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 C 750 and C 751.

3.1.1 An assay is performed to determine whether the material has the specified boron content.

3.1.2 Determination of the isotopic content of the boron is made to establish whether the content is in compliance with the purchaser's specifications.

3.1.3 Impurity content is determined to ensure that the maximum concentration limit of certain impurity elements is not exceeded.

4. Reagents

4.1 *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.⁵ 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193.

5. Safety Precautions

5.1 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.

6. Sampling

6.1 Criteria for sampling this material are given in Specifications C 750 and C 751.

TOTAL CARBON BY COMBUSTION AND GRAVIMETRY

7. Scope

7.1 This method covers the determination of total carbon in

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² *Annual Book of ASTM Standards*, Vol 12.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

⁵ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N.Y., and the "United States Pharmacopeia."