Designation: C809 − 19

Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Aluminum Oxide and Aluminum Oxide-Boron Carbide Composite Pellets[1]

This standard is issued under the fixed designation C809; 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 These test methods cover procedures for the chemical, mass spectrometric, and spectrochemical analysis of nuclear-grade aluminum oxide and aluminum oxide-boron carbide composite pellets to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

	Sections
Boron by Titrimetry and ICP OES	8 to 17
Separation of Boron for Mass Spectrometry	18 to 23
Isotopic Composition by Mass Spectrometry	24 to 27
Separation of Halides by Pyrohydrolysis	28 to 31
Chloride and Fluoride by Ion-Selective Electrode	32 to 34
Chloride, Bromide, and Iodide by Amperometric Microtitrimetry	35 to 37
Trace Elements by Emission Spectroscopy	38 to 50
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1.3 The values stated in SI units are to be regarded as the standard.

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.* (For specific precautionary statements, see Section 6.)

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C784 Specification for Nuclear-Grade Aluminum Oxide-Boron Carbide Composite Pellets
C785 Specification for Nuclear-Grade Aluminum Oxide Pellets
C791 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Boron Carbide
C799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions
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
E115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis (Withdrawn 2002)[3]
E116 Practice for Photographic Photometry in Spectrochemical Analysis (Withdrawn 2002)[3]

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard:*

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

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

3.2.3 *calibration samples or solutions (standards)*—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.

3.2.4 *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

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[1] 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] 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] The last approved version of this historical standard is referenced on www.astm.org.

property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

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

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

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

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

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

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

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

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

3.2.13 *TIMS*—thermal ionization mass spectrometry.

3.2.14 *titrimetry*—a method of quantitatie chemical analysis that is used to determine the concentration of an identified analyte.

4. Significance and Use

4.1 Aluminum oxide pellets are used in a reactor core as filler or spacers within fuel, burnable poison, or control rods. In order to be suitable for this purpose, the material must meet certain criteria for impurity content. These test methods are designed to show whether or not a given material meets the specifications for these items as described in Specification C785.

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

4.2 Aluminum oxide-boron carbide composite pellets are used in a reactor core as a component in neutron absorber rods. In order to be suitable for this purpose, the material must meet certain criteria for boron content, isotopic composition, and impurity content as described in Specification C784.

4.2.1 The material is assayed for boron to determine whether the boron content is as specified by the purchaser.

4.2.2 Determination of the isotopic content of the boron is made to establish whether the ^{10}B concentration is in compliance with the purchaser's specifications.

4.2.3 Impurity content is determined to ensure that the maximum concentration limit of certain impurity elements 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.[4] 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, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type III.

6. Safety Precautions

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

7. Sampling

7.1 Criteria for sampling aluminum oxide pellets are given in Specification C785.

7.2 Criteria for sampling aluminum oxide-boron carbide composite pellets are given in Specification C784.

BORON BY TITRIMETRY AND ICP OES

8. Scope

8.1 For the determination of boron in aluminum oxide-boron carbide composites by titrimetry and ICP OES the procedures detailed in Test Method C791, Sections 18 – 27 shall be used.

9. Summary of Test Method

9.1 Powdered aluminum oxide-boron carbide composite is mixed with alkaline reagents and this mixture is fused to decompose the aluminum oxide-boron carbide. The melt is dissolved in diluted hydrochloric acid and heated or purged with nitrogen to remove carbon dioxide. The boron as boric acid is titrated with standardized sodium hydroxide solution, using the mannitoboric acid procedure. Alternatively, the boron in the samples solution is measured using ICP OES.

NOTE 1—Sodium carbonate or a mixture of sodium carbonate and potassium carbonate (1:1) is normally used as alkaline reagent to decompose the aluminum oxide-boron carbide composite.

10. Interferences

10.1 *Titrimetry*—Because metallic impurities in high concentrations may distort the inflection points of the titration aluminum should be precipitated from the sample solution using barium carbonate. No distortion was found for concentrations of Fe < 2 %, Ti < 1 %. Interferences by dissolved CO_2 shall be removed by heating the sample solution or by purging the sample solution with nitrogen.

[4] *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.

10.2 *ICP OES*—Interference effects depend primarily upon the resolving power of the spectrometer and the selection of the analytical lines. In practice, line interferences (spectral interferences) and non spectral interferences are critical. Non spectral interferences are caused primarily by different chemical composition of calibration solution and sample solution, resulting in an alteration of nebulization and excitation properties. Also memory-effects can play a role. The best way to minimize non spectral interferences is the use of calibration samples with the same composition of matrix, ideally certified reference materials. When certified reference materials are not available, consider Guide C1128 for preparation of working reference materials.

11. Apparatus

11.1 See Test Methods C791, Section 21.

12. Reagents

12.1 See Test Methods C791, Section 22.

13. Precautions

13.1 See Test Methods C791, Section 23.

14. Sampling and Sample Preparation

14.1 See Test Methods C791, Section 24.

15. Procedure

15.1 See Test Methods C791, Section 25.

16. Titrimetric Determination of Boron

16.1 See Test Methods C791, Section 26.

17. ICP OES

17.1 See Test Methods C791, Section 27.

SEPARATION OF BORON FOR MASS SPECTROMETRY

18. Scope

18.1 This test method covers the separation of boron from aluminum and other impurities. The isotopic composition of the separated boron is measured using mass spectrometry. The test method for isotopic composition is found in this standard.

18.2 Alternatively, the separation of boron can be performed by pyrohydrolysis. A stream of moist oxygen is passed over the powdered sample at 1100°C. The pyrohydrolytic reaction releases boric acid and boron oxide from the sample, which volatilize and collect in the condensate (see Test Methods C791, Section 41). The condensate is used for measurement of isotopic composition by ICP-MS (see Test Methods C791, Section 33).

19. Summary of Test Method

19.1 Boron is put into solution using a sealed-tube dissolution method. It is separated from aluminum and other impurities by solvent extraction and ion exchange.

20. Interferences

20.1 There are no known interferences not eliminated by this separation test method.

21. Apparatus

21.1 *Analytical Balance,* capable of weighing to ±0.1 mg.

21.2 *Mortar,* diamond (Plattner) (or equivalent).

21.3 *Sieve,* No. 100 (150-µm) U.S. Standard Sieve Series, 76-mm diameter, brass or stainless steel.

21.4 *Glass Boats,* borosilicate, 4-mm wide, 3-mm deep, 40-mm long.

21.5 *Glass Tubing,* heavy-wall borosilicate, 5-mm inside diameter by 250-mm long, sealed at one end.

21.6 *Glass Blower's Torch.*

21.7 *Iron Pipe,* 12.7 by 254-mm long with threaded end caps.

21.8 *Muffle Furnace,* capable of operation at 300°C. The heated area must be of sufficient size to hold the capped iron pipe.

21.9 *Separatory Funnel,* 60-mL with TFE-fluorocarbon stopcock.

21.10 *Mixer,* vortex type.

21.11 *Filter Paper,* ashless, slow filtering for fine precipitates.

21.12 *Ion Exchange Column,* borosilicate glass, 5-mm inside diameter, 100-mm long with a TFE-fluorocarbon stopcock.

21.13 *Beaker,* 50-mL, quartz or TFE-fluorocarbon.

22. Reagents

22.1 *Nitric Acid* (sp gr 1.42)—Concentrated Nitric Acid (HNO_3).

22.2 *Cation Exchange Resin,*[5] 80 to 100 mesh. Prepare the resin by treatment with 3 *N* HCl followed by water wash until the effluent is neutral to pH paper.

22.3 *Chloroform* ($CHCl_3$).

22.4 *2-Ethyl-1,3Hexanediol Solution,* 5 volume % in chloroform.

22.5 *Nitric Acid* (HNO_3), 2 *M*.

22.6 *Sodium carbonate* (Na_2CO_3), powder.

22.7 *Sodium Hydroxide* (NaOH) *Solution,* 0.1 *N*, carbonate-free. Store in a plastic bottle.

23. Procedure

23.1 Crush the aluminum oxide/boron carbide composite pellet using a diamond mortar until all the sample is passed through a No. 100 (150-µm) screen.

23.2 Weigh a 250-mg sample into a glass boat.

[5] Dowex 50 × 8 (or equivalent).