



Standard Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Aluminum Oxide and Aluminum Oxide-Boron Carbide Composite Pellets¹

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	7 to 16
Separation of Boron for Mass Spectrometry	17 to 22
Isotopic Composition by Mass Spectrometry	23 to 26
Separation of Halides by Pyrohydrolysis	27 to 30
Chloride and Fluoride by Ion-Selective Electrode	31 to 33
Chloride, Bromide, and Iodide by Amperometric Microtitrimetry	34 to 36
Trace Elements by Emission Spectroscopy	37 to 49
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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 and health practices and determine the applicability of regulatory limitations prior to use.* (For specific precautionary statements, see Section 5.)

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](#)

[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[E115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis \(Withdrawn 2002\)³](#)

[E116 Practice for Photographic Photometry in Spectrochemical Analysis \(Withdrawn 2002\)³](#)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. Significance and Use

3.1 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](#).

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

3.2 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](#).

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

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

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

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.

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.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.

4.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193, Type III.

5. Safety Precautions

5.1 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.

6. Sampling

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

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

BORON BY TITRIMETRY AND ICP OES

7. Scope

7.1 For the determination of boron in aluminum oxide-boron carbide composites by titrimetry and ICP OES the procedures detailed in Test Method C791, Sections 17–26 shall be used.

8. Summary of Test Method

8.1 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.

9. Interferences

9.1 *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 concen-

trations of Fe < 2 %, Ti < 1 %. Interferences by dissolved CO₂ shall be removed by heating the sample solution or by purging the sample solution with nitrogen.

9.2 *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.

10. Apparatus

10.1 See Test Methods C791, Section 20.

11. Reagents

11.1 See Test Methods C791, Section 21.

12. Precautions

12.1 See Test Methods C791, Section 22.

13. Sampling and Sample Preparation

13.1 See Test Methods C791, Section 23.

14. Procedure

14.1 See Test Methods C791, Section 24.

15. Titrimetric Determination of Boron

15.1 See Test Methods C791, Section 25.

16. ICP OES

16.1 See Test Methods C791, Section 26.

SEPARATION OF BORON FOR MASS SPECTROMETRY

17. Scope

17.1 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.

NOTE 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 40). The condensate is used for measurement of isotopic composition by ICP-MS (see Test Methods C791, Section 32).

18. Summary of Test Method

18.1 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.

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.

19. Interferences

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

20. Apparatus

- 20.1 *Analytical Balance*, capable of weighing to ± 0.1 mg.
- 20.2 *Mortar*, diamond (Plattner) (or equivalent).
- 20.3 *Sieve*, No. 100 (150- μ m) U.S. Standard Sieve Series, 76-mm diameter, brass or stainless steel.
- 20.4 *Glass Boats*, borosilicate, 4-mm wide, 3-mm deep, 40-mm long.
- 20.5 *Glass Tubing*, heavy-wall borosilicate, 5-mm inside diameter by 250-mm long, sealed at one end.
- 20.6 *Glass Blower's Torch*.
- 20.7 *Iron Pipe*, 12.7 by 254-mm long with threaded end caps.
- 20.8 *Muffle Furnace*, capable of operation at 300°C. The heated area must be of sufficient size to hold the capped iron pipe.
- 20.9 *Separatory Funnel*, 60-mL with TFE-fluorocarbon stopcock.
- 20.10 *Mixer*, vortex type.
- 20.11 *Filter Paper*, ashless, slow filtering for fine precipitates.
- 20.12 *Ion Exchange Column*, borosilicate glass, 5-mm inside diameter, 100-mm long with a TFE-fluorocarbon stopcock.
- 20.13 *Beaker*, 50-mL, quartz or TFE-fluorocarbon.

21. Reagents

- 21.1 *Nitric Acid* (sp gr 1.42)—Concentrated Nitric Acid (HNO_3).
- 21.2 *Cation Exchange Resin*,⁵80 to 100 mesh. Prepare the resin by treatment with 3 N HCl followed by water wash until the effluent is neutral to pH paper.
- 21.3 *Chloroform* (CHCl_3).
- 21.4 *2-Ethyl-1,3Hexanediol Solution*, 5 volume % in chloroform.
- 21.5 *Nitric Acid* (HNO_3), 2 M.
- 21.6 *Sodium carbonate* (Na_2CO_3), powder.
- 21.7 *Sodium Hydroxide* (NaOH) *Solution*, 0.1 N, carbonate-free. Store in a plastic bottle.

22. Procedure

- 22.1 Crush the aluminum oxide/boron carbide composite pellet using a diamond mortar until all the sample is passed through a No. 100 (150- μ m) screen.
- 22.2 Weigh a 250-mg sample into a glass boat.

22.3 Introduce the boat and sample into a heavy-wall glass tube, being very careful to prevent any of the sample from adhering to the wall of the tube near the open end.

22.4 Introduce 0.5 mL of concentrated HNO_3 into the glass tube.

22.5 Mix the sample and acid using the vortex mixer.

22.6 Flame the glass tube to remove the moisture from the walls.

22.7 Seal the glass tube. There are two methods available:

22.7.1 Sealing the glass tube may be accomplished by constriction, then drawing off a short piece of the tube, then working down the sealed end.

22.7.2 A seal can be made by allowing the open end of the tube to flow together by heating and revolving the tube slowly. While the tube is red with heat, the tube is warmed enough to blow out the seal to a rounded shape.

22.8 Place the glass tube into a safety container which consists of a 12.7-mm inside diameter black iron pipe with screw caps on each end. The caps can be tightened with finger tip control.

22.9 Insert the assembly into a 300°C muffle furnace with the top end of the assembly elevated and heat for 6 h.

22.10 Remove the assembly from the muffle furnace and place into a tray, keeping the same end of the assembly elevated.

22.11 Allow the assembly to cool to room temperature.

22.12 Withdraw the glass tube from the safety container and file a notch about 13 mm from one end of the tube.

NOTE 3—Contents of the tube may be under pressure.

22.13 Heat a glass rod to red heat, then place the rod on the notch. This action should crack the glass tube; however, a light tap may be needed to complete the break.

22.14 Pipet 4 mL of water into the glass tube and mix using a vortex mixer.

22.15 Filter the solution through filter paper (15.3). Catch the filtrate in a 60-mL separatory funnel.

22.16 Wash the paper with 15-mL of 2 M HNO_3 . Catch the wash in the separatory funnel.

22.17 Add 10 mL of 5 % 2-ethyl-1,3 hexanediol solution to the separatory funnel and shake for 2 min.

22.18 Drain the organic (lower) layer into a clean 100-mL beaker.

22.19 Repeat 22.17 and 22.18.

22.20 Transfer the 2-ethyl-1,3 hexanediol solution to a clean 60-mL separatory funnel.

22.21 Extract the boron by shaking for 2-min with a NaOH solution containing the amount of sodium calculated to give a B/Na ratio of two and a volume sufficient to give 1 mg B/mL.

22.22 Discard the organic phase.

22.23 Wash the aqueous phase with two 5-mL portions of CHCl_3 . Discard the organic wash.

⁵ Dowex 50 $\times$ 8 (or equivalent).

22.24 Transfer the aqueous phase containing the boron to a 50-mL quartz or TFE-fluorocarbon beaker.

22.25 Evaporate the solution to a volume of about 1 mL.

22.26 Add 0.5 mL of ion exchange resin to the beaker and swirl.

NOTE 4—Addition of the resin to the beaker prevents formation of CO₂ bubbles on the resin column in the subsequent step.

22.27 Transfer the slurry to a prepared resin column containing a 10-mm depth of resin.

22.28 Elute the boron with an amount of water calculated to yield a solution containing 1 mg B/mL.

22.29 Add solid Na₂CO₃ to give a Na/B ratio of 0.9 to 1.0. Transfer the solution to a plastic bottle. The solution is used for measurement of isotopic composition by mass spectrometry (see 23-26).

ISOTOPIC COMPOSITION BY MASS SPECTROMETRY

23. Scope

23.1 This test method covers the determination of the isotopic composition of boron in nuclear-grade aluminum oxide/boron carbide composite pellets containing natural to highly enriched boron.

24. Summary of Test Method

24.1 Boron isotopic ratios are measured in aluminum oxide/boron carbide composites by thermal ionization mass spectrometry (TIMS) following chemical separation of boron (see 17-22). The loaded filament is transferred to the mass spectrometer where boron isotopic ratios are measured using the Na₂BO₂⁺ ion. When mixing the boron and sodium carbonate, a Na to B ratio of 1:1 is maintained, which gives a stable ion emission within a few minutes after operational vacuum is attained.

25. Interferences

25.1 Impurity elements, at the specification limits usually established for nuclear-grade composites, do not interfere. Strontium is a potential interference and it is an impurity element in the tantalum filament material. At the temperature used to ionize sodium borate, however, the strontium impurity in the filament does not volatilize to cause a high bias at mass 88.

NOTE 5—This potential interference does not occur in ICP-MS.

26. Procedure

26.1 Continue with the determination of the isotopic composition by TIMS in accordance with Section 31 of Test Methods C791.

NOTE 6—Alternatively, the isotopic composition of boron in aluminum oxide-boron carbide composites can be measured by ICP-MS following chemical separation of boron by pyrohydrolysis in accordance with Sections 32 and 40 of Test Method C791.

SEPARATION OF HALIDES BY PYROHYDROLYSIS

27. Scope

27.1 This method covers the separation of up to 100 µg of chloride and fluoride per gram of sample. The separated halides are measured using other methods found in this standard.

28. Summary of Test Method

28.1 A stream of moist argon is passed over a mixture of powdered sample and U₃O₈ or sodium tungstate with tungsten trioxide accelerator heated at 1000 to 1100°C. Alternatively, a stream of moist oxygen is passed over the powdered sample at 1100°C, which requires no accelerators. The pyrohydrolytic reaction releases chloride and fluoride as hydrochloric and hydrofluoric acids as well as boric acid and boron oxide, which volatilize and collect in the condensate.

29. Interferences

29.1 Interferences are not expected. The conditions given in this test method for pyrohydrolysis must be controlled to ensure complete recovery of the halides.

30. Procedure

30.1 Continue with pyrohydrolysis in accordance with Sections 33 to 39 of Test Methods C791.

CHLORIDE AND FLUORIDE BY ION-SELECTIVE ELECTRODE

31. Scope

31.1 This method covers the measurement of chloride and fluoride after separation from aluminum oxide-boron carbide composites by pyrohydrolysis. The limit of detection for chloride and fluoride in aluminum oxide-boron carbide composites is 3 mg/kg and 2 mg/kg, respectively.

32. Summary of Test Method

32.1 The condensate from the pyrohydrolysis step is analyzed for chloride and fluoride with ion-selective electrodes.

33. Procedure

33.1 Continue with the determination of chloride and fluoride in accordance with Sections 50 to 58 of Test Methods C791.

CHLORIDE, BROMIDE, AND IODIDE BY AMPEROMETRIC MICROTITRIMETRY

34. Scope

34.1 This test method covers the determination of halogens, except fluorine, as separated by pyrohydrolysis. The detection limit is 1.5 µg Cl/g sample.

35. Summary of Test Method

35.1 An aliquot of the distillate equivalent to about 2.0 g of sample and adjusted to pH 8 is concentrated and titrated by constant current coulometric generation of silver ions to an amperometric end point. Standards are run to determine the

calibration factor in $\mu\text{g Cl}^-/\text{s}$, and the total halides present in the sample, excluding fluorides, are determined and reported as chloride.

36. Procedure

36.1 Continue with the determination of chloride, bromide, and iodide in accordance with the relevant sections of Test Methods C799.

TRACE ELEMENTS BY EMISSION SPECTROSCOPY

37. Scope

37.1 This spectrochemical test method provides for the determination of 14 impurity elements. The elements and concentration ranges are as follows:

Elements	Concentration Range, ppm
Boron	10 to 10 000
Calcium	10 to 10 000
Chromium	10 to 10 000
Dysprosium	30 to 10 000
Europium	10 to 10 000
Gadolinium	30 to 10 000
Hafnium	30 to 10 000
Iron	10 to 10 000
Magnesium	10 to 10 000
Nickel	10 to 10 000
Samarium	10 to 10 000
Silicon	10 to 10 000
Sodium	10 to 10 000
Titanium	10 to 10 000

37.2 The test method can also be extended to cover the determination of other elements of interest.

38. Summary of Test Method

38.1 The sample is pulverized using a tungsten-carbide mortar and a weighed portion mixed with a weighed amount of graphite powder. An aliquant of the mixture is transferred to an under-cut graphite electrode and burned to completion in a d-c arc. The spectra are recorded on photographic emulsion and the impurity concentrated determined by visual comparison with standard spectra arced and photographed in the same manner as the sample.

NOTE 7—For measurement of metallic impurities spectrographs and recording of emission spectra on photographic plates is still in use. However, this apparatus and procedure does not represent state of the art technology and methodology. In Section 63 of Test Method C791, a state of the art DCarc-OES procedure for measurement of metallic impurities in boron carbide is described where a modern DCarc excitation source is coupled to a modern simultaneous optical emission spectrometer. The light emitted from the excitation source is guided into the spectrometer by coupling via fiber-optics or directly. Using fiber-optics the DCarc can be coupled to most off-the-shelf ICP OES spectrometers which significantly improves the versatility of these spectrometers. The procedure described in Section 63 of Test Method C791 is also applicable for measurement of metallic impurities in aluminum oxide-boron carbide composites provided that calibration is performed using matrix-analogous calibration standards. Wavelength and concentration range (see Table 1 of Test Method C791) and precision and bias data (see Section 63.12 of Test Method C791) were obtained for boron carbide. For aluminum oxide-boron carbide composites different data may be obtained.

Besides the DCarc OES method also wet chemical methods can be applied for the measurement of metallic impurities in aluminum oxide-boron carbide composites. The sample is decomposed by melt-fusion with sodium carbonate or sodium/potassium carbonate and the concentration of

metallic impurities in the resulting sample solutions is measured using ICP OES, ICP-MS or AAS. The wet chemical method described in Sections 64.1 to 64.4 and 64.6 of Test Method C791 can also be applied to aluminum oxide-boron carbide composites.

39. Apparatus

39.1 *Comparator*, capable of projecting standards and sample spectra for visual reading.

39.2 *Excitation Source*, a continuous d-c arc source capable of providing 16 A.

39.3 *Mixer Mill*,⁶ mechanical.

39.4 *Mortar and Pestle* made of materials other than elements to be determined. Tungsten carbide is recommended.

39.5 *Photographic Processing Equipment*, providing for a complete photographic processing operation.

39.6 *Spectrograph*,⁷ having a reciprocal linear dispersion of at least 51 Å/min in the first order.

40. Reagents and Materials

40.1 *Aluminum Oxide*, high-purity.

40.2 *Boron Carbide*, high-purity.

40.3 *Graphite Electrodes*, crater (anode) piece, made from 6.4 by 38-mm stock with a 5-mm wide and a 2-mm deep crater. Upper (counter) electrode, made from 3.2 by 102-mm stock, pointed at one end.

40.4 *Graphite Powder*, spectrographically pure, 200 mesh.

40.5 *Oxides*, high-purity, of the elements to be determined.

40.6 *Photographic Emulsions*, SA No. 1 and 1-N plates or film.

40.7 *Photographic Processing Solutions*.

40.8 *Vials*, plastic.

41. Preparation of $\text{Al}_2\text{O}_3/\text{B}_4\text{C}$ Base Material

41.1 Weigh the amount of Al_2O_3 and B_4C (Note 8) that corresponds with the samples to be analyzed. Pregrinding of the Al_2O_3 and B_4C in a tungsten carbide mortar may be necessary to obtain a homogeneous mixture.

NOTE 8—The Al_2O_3 and B_4C used for the base material should be similar in physical characteristics to that in the samples.

41.2 Transfer to a plastic vial and mix thoroughly using the mechanical mixer.

42. Preparation of Standards

42.1 Weigh the calculated amount of each compound required to give 100 ng of each element to be determined and transfer to a mortar. Grind to a fine homogeneous powder.

42.2 Make the desired dilutions by grinding the calculated amount of impurity mix with either aluminum oxide or aluminum oxide/boron carbide base material. This can be done in the mortar or in plastic vials with the aid of a mechanical

⁶ A satisfactory mill is manufactured and sold by Spex SamplePrep, LLC, Metuchen, NJ.

⁷ For example, Jarrell-Ash, 21-ft Wadsworth mount.

mixer. Standards with a gradation of 10, 30, 70, 100, 1000, and 10 000 ppm should cover the impurity levels to be determined.

42.3 Follow 43.1 for mixing standards and graphite powder.

43. Sample Preparation

43.1 Weigh 0.1 g of the pulverized sample and 0.15 g of graphite powder and transfer to a plastic vial in which a plastic ball has been placed. Grind the sample and graphite in the mechanical mixer.

44. Electrode Charging

44.1 Weigh 0.025 g of each sample (in duplicate) and transfer to a crater electrode.

44.2 Weigh 0.025 g of each standard and transfer to a crater electrode (single aliquant).

44.3 Tamp each electrode lightly with a graphite rod, and heat the charged electrodes at 120°C for 30 min before arcing.

45. Excitation and Exposure

45.1 *Excitation Parameters:*

Current 16 A

Discharge d-c arc

Analytical gap 5 mm

Slit width 20 µm

45.2 *Exposure:*

Time 120 s

Diaphragm 2/3

Spectral region 2000 to 7700 Å

46. Photographic Processing

46.1 Process the emulsion in accordance with Practices E115 and E116.

47. Photometry

47.1 Determine the impurity concentration by visual comparison using the analytical lines listed below:

Element	Analytical Line, Å
Boron	2497.7
Calcium	3968.4
Chromium	2835.6
Dysprosium	3385.0
Europium	2727.9, 2813.8
Gadolinium	3350.5, 3362.2
Hafnium	2820.2, 2916.5
Iron	2483.2
Magnesium	2795.5, 2802.7
Nickel	3002.4, 3003.6
Samarium	3307.0, 3306.4
Silicon	2516.1
Sodium	5889.0
Titanium	3349.4

48. Microphotometry

48.1 If a more precise measurement of impurity elements is needed, process in accordance with appropriate sections of E2 SM 10-14, Suggested Method for Spectrochemical Analysis of Alumina Ceramic Materials by the Powder-D-C Arc Technique.⁸

49. Precision and Bias

49.1 The relative standard deviation is 15 % or less for microdensitometric measurements and a factor of 2 (–50 % to + 100 %) for visual comparison measurements.

50. Keywords

50.1 alumina/boron carbide pellets; alumina pellets; Al₂O₃; Al₂O₃/B₄C; analytical methods

⁸ *Methods for Emission Spectrochemical Analysis*, ASTM, 6th ed. 1971.

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