



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

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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	7 to 13
Separation of Boron for Mass Spectrometry	14 to 19
Isotopic Composition by Mass Spectrometry	20 to 23
Separation of Halides by Pyrohydrolysis	24 to 27
Fluoride by Ion-Selective Electrode	28 to 30
Chloride, Bromide, and Iodide by Amperometric Microtitrimetry	31 to 33
Trace Elements by Emission Spectroscopy	34 to 46

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:

C 784 Specification for Nuclear-Grade Aluminum Oxide-Boron Carbide Composite Pellets²

C 785 Specification for Nuclear-Grade Aluminum Oxide Pellets²

C 791 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Boron Carbide²

C 799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions²

D 1193 Specification for Reagent Water³

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 Absorbers Materials Specifications.

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

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

E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis⁴

E 116 Practice for Photographic Photometry in Spectrochemical Analysis⁴

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 C 785.

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 C 784.

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.

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

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

⁵ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

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 D 1193, 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 C 785.

6.2 Criteria for sampling aluminum oxide-boron carbide composite pellets are given in Specification C 784.

BORON BY TITRIMETRY

7. Scope

7.1 This test method covers the determination of boron in aluminum oxide-boron carbide composites. As an alternative, the procedure for total boron by titrimetry detailed in Test Methods C 791 may be used.

8. Summary of Test Method

8.1 The sample is crushed, passed through a 100-mesh screen, weighed in a glass boat, and introduced into a heavy-wall glass tube. Nitric acid is added to the tube and the contents mixed using a vortex mixer. The tube is sealed, placed into a safety container, heated for 6 h, cooled to room temperature, opened, and the contents washed into a beaker.⁶ The solution is adjusted to pH 9.0 and filtered, then adjusted to pH 3.5 and boiled to remove CO₂. Substantially, a pure boric acid is obtained which can be titrated in the presence of mannitol with a standard solution of sodium hydroxide.^{7,8}

9. Apparatus

- 9.1 *Analytical Balance*, capable of weighing to ± 0.1 mg.
- 9.2 *Mortar*, diamond (Plattner) (or equivalent).
- 9.3 *Sieve*, No. 100 (150- μ m) U.S. Standard Sieve Series, 76-mm diameter, brass or stainless steel.
- 9.4 *Glass Boats*, borosilicate, 4-mm wide, 3-mm deep, 40-mm long.
- 9.5 *Glass Tubing*, heavy-wall borosilicate, 5-mm inside diameter by 250-mm long, sealed at one end.
- 9.6 *Mixer*, vortex type.
- 9.7 *Glass Blower's Torch*.
- 9.8 *Iron Pipe*, 12.7 by 254-mm long with threaded end caps.

⁶ Wichers, E., Schlecht, W. G., and Gordon, C. L., "Preparing Refractory Oxides, Silicates, and Ceramic Materials for Analysis by Heating with Acids in Sealed Tubes at Elevated Temperatures," *Journal of Research of the National Bureau of Standards*, Vol 33, 1944, p. 451.

⁷ Lerner, M. W., *The Analysis of Elemental Boron*, New Brunswick Laboratory, U. S. Atomic Energy Commission, TID-25190, November 1970.

⁸ Rodden, C. J., *Analysis of Essential Nuclear Reactor Materials*, U.S. Atomic Energy Commission, Washington, DC, Government Printing Office, 1964.

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

9.10 *pH Meter*, with pH electrodes and magnetic stirrer.

9.11 *Steam Bath*.

9.12 *Hot Plate*.

9.13 *Filter Paper*, 11 cm, ashless slow filtering for fine precipitates.

9.14 *Buret*, Class A, 25-mL.

10. Reagents

10.1 *Boric Acid*, NIST SRM 951 or its replacement.

10.2 *Hydrochloric Acid (HCl)*, 1 N.

10.3 *Hydrochloric Acid (HCl)*, 0.1 N.

10.4 *Mannitol*.

10.5 *Nitric Acid* (sp gr 1.42)—Concentrated Nitric Acid (HNO₃).

10.6 *Sodium Hydroxide (NaOH) Solution*, 1 N, carbonate-free.

10.7 *Sodium Hydroxide (NaOH) Solution*, 0.1 N, carbonate-free.

10.8 *Sodium Hydroxide (NaOH) Solution*, 0.025 N, carbonate-free, standardized against NIST SRM 951.

11. Procedure

11.1 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.

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

11.3 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.

11.4 Introduce 0.5 mL of concentrated HNO₃ into the glass tube.

11.5 Mix the sample and acid using the vortex mixer.

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

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

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

11.7.2 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.

11.8 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.

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

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

11.11 Allow the assembly to cool to room temperature.

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

NOTE 1—Contents of the tube may be under pressure.

11.13 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.

11.14 Wash the contents from the glass tube into a 250-mL beaker; however, if the aluminum oxide is stuck to the walls of the tube, shake on a vortex mixer.

NOTE 2—The matrix Al_2O_3 does not completely dissolve, but all of the boron is in solution.

11.15 Precipitate the iron and the aluminum by using 1 *N* sodium hydroxide solution to adjust the pH to 9.0.

11.16 Place the beaker on a steam bath and digest for 1 h.

11.17 Filter the sample through the filter paper (9.13) and wash the precipitate with several portions of hot deionized water.

11.18 Adjust the pH between 3.5 and 4.0 using 1 *N* HCl.

11.19 Cover the solution with a flat watch glass, then place the beaker on a hot plate and boil for about 5 min to remove carbon dioxide.

11.20 Remove the sample from the hot plate and cool to room temperature in a water bath.

11.21 Adjust the pH of the sample to 5.6 to 5.7 using 0.1 *N* NaOH solution and 0.1 *N* HCl. Add 1 to 3 g of mannitol.

11.22 Titrate the sample to pH 8.0 using a 0.025 *N* NaOH solution.

11.23 Determine a blank by performing 11.3-11.22 without the sample.

12. Calculation

12.1 Calculate the percent boron in the sample as follows:

$$B, \% = \frac{(V-B)(N)(A)(100)}{W} \quad (1)$$

where:

V = millilitres of NaOH solution used in titration of the sample,

B = millilitres of NaOH solution used in titration of the blank,

N = normality of the NaOH solution,

A = atomic weight of boron computed for the sample based upon the measured isotopic composition, and

W = milligrams of sample weight.

13. Precision

13.1 The limit of error at the 95 % confidence level for a single determination is ± 0.10 % absolute.

SEPARATION OF BORON FOR MASS SPECTROMETRY

14. Scope

14.1 This test method covers the separation of boron from aluminum and other impurities. The isotopic composition of the separated boron is measured using another test method found herein.

15. Summary of Test Method

15.1 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.

16. Interferences

16.1 There are no known interferences not eliminated by

this separation test method.

17. Apparatus

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

17.2 *Mixer*, vortex type.

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

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

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

18. Reagents

18.1 *Cation Exchange Resin*,⁹ 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.

18.2 *Chloroform* (CHCl_3).

18.3 *2-Ethyl-1,3Hexanediol Solution*, 5 volume % in chloroform.

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

18.5 *Sodium carbonate* (Na_2CO_3), powder.

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

19. Procedure

19.1 Prepare an aliquot of sample by following 11.1-11.13.

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

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

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

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

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

19.7 Repeat 19.5 and 19.6.

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

19.9 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.

19.10 Discard the organic phase.

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

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

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

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

NOTE 3—Addition of the resin to the beaker prevents formation of CO_2 bubbles on the resin column in the subsequent step.

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

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

⁹ Dowex 50 $\times$ 8 (or equivalent).

19.17 Add solid Na_2CO_3 to give a Na/B ratio of 0.9 to 1.0. Transfer the solution to a plastic bottle.

ISOTOPIC COMPOSITION BY MASS SPECTROMETRY

20. Scope

20.1 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.

21. Summary of Test Method

21.1 Boron isotopic ratios are measured in aluminum oxide/boron carbide composites by thermal ionization mass spectrometry following chemical separation of boron. The loaded filament is transferred to the mass spectrometer where boron isotopic ratios are measured using the Na_2BO_2^+ 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.

22. Interferences

22.1 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.

23. Procedure

23.1 Continue with the determination of the isotopic composition in accordance with the relevant sections of Test Methods C 791.

SEPARATION OF HALIDES BY PYROHYDROLYSIS

24. Scope

24.1 This test method covers the separation of up to 100 μg of halides per gram of sample. The separated halides are measured using other test methods found herein.

25. Summary of Test Method

25.1 A stream of moist gas is passed over a mixture of powdered sample and U_3O_8 accelerator heated at 1000 to 1100°C. The pyrohydrolytic reaction releases the halides as their respective acids, which volatilize and collect in the condensate.

26. Interferences

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

27. Procedure

27.1 Continue with pyrohydrolysis in accordance with the relevant sections of Test Methods C 791.

FLUORIDE BY ION-SELECTIVE ELECTRODE

28. Scope

28.1 This test method can determine as low as 2- μg of F/g sample in condensate containing all the halogens.

29. Summary of Test Method

29.1 An aliquot of the condensate representing 1 g of sample is measured by ion-selective electrode and compared to a standard curve prepared by spiking equivalent-size aliquots taken from a blank distillation.

30. Procedure

30.1 Continue with the determination of fluoride in accordance with the relevant sections of Test Methods C 799.

CHLORIDE, BROMIDE, AND IODIDE BY AMPEROMETRIC MICROTITRIMETRY

31. Scope

31.1 This test method covers the determination of halogens, except fluorine, as separated by pyrohydrolysis. The detection limit is 1.5 $\mu\text{g Cl/g}$ sample.

32. Summary of Test Method

32.1 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.

33. Procedure

33.1 Continue with the determination of chloride, bromide, and iodide in accordance with the relevant sections of Test Methods C 799.

TRACE ELEMENTS BY EMISSION SPECTROSCOPY

34. Scope

34.1 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

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

35. Summary of Test Method

35.1 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.

36. Apparatus

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

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

36.3 *Mixer Mill*,¹⁰ mechanical.

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

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

36.6 *Spectrograph*,¹¹ having a reciprocal linear dispersion of at least 51 Å/min in the first order.

37. Reagents and Materials

37.1 *Aluminum Oxide*, high-purity.

37.2 *Boron Carbide*, high-purity.

37.3 *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.

37.4 *Graphite Powder*,¹² spectrographically pure, 200 mesh.

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

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

37.7 *Photographic Processing Solutions*.

37.8 *Vials*, plastic.

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

38.1 Weigh the amount of Al_2O_3 and B_4C (Note 4) 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 4—The Al_2O_3 and B_4C used for the base material should be similar in physical characteristics to that in the samples.

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

39. Preparation of Standards

39.1 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.

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

39.3 Follow 40.1 for mixing standards and graphite powder.

40. Sample Preparation

40.1 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.

41. Electrode Charging

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

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

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

42. Excitation and Exposure

42.1 Excitation Parameters:

Current 16 A
Discharge d-c arc
Analytical gap 5 mm
Slit width 20 μm

42.2 Exposure:

Time 120 s
Diaphragm 2/3
Spectral region 2000 to 7700 Å

43. Photographic Processing

43.1 Process the emulsion in accordance with Practices E 115 and E 116.

¹⁰ A satisfactory mill is manufactured and sold by Spex Industries Inc., Scotch Plains, NJ.

¹¹ Jarrell-Ash, 21-ft Wadsworth mount.

¹² Graphite powder, available from Union Carbide Corp., Carbon Products Div., 270 Park Ave., New York, NY 10017, has been found satisfactory for this purpose.

44. Photometry

44.1 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

45. Microphotometry

45.1 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.¹³

46. Precision and Bias

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

47. Keywords

47.1 alumina/boron carbide pellets; alumina pellets; Al_2O_3 ; $\text{Al}_2\text{O}_3/\text{B}_4\text{C}$; analytical methods

¹³ *Methods for Emission Spectrochemical Analysis*, ASTM, 6th ed. 1971.

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