~~Designation:C 809–94~~ Designation: C809 – 94 (Reapproved 2007)

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	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:*[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

E116 Practice for Photographic Photometry in Spectrochemical Analysis

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~~C785.

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

~~3.2Aluminum~~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

[1] These test methods are under the jurisdiction of ASTM Committee ~~C-26~~ C26 on Nuclear Fuel Cycle and are the direct responsibility of Subcommittee C26.03 ~~on Neutron Absorbers Materials Specifications~~.

~~Current edition approved Oct. 15, 1994. Published December 1994. Originally published as C 809–80. Last previous edition C 809–80.~~on Neutron Absorber Materials Specifications.

Current edition approved July 1, 2007. Published August 2007. Originally approved in 1980. Last previous edition approved in 2000 as C809 – 94 (2000). DOI: 10.1520/C0809-94R07.

[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* ~~, Vol 12.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

composition, and impurity content as described in Specification ~~C 784~~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 ^{10}B 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.[3] 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 ~~D 1193~~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 ~~C 785~~C785.

6.2 Criteria for sampling aluminum oxide-boron carbide composite pellets are given in Specification ~~C 784~~C784.

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~~C791 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.[4] The solution is adjusted to pH 9.0 and filtered, then adjusted to pH 3.5 and boiled to remove CO_2. Substantially, a pure boric acid is obtained which can be titrated in the presence of mannitol with a standard solution of sodium hydroxide.[5,6]

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-μm) 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.

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.

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[3] ~~*Annual Book of ASTM Standards*, Vol 11.01.~~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.

[4] ~~Annual Book of ASTM Standards, Vol 03.05.~~

[4] 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.

[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.~~

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

[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.~~

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

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 (HNO_3).
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-µm) 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 HNO_3 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,*[7] 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.

[7] Lerner, M. W., *The Analysis of Elemental Boron,* New Brunswick Laboratory, U. S. Atomic Energy Commission, TID-25190, November 1970.
[7] Dowex 50 × 8 (or equivalent).