Designation: C 889 – 99

Standard Test Methods for Chemical and Mass Spectrographic Analysis of Nuclear-Grade Gadolinium Oxide (Gd_2O_3) Powder[1]

This standard is issued under the fixed designation C 889; 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 and mass spectrographic analysis of nuclear-grade gadolinium oxide powders to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

	Sections
Carbon by Direct Combustion—Thermal Conductivity	2
C 1408 Test Method for Carbon (Total) in Uranium Oxide Powders and Pel lets By Direct Combustion-Infrared Detection Method	3
Total Chlorine and Fluorine by Pyrohydrolysis Ion—Selective Electrode	7-13
Loss of Weight on Ignition	14-20
Sulfur by Combustion—Iodometric Titration	2
Impurity Elements by a Spark-Source Mass Spectrographic	2
C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical,Nuclear, and Radiochemical Analysis of Uranium Hexafluoride	3
C 1287 Test Method for Determination of Impurities In Uranium Dioxide By Inductively Coupled Plasma Mass Spectrometry	3
Gadolinium Content in Gadolinium Oxide by Impurity Correction	21-24

1.3 *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 hazard statements, see Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*

C 696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets[3]

C 761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical,Nuclear, and Radiochemical Analysis of Uranium Hexafluoride[3]

C 888 Specification for Nuclear-Grade Gadolinium Oxide (Gd_2O_3) Powder[3]

C 1287 Test Method for Determination of Impurities In Uranium Dioxide By Inductively Coupled Plasma Mass Spectrometry[3]

D 1193 Specification for Reagent Water[4]

E 115 Practice for Photographic Processing in Optical Emission Spectrographic Analysis[5]

E 116 Practice for Photographic Photometry in Spectrochemical Analysis[5]

E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes[5]

C 1408 Test Method for Carbon (Total) in Uranium Oxide Powders and Pel lets By Direct Combustion-Infrared Detection Method[3]

3. Significance and Use

3.1 Gadolinium oxide powder is used, with subsequent processing, in nuclear fuel applications, such as an addition to uranium dioxide. These test methods are designed to determine whether the material meets the requirements described in Specification C 888.

3.1.1 The material is analyzed to determine whether it contains the minimum gadolinium oxide content specified.

3.1.2 The loss on ignition and impurity content are determined to ensure that the weight loss and the maximum concentration limit of specified impurity elements are 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.[6] 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.

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[1] These test methods are under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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[2] Discontined January 1999. See C 889–90.

[3] *Annual Book of ASTM Standards,* Vol 12.01.

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

[5] *Annual Book of ASTM Standards*, Vol 03.05.

[6] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. 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, NY, and the "United States Pharmacopeia."

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

5. Hazards

5.1 Proper precautions should be taken to prevent inhalation or ingestion of gadolinium oxide powders or dust during grinding or handling operations.

5.2 Workers should observe precautions as specified in vendor supplied Material Safety Data Sheets (MSDS).

6. Sampling

6.1 Criteria for sampling this material are given in Specification C 888.

CARBON BY DIRECT COMBUSTION—THERMAL CONDUCTIVITY

This Test Method was discontinued in January 1999 and replaced by C 1408

TOTAL CHLORINE AND FLUORINE BY PYROHYDROLYSIS ION—SELECTIVE ELECTRODE

7. Scope

7.1 This test method covers the determination of chlorine and fluorine in nuclear-grade gadolinium oxide (Gd_2O_3) powder. With a 1 to 10-g sample of Gd_2O_3, concentrations of 5 to 200 µg of chlorine and 1 to 200 µg of fluorine per gram of Gd_2O_3 are determined without interference.

8. Summary of Test Method

8.1 The halogens are separated from powdered gadolinium oxide by pyrohydrolysis in a quartz tube with a stream of wet oxygen at a temperature of 900 to 1000°C **(1, 2, 3, 4).**[7] Chlorine and fluorine are volatilized simultaneously as acids, absorbed in a buffer solution as chloride and fluoride, and measured with ion-selective electrodes **(4, 5, 6).**

9. Apparatus

9.1 *Pyrohydrolysis Equipment*—A suitable assembly of apparatus as shown in Fig. 1.

9.1.1 *Gas Flow Regulator and Flowmeter*.

9.1.2 *Hot Plate*, used to warm the water saturating the sparge gas to 50 to 80°C.

9.1.3 *Combustion Tube Furnace*, having a bore of about 32 mm (1¼in.), a length of about 305 mm (12 in.), and the capability of maintaining a temperature of 1000°C.

9.1.4 *Quartz Reaction Tube* (Fig. 2)—The exit end should not extend over 51 mm (2 in.) beyond the furnace with a ground joint connecting to the delivery tube. The delivery tube extends into a polyethylene absorption vessel with a tip capable of giving a stream of fine bubbles.

9.1.5 *Combustion Boat*—A ceramic, platinum, or quartz boat with a 10-mL capacity, 89 to 102 mm (3½ to 4 in.) long, 12.7 mm (½ in.) wide, and 9.53 mm (⅜ in.) high.

9.1.6 *Absorption Vessel*—A 50-mL polyethylene graduate or tube is satisfactory.

9.2 *Ion-Selective Electrodes*—A chloride-ion-selective activity electrode;[8] fluoride-ion-selective activity electrode.[9]

9.3 *pH Meter and Double-Junction Reference Electrode*, such as a mercuric sulfate, sleeve junction type. The meter should have an expandable scale with a sensitivity of 1 mV.

9.4 *Magnetic Stirrer*.

9.5 *Beakers*, 50-mL, polyethylene.

10. Reagents

10.1 *Accelerator, U_3O_8 (Halogen-free)*, can be used, but a flux of sodium tungstate (Na_2WO_4) with tungsten trioxide (WO_3) may be used to advantage **(1).** (See Method C 696.) Special preparation of the mixture is necessary, that is, dehydrate 165 g of Na_2WO_4 in a large platinum dish. Transfer the dried material to a mortar, add 116 g of WO_3, and grind the mixture to ensure good mixing. Transfer the mixture into a platinum dish and heat with a burner for 2 h. Cool the melt, transfer the flux to a mortar, and grind to a coarse powder. Store the flux in an airtight bottle. Mix about 8 g of flux with each portion of sample to be pyrohydrolyzed.

[7] The boldface numbers in parentheses refer to the list of references appended to this standard.

[8] The Orion Model No. 96–17 has been found satisfactory.

[9] The Orion Model No. 9409 has been found satisfactory.

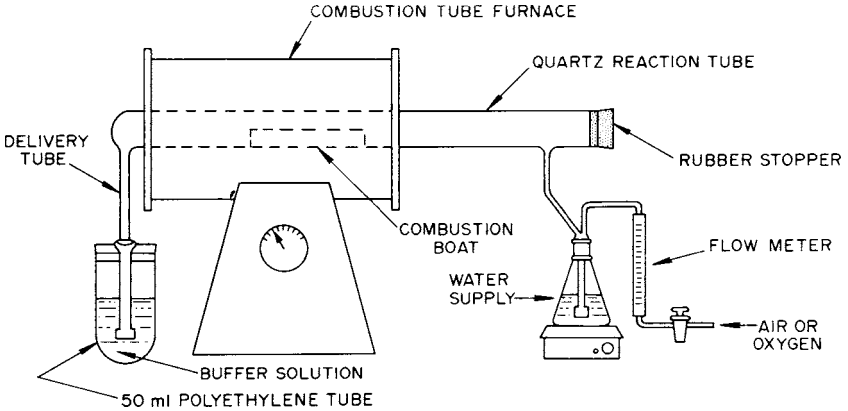

FIG. 1 Pyrohydrolysis of Gadolinium Oxide