



Standard Test Methods for Chemical and Mass Spectrometric Analysis of Nuclear- Grade Gadolinium Oxide (Gd₂O₃) Powder¹

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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover procedures for the chemical and mass spectrometric 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 Pellets By Direct Combustion-Infrared Detection Method	3
Total Chlorine and Fluorine by Pyrohydrolysis Ion—Selective Electrode	-
Loss of Weight on Ignition	7-13
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	14-17
C 1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide	4

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:³

C 761 Test Methods for Chemical, Mass Spectrometric,

Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C 888 Specification for Nuclear-Grade Gadolinium Oxide (Gd₂O₃) Powder

C 1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry

C 1408 Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method

C 1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide

D 1193 Specification for Reagent Water

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.⁵ 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 “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.”

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

Current edition approved Jan. 1, 2006. Published February 2006. Originally approved in 1978. Last previous edition approved in 1999 as C 889 – 99.

² Discontinued January 1999. See C 889–90.

³ 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.

⁴ Discontinued March 2005. See C 889–90.

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

This Test Method was discontinued in March 2005 and replaced by **C 1408**

LOSS OF WEIGHT ON IGNITION

7. Scope

7.1 This test method covers the loss-on-ignition of volatile constituents from nuclear-grade gadolinium oxide (Gd_2O_3) powder.

8. Summary of Test Method

8.1 A weighed sample of gadolinium oxide is heated to a minimum of 900°C for 2 h in air. Upon cooling, the sample is reweighed. The loss in weight is the difference between the initial and final weight.

9. Apparatus

9.1 *Combustion Equipment*—A suitable muffle furnace capable of heating to 1000°C.

9.2 *Crucible*, ceramic, nickel, or platinum with a 10-g capacity.

9.3 *Desiccator*.

9.4 *Balance*.

10. Reagent

10.1 *Drying Desiccant*.

11. Procedure

11.1 Heat the furnace to $925 \pm 25^\circ C$.

11.2 Weigh 5 g of Gd_2O_3 to the nearest 1 mg.

11.3 Insert the crucible into the furnace.

11.4 Heat for 2 h.

11.5 Cool the Gd_2O_3 sample in a desiccator to room temperature.

11.6 Remove the crucible from the desiccator and reweigh.

12. Calculation

12.1 Calculate the percent weight loss as follows:

$$\text{Percent weight loss} = (A - B)(100) / W \quad (1)$$

where:

A = weight of crucible plus Gd_2O_3 before heating, g,

B = weight of crucible plus Gd_2O_3 after heating, g, and

W = sample mass, g of Gd_2O_3 .

13. Precision and Bias

13.1 The relative standard deviation for measuring loss of volatile constituents on ignition is 1%.

13.2 Since there is no accepted reference material suitable for determining bias for the procedure in this test method, no statement on bias is being made.

SULFUR BY COMBUSTION—IODOMETRIC TITRATION

This Test Method was discontinued in January 1999.

IMPURITY ELEMENTS BY A SPARK-SOURCE MASS SPECTROGRAPHIC METHOD

ICP-AES as described in **C 761** or Inductively Coupled Plasma Mass Spectrometry (ICP-MS) described in **C 1287** may be used to determine boron, cadmium, thorium, dysprosium, europium, samarium, terbium, and ytterbium as impurity elements and other impurities. Powder must be analyzed as-received (not ignited), and reported in μg oxide per g gadolinium oxide to perform impurity calculations in sections **14-17**

GADOLINIUM CONTENT IN GADOLINIUM OXIDE BY IMPURITY CORRECTION METHOD

14. Scope

14.1 The percent gadolinium oxide content of powders, exclusive of volatiles, is determined by calculation after the material has been analyzed to determine the total impurities.

15. Summary of Test Method

15.1 The total gadolinium content of gadolinium oxide powders is determined by calculation after the sample has been analyzed by the following procedures:

15.1.1 Loss of Weight on Ignition, Sections **7-13**.

15.1.2 Impurity Elements by ICP-MS or ICP-AES, elements as specified in **C 888**

16. Calculation

16.1 If the concentration of an impurity element is a “less-than” (<), (that is, a concentration expressed as being less than the lower detection limit of the analytical method), this less-than value shall be used as the concentration of that element in the following calculations.

16.2 Calculate the percent gadolinium oxide as follows:

$$G = 100 - (I_G)(10^{-4}) + (I_L)(100) \quad (2)$$

where:

G = percent gadolinium oxide

I_S = total μg spectrographic impurities as oxides, per gram gadolinium oxide powder sample,
 I_{LI} = gram loss-on-ignition impurity per gram gadolinium oxide powder sample,

impurities analyzed. Therefore, no statement on precision and bias is being made.

17. Precision and Bias

17.1 Since this is a calculation, precision and bias will depend upon relative standard deviation of the individual

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).