



Designation: ~~C889-06~~ Designation: C889 – 11

# Standard Test Methods for Chemical and Mass Spectrometric Analysis of Nuclear-Grade Gadolinium Oxide (Gd<sub>2</sub>O<sub>3</sub>) Powder<sup>1</sup>

This standard is issued under the fixed designation C889; 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 ( $\epsilon$ ) 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        |
| C1408 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  | 4        |
| C1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide                       | 5        |
| Loss of Weight on Ignition   | 7-13     |
| Sulfur by Combustion—Iodometric Titration  | 2        |
| Impurity Elements by a Spark-Source Mass Spectrographic  | 2        |
| C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride | 3        |
| C1287 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    |

C1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide

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1.3

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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 hazard statements, see Section 5.

## 2. Referenced Documents

2.1 ASTM Standards:<sup>3</sup>

C761 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride

C888 Specification for Nuclear-Grade Gadolinium Oxide (Gd<sub>2</sub>O<sub>3</sub>) Powder

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

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<sup>2</sup> Discontinued January 1999. See C889-90.

<sup>3</sup> 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.

<sup>4</sup> Discontinued March 2005. See C1408.

<sup>5</sup> Discontinued March 2005. See C889-90.

- C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry  
 C1408 Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method  
 C1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide  
 D1193 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 C888.

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.<sup>6</sup> 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, references to water shall be understood to mean reagent water as defined in Specification D1193.

### 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 C888.

#### CARBON BY DIRECT COMBUSTION— THERMAL CONDUCTIVITY

This Test Method was discontinued in January 1999 and replaced by C1408

#### TOTAL CHLORINE AND FLUORINE BY PYROHYDROLYSIS ION—SELECTIVE ELECTRODE

This Test Method was discontinued in March 2005 and replaced by ~~C1408~~C1502

#### 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<sub>2</sub>O<sub>3</sub>) 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*.

<sup>6</sup> *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.