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Standard Test Methods for Analysis of Sintered Gadolinium Oxide-Uranium Dioxide Pellets[1]

This standard is issued under the fixed designation C968; 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 analysis of sintered gadolinium oxide-uranium dioxide pellets to determine compliance with specifications.

1.2 The analytical procedures appear in the following order:

	Section
Carbon (Total) by Direct Combustion—Thermal Conductivity Method	2
C1408 Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method	3
Chlorine and Fluorine by Pyrohydrolysis Ion-Selective Electrode Method	4
C1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide	3
Gadolinia Content by Energy-Dispersive X-Ray Spectrometry	4
C1456 Test Method for Determination of Uranium or Gadolinium, or Both, in Gadolinium Oxide-Uranium Oxide Pellets or by X-Ray Fluorescence (XRF)	3
Hydrogen by Inert Gas Fusion	4
C1457 Test Method for Determination of Total Hydrogen Content of Uranium Oxide Powders and Pellets by Carrier Gas Extraction	3
Isotopic Uranium Composition by Multiple-Filament Surface-Ionization Mass Spectrometric Method	2
C1413 Test Method for Isotopic Analysis of Hydrolysed Uranium Hexafluoride And Uranyl Nitrate Solutions By Thermal Ionization Mass Spectrometry	3
C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis	3
Nitrogen by Distillation—Nessler Reagent (Photometric) Method	6 to 16
Oxygen-to-Metal Ratio of Sintered Gadolinium Oxide-Uranium Dioxide Pellets	4
C1430 Test Method for Determination of Uranium, Oxygen to Uranium, and Oxygen to Metal (O/M) in Sintered Uranium Dioxide and Gadolinia-Uranium Dioxide Pellets by Atmospheric Equilibration	3
Spectrochemical Determination of Trace Impurity Elements	4
C1517 Test Method for Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy	3
Total Gas by Hot Vacuum Extraction	2
Ceramographic Determination of Free Gd_2O_3 and Free UO_2 to Estimate the Homogeneity of $(U,Gd)O_2$ Pellets	17 to 24
Ceramographic Determination of Average Grain Size by Linear Intercept after Chemical Etching	25 to 32

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.*

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

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[2] Discontinued 1999. See C968 – 94.

[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 2005. See C968 – 99.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets
C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis
C1408 Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method
C1413 Test Method for Isotopic Analysis of Hydrolyzed Uranium Hexafluoride and Uranyl Nitrate Solutions by Thermal Ionization Mass Spectrometry
C1430 Test Method for Determination of Uranium, Oxygen to Uranium (O/U), and Oxygen to Metal (O/M) in Sintered Uranium Dioxide and Gadolinia-Uranium Dioxide Pellets by Atmospheric Equilibration
C1456 Test Method for Determination of Uranium or Gadolinium (or both) in Gadolinium Oxide-Uranium Oxide Pellets or by X-Ray Fluorescence (XRF)
C1457 Test Method for Determination of Total Hydrogen Content of Uranium Oxide Powders and Pellets by Carrier Gas Extraction
C1502 Test Method for Determination of Total Chlorine and Fluorine in Uranium Dioxide and Gadolinium Oxide
C1517 Test Method for Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy
D1193 Specification for Reagent Water
E112 Test Methods for Determining Average Grain Size
E146 Methods of Chemical Analysis of Zirconium and Zirconium Alloys (Silicon, Hydrogen, and Copper)[5]

3. Significance and Use

3.1 The test methods in this method are designed to show whether a given material is in accordance with Specification C922.

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 Reagent 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type IV of Specification D1193.

5. Safety Precautions

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

CARBON (TOTAL) BY DIRECT COMBUSTION—THERMAL CONDUCTIVITY METHOD
This Test Method was discontinued in January 1999 and replaced by Test Method C1408

CHLORINE AND FLUORINE BY PYROHYDROLYSIS ION-SELECTIVE ELECTRODE METHOD
This Test Method was discontinued in March 2005 and replaced by Test Method C1502

GADOLINIA CONTENT BY ENERGY-DISPERSIVE X-RAY SPECTROMETRY
This Test Method was discontinued in March 2005 and replaced by Test Method C1456

HYDROGEN BY INERT GAS FUSION
This Test Method was discontinued in March 2005 and replaced by Test Method C1457

ISOTOPIC URANIUM COMPOSITION BY MULTIPLE-FILAMENT SURFACE-IONIZATION MASS SPECTROMETRIC METHOD
This Test Method was discontinued in January 1999 and replaced with C1413
Samples can be dissolved using the appropriate dissolution techniques described in Practice C1347

NITROGEN BY DISTILLATION—NESSLER REAGENT (PHOTOMETRIC) METHOD

6. Scope

6.1 This test method describes the determination of nitrogen in gadolinium oxide-uranium dioxide pellets (Gd_2O_3/UO_2). With

[5] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.
[6] *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.

a 2 to 5-g sample, concentrations from 5 to 100 μg of nitrogen are determined without interference.

7. Summary of Test Method

7.1 Pellet samples of gadolinium oxide-uranium dioxide are crushed, then dissolved in phosphoric acid. Hydrochloric acid with hydrogen peroxide can also be used. The resulting solution is made alkaline with sodium hydroxide, and the nitrogen is separated as ammonia by steam distillation (see Method E146). Nessler reagent is added to the distillate to form the yellow ammonium complex, and the absorbance of the solution is measured at approximately 430 nm, using a cell depth of 2 cm **(1, 2)**.[7]

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

NOTE 1—This procedure has been written for a cell having a 2-cm light path. The range of the method can be extended by suitably varying sample mass, aliquot size, amounts of reagents, and cell depth.

8. Interferences

8.1 There are no known interfering elements.

9. Apparatus

9.1 *Nitrogen Distillation Apparatus*, with 100-mL flask, Fig. 1; micro-Kjeldahl apparatus.

9.2 *Photometer*—A filter photometer with a narrow-band filter; or a spectrophotometer equipped with 2-cm cells.

9.3 *Heater*, 750-W, electric, full-control.

10. Reagents and Materials

10.1 *Nessler Reagent*—Dissolve 50 g of potassium iodide (KI) in a minimum of cold water (approximately 35 mL). Add a saturated solution of mercuric chloride ($HgCl_2$) slowly until the first slight precipitate of red mercuric iodide persists. Add 400 mL of potassium or sodium hydroxide solution (505 g of KOH or 360 g of NaOH/L). Dilute the solution to 1 L with ammonia-free water, mix, and allow the solution to stand overnight. Decant the supernatant liquid and store it in a brown bottle. This reagent is stable indefinitely.

10.2 *Ammonium Chloride* (NH_4 Cl)—Dry the ammonium chloride at 110 to 120°C for 2 h.

10.3 *Nitrogen Reference Solution* (1 mL = 10 µg N)—Dissolve 3.819 g of dried NH_4Cl in water and dilute the solution to 1 L. Transfer 10 mL of this solution to a 1-L volumetric flask and dilute it to volume with water.

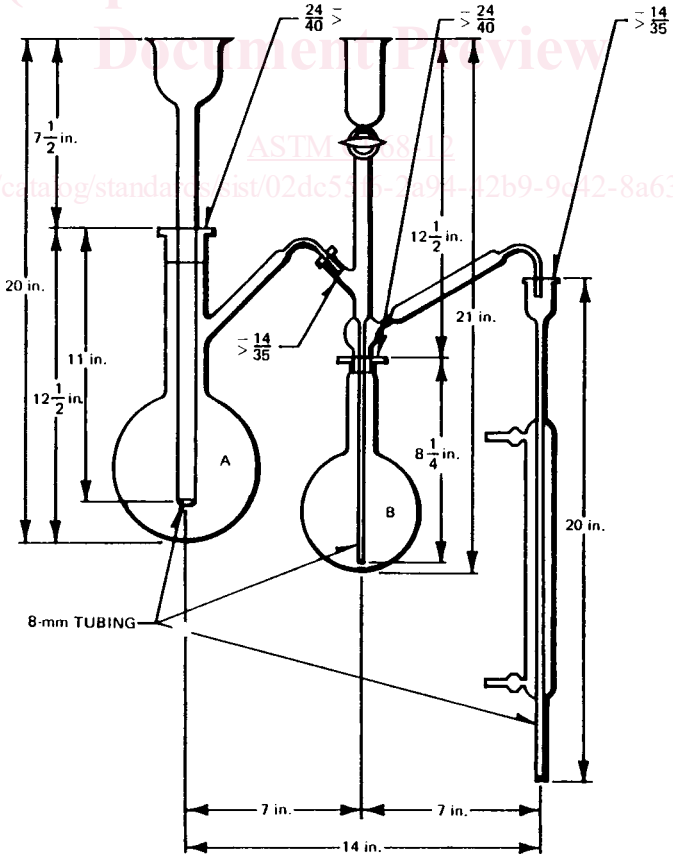
10.4 *Hydrochloric Acid* (6 N)—Dilute 6 volumes of concentrated hydrochloric acid (HCl) to 12 volumes.

10.5 *Hydrogen Peroxide* (30 %).

11. Precautions

11.1 The use of ammonia or other volatile nitrogenous compounds in the vicinity of the experiment can lead to serious errors. To ensure freedom from contamination, take the following precautionary measures:

11.1.1 Steam clean all glassware immediately prior to use.



A–1000-ml ROUND-BOTTOM FLASK
B–100-ml ROUND-BOTTOM FLASK

FIG. 1 Micro-Kjeldahl Apparatus