



Standard Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method¹

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1. Scope

1.1 This test method covers the determination of carbon in nuclear-grade uranium oxide powders and pellets to determine compliance with specifications.

1.2 Gadolinium oxide (Gd_2O_3) and gadolinium oxide-uranium oxide powders and pellets may also be analyzed using this test method.

1.3 This test method covers the determination of 5 to 500 μg of residual carbon.

1.4 This test method describes an induction furnace carrier gas combustion system equipped with an infrared detector. It may also be applied to a similar instrument equipped with a thermal conductivity detector.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5.1 The preferred system of units is micrograms carbon per gram of sample ($\mu g/g$ sample) or micrograms carbon per gram of uranium ($\mu g/g$ U).

1.6 *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.*

2. Referenced Documents

2.1 ASTM Standards:²

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets

C888 Specification for Nuclear-Grade Gadolinium Oxide (Gd_2O_3) Powder

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

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

C922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets

3. Summary of Test Method

3.1 The powered or crushed test specimen and an appropriate accelerator (metal flux) are added to a crucible, placed within an induction-heated furnace and burned at a nominal temperature of 1600 to 1700°C in a stream of oxygen. A catalyst converts the carbon monoxide (CO) to carbon dioxide (CO_2) and the products of combustion are scavenged free of sulfur compounds, halogens, and water vapor. The CO_2 is swept into an infrared cell detector. The amount of carbon is automatically determined from stored calibration data, and is displayed or printed out, or both, by the carbon analyzer.

3.2 The actual configuration of the system may vary with vendor and model. Typical systems include columns of materials such as copper oxide, platinized silica gel, magnesium perchlorate, sodium hydroxide, and cellulose to purify the CO_2 stream.

4. Significance and Use

4.1 Uranium dioxide is used as a nuclear-reactor fuel. Gadolinium oxide is used as an additive to uranium dioxide. In order to be suitable for this purpose, these materials must meet certain criteria for impurity content. This test method is designed to determine whether the carbon content meets Specifications C753, C776, C888, and C922.

5. Interferences

5.1 Contamination of carrier gas, crucibles, or samples with extraneous sources of carbon may cause a positive bias. The blank correction will help to minimize the bias from carrier gas and crucibles. Interference from absorbed carbon on samples may be eliminated by keeping the sample in an inert atmosphere or vacuum.

5.2 Powdered Gd_2O_3 samples may adsorb CO/ CO_2 from the atmosphere. Sample preheating to 120° for 2 h is recommended in this case.

5.3 The purification system typically associated with the recommended combustion and detection equipment is designed to minimize other expected sources of interferences, such as sulfur, halogens, and water.

6. Apparatus

6.1 *Low-Carbon Analyzer*, consisting of an induction-heated furnace suitable for operation at 1600 to 1700°C, an infrared detector for measuring carbon dioxide, and auxiliary purification systems.

6.2 *Crucibles*, expendable alumina or similar refractory material. Both the crucible and cover, if used, must be pre-ignited at a temperature of 1000°C or higher for a time sufficient to produce constant blank values.

6.3 *Muffle Furnace*, capable of attaining temperature of 1000°C, for pre-igniting crucibles.

6.4 *Tongs and Forceps*, for handling crucibles and lids.

6.5 *Stainless Steel Scoops and Spatulas*

7. Reagents and Materials

7.1 *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.

7.2 *Accelerators*—Copper metal, tin-tungsten mixture⁴, copper oxide, granular tin, and high purity iron chip accelerators for increased combustion temperature. These materials are available in appropriate purity and form from carbon analyzer vendors. The criterion for satisfactory results is the absence of significant additional carbon release upon recombustion of the specimen.

7.3 *Cellulose Trap Packing*—Surgical grade cotton or equivalent.

7.4 *Carbon Dioxide and Moisture Absorbents*—Sodium hydroxide (NaOH) on a fibrous support⁵ and magnesium perchlorate⁶

8. Carbon Standard Materials

8.1 NIST SRM steel standards or equivalent:

³ *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 The sole source of supply of the apparatus, Lecocel accelerator, known to the committee at this time is LECO Corporation. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ that you may attend.

⁵ The sole source of supply of the apparatus, ascarite, known to the committee at this time is J.T. Baker, Inc. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ that you may attend.

⁶ The sole source of supply of the apparatus, anhydrone, known to the committee at this time is J.T. Baker Inc. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ that you may attend.

8.1.1 The 101, 131, 133, 339, and 343 series, ranging from approximately 20 µg/g sample to 1500 µg/g sample have been found satisfactory.

8.2 LECO⁷ or SYLAB steel standards⁸

8.2.1 The 1 g steel pin standards or steel rings, ranging from approximately 5 µg/g sample to 500 µg/g sample have been found satisfactory.

9. Hazards and Precautions

9.1 Take proper safety precautions to prevent inhalation, or ingestion of uranium dioxide powders or dust during grinding or handling operations.

9.2 Operation of equipment presents electrical and thermal hazards. Follow manufacturer recommendations for safe operation.

9.3 This procedure uses hazardous chemicals. Use appropriate precautions for handling corrosives, oxidizers, and gases.

10. Preparation of Apparatus

10.1 Change instrument column packing and reagents as recommended by manufacturer.

10.2 Set the operating controls of the instrument system according to the operating instructions for the specific equipment used.

10.3 Condition the apparatus by combustion of several blanks prepared with sample crucible and accelerator in the amount to be used with the samples. Successive blanks should approach a constant value, allowing for normal statistical fluctuations.

11. Calibration

11.1 The calibration range and number of standards will depend upon the instrument used. Two to four standards containing 50 to 600 µ g carbon are recommended.

11.2 Add a volumetrically measured or weighed portion of accelerator and weighed portion of standard to a pre-ignited sample crucible. (The amount of accelerator will depend upon the instrument used and operating conditions.)

11.3 Load and combust the standards according to the manufacturer's recommended operating conditions.

11.4 Calibrate the instrument according to operating instructions. Calibration coefficients are normally stored in the microprocessor memory.

11.5 Recalibration frequency will depend upon the type of instrument used. As a minimum, recalibration is required when critical instrument components are changed, or when control standards data indicate that the instrument is failing to meet performance criteria.

⁷ LECO, 300 Lakeview Ave. St. Joesph, Michigan 49085 USA.

⁸ SYLAB, 11 Imp. Courtes patures, F57073 Metz Cedex, 03, France.

12. Procedure

12.1 Crush pellet samples to obtain approximately 1-mm (18 mesh) particles. (The particle size required may vary according to the instrument used and operating conditions.)

12.2 Measure or weigh accelerator into a pre-ignited sample crucible. (The amount of accelerator will depend upon the instrument used and operating conditions.)

12.3 Weigh a portion of sample, (0.3 g to 2 g is suggested) to the nearest 0.01 g, into the crucible. The sample size should be chosen to provide adequate sensitivity and accuracy at low carbon concentrations, but should contain less than 200 µg of carbon.

12.4 Load the crucible into the induction furnace and combust the sample according to the manufacturer's recommended operating conditions. (Combustion time will vary with the instrument used, but it is typically in the 30 to 120-s range.) Record the result.

12.5 Remove the sample crucible and examine it for incomplete combustion. The crucible contents should be a uniformly fused mass. Results from incompletely fused samples shall not be accepted.

13. Calculation

13.1 Calculate the carbon content as follows:

$$\mu\text{g C per g of sample} = (C_s - C_b)/W \quad (1)$$

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where:

C_s = micrograms of carbon in test specimen,
 C_b = micrograms of carbon in a blank run, and
 W = grams of test specimen.

13.2 For samples requiring carbon results expressed as µg carbon per g U, convert results to uranium basis as follows:

$$C, \mu\text{g/g U basis} = \frac{C \mu\text{g/g} \times 100}{\% \text{ U content of sample}} \quad (2)$$

14. Precision and Bias

14.1 The precision and bias for this test method will depend upon the instrument used and the operating conditions.

14.2 The relative standard deviation for a NIST 101G steel standard (136 µg/g) analyzed on the same instrument was 3.4 %. The bias, as measured by percent recovery of the standard's value, was +1.4 %. These data represent 45 standards collected by multiple operators using one instrument, over a one-year period.

14.3 The relative standard deviation for a 36 µg/g working uranium oxide powder standard analyzed at one laboratory was 8.0 %. The bias, as measured by percent recovery of the standard's value, was +7.0 %. These data represent 214 standards measured by multiple operators using one instrument, over a one-year period.

15. Keywords

15.1 carbon content; gadolinium oxide; gadolinium oxide-uranium oxide; impurity content; uranium oxide