Designation: C1408 – 16

Standard Test Method for Carbon (Total) in Uranium Oxide Powders and Pellets By Direct Combustion-Infrared Detection Method[1]

This standard is issued under the fixed designation C1408; 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 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 (µg/g sample) or micrograms carbon per gram of uranium (µg/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

C859 Terminology Relating to Nuclear Materials

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

C922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets

2.2 *NIST Standard:*[3]

NIST SRM 101G Standard Reference Materials—Stainless Steel

3. Terminology

3.1 *Definitions*—Except as otherwise defined herein, definitions of terms are given in Terminology C859.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *accelerator*—an igniter and a flux which promotes both combustion and a fluid melt by effectively lowering the melting point of the sample.

4. Summary of Test Method

4.1 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. The carbon in the sample is oxidized to primarily carbon dioxide (CO_2) with some carbon monoxide (CO) formed. A catalyst converts the CO to 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.

4.2 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.

5. Significance and Use

5.1 Uranium dioxide is used as a nuclear-reactor fuel. Gadolinium oxide is used as an additive to uranium dioxide. In

[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.

[3] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

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.

6. Interferences

6.1 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.

6.2 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.

6.3 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. The external/auxiliary purification systems are designed to minimize/remove hydrocarbons, water, and CO_2. Special scrubbers are used for halogens created during sample combustion.

7. Apparatus

7.1 *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.

7.2 *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.

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

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

7.5 *Stainless Steel Scoops and Spatulas*

8. Reagents and Materials

8.1 *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.[4] 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.

8.2 *Accelerators*—Copper metal and iron chip mixture, tin-tungsten mixture[5], granular tin and iron chip mixture, or 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.

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

8.4 *Carbon Dioxide and Moisture Absorbents*—Sodium hydroxide (NaOH) on an inert support and magnesium perchlorate ($Mg(ClO_4)_2$.

9. Carbon Standard Materials

9.1 NIST SRM steel standards or equivalent:

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

9.2 LECO[6] or SYLAB steel standards[7]

9.2.1 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.

10. Hazards and Precautions

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

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

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

11. Preparation of Apparatus

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

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

11.3 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.

12. Calibration

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

[4] *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.

[5] 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.

[6] LECO, 3000 Lakeview Ave. St. Joesph, Michigan 49085 USA.

[7] SYLAB, 11 Imp. Courtes patures, F57073 Metz Cedex, 03, France.