



Designation: C 1408 – 98

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

This standard is issued under the fixed designation C 1408; 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 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:

C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder²

C 776 Specification for Sintered Uranium Dioxide Pellets²

C 888 Specification for Nuclear-Grade Gadolinium Oxide Powder²

C 922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets²

3. Summary of Test Method

3.1 The powdered 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 (CO₂) and the products of combustion are scavenged free of sulfur compounds, halogens, and water vapor. The CO₂ 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 CO₂ 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 C 753, C 776, C 888, and C 922.

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/CO₂ 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.

¹ This test method is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

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² *Annual Book of ASTM Standards*, Vol 12.01.