Designation: C 1457 – 00

Standard Test Method for Determination of Total Hydrogen Content of Uranium Oxide Powders and Pellets by Carrier Gas Extraction[1]

This standard is issued under the fixed designation C 1457; 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 applies to the determination of hydrogen in nuclear-grade uranium oxide powders and pellets to determine compliance with specifications. Gadolinium oxide (Gd_2O_3) and gadolinium oxide-uranium oxide powders and pellets may also be analyzed using this test method.

1.2 This standard describes a procedure for measuring the total hydrogen content of uranium oxides. The total hydrogen content results from absorbed water, water of crystallization, hydro-carbides and other hydrogenated compounds which may exist as fuel's impurities.

1.3 This test method covers the determination of 0.05 to 200 µg of residual hydrogen.

1.4 This test method describes an electrode furnace carrier gas combustion system equipped with a thermal conductivity detector.

1.5 The preferred system of units is micrograms hydrogen per gram of sample (µg/g sample) or micrograms hydrogen 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:*

C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder[2]

C 776 Specification for Sintered Uranium Dioxide Pellets[2]

C 888 Specification for Nuclear-Grade Gadolinium Oxide (Gd_2O_3) Powder[2]

C 922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets[2]

3. Summary of Test Method

3.1 The total hydrogen content is determined using a hydrogen analyzer. The hydrogen analyzer is based on the carrier gas method using argon or nitrogen as carrier gas. The actual configuration of the system may vary with vendor and model.

3.2 The samples to be analyzed are dropped into a preheated graphite crucible, and then, heated up to a temperature of more than 1700°C in a graphite crucible. At that temperature hydrogen, oxygen, nitrogen, and carbon monoxide (oxygen is converted to CO when it reacts with the crucible) are released. The release gas is purified in the carrier gas stream by oxidation and absorption columns. The hydrogen is separated by chromatographic means and analyzed in a thermal conductivity detector.

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 hydrogen 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 hydrogen may cause a positive bias. A blank correction will help to minimize the bias from carrier gas and crucibles. Interference from adsorbed hydrogen on samples may be eliminated by keeping the sample in an inert atmosphere or vacuum.

5.2 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, carbon monoxide, carbon dioxide, and water.

5.2.1 The nitrogen and hydrogen peaks are close together and must be well-separated to prevent falsely high result from the nitrogen. The molecular sieve must be sufficiently long to

[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.