Designation: C1430 – 07 (Reapproved 2011)ε1

Standard 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[1]

This standard is issued under the fixed designation C1430; 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.

ε[1] NOTE—A units statement was added editorially in June 2011.

1. Scope

1.1 This test method applies to the determination of uranium, the oxygen to uranium (O/U) ratio in sintered uranium dioxide pellets, and the oxygen to metal (O/M) ratio in sintered gadolinium oxide-uranium dioxide pellets with a Gd_2O_3 concentration of up to 12 weight %. The O/M calculations assume that the gadolinium and uranium oxides are present in a metal dioxide solid solution.

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

1.3 *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.* For specific hazards statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets

C776 Specification for Sintered Uranium Dioxide Pellets

C922 Specification for Sintered Gadolinium Oxide-Uranium Dioxide Pellets

C968 Test Methods for Analysis of Sintered Gadolinium Oxide-Uranium Dioxide Pellets

C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry

3. Summary of Test Method

3.1 The uranium, and either O/U or O/M, are determined by measuring the weight change of a sintered pellet after it has been exposed to an equilibrating atmosphere to bring it to the stoichiometric condition. Sintered pellets are weighed and loaded into a sample boat. The boat is placed in a tube furnace capable of holding a temperature of 800 ± 10°C. The furnace is purged with a moist gas flow of 4 % hydrogen and 96 % argon or nitrogen to remove all air. The temperature of the furnace is raised to 800°C and held at this temperature with constant gas flow for 4 h. The furnace then is turned off and allowed to cool, with gas purge on, to room temperature. The samples are removed from the furnace and reweighed.

3.2 The weight change, gadolinia content, and chemical impurity content are used to calculate % uranium and the O/U or O/M.

4. Significance and Use

4.1 Uranium dioxide is used as a nuclear-reactor fuel. This test method is designed to determine whether the percent uranium and O/U or O/M content meet Specifications C776 and C922.

5. Interferences

5.1 Parameters for temperature, gas composition, gas flow, and moist air purge must be monitored and maintained carefully within the limits set in the procedure.

5.2 This test method assumes that chemical impurities meet Specifications C776 and C922 limits. Potential method interferences from higher impurity concentrations will require evaluation.

5.3 Furnace tubes or boats made from metals that oxidize under the test conditions may prevent proper equilibration by consuming available oxygen.

[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.

5.4 Precise weighing of samples is critical to the accuracy of this test method.

5.5 Loss of weight due to pellet chipping would invalidate the analysis. Handle pellets with care.

5.6 This test method assumes that pellets are sintered. It does not correct for moisture or volatile additives.

5.7 This test method assumes that UO_2-Gd_2O_3 pellets have formed a solid solution; however, the error from incomplete dissolution of Gd_2O_3 would be very small (see the calculation in 10.2).

6. Apparatus

6.1 *Analytical Balance,* capable of weighing to ± 0.0001 g.

6.2 *Tube Furnace,* capable of controlling temperatures at 800 ± 10°C, that has been fitted with a fused quartz furnace tube.

6.3 *Fused Quartz Sample Boats.*

6.4 *Assorted Connectors, Tubing, Flasks, Stoppers, and Delivery Tubes*—The purge gas is passed through a humidifier, into the tube furnace. A bubbler flask is attached to the furnace outlet to monitor gas flow (see Fig. 1).

6.5 *Gas Pressure Gage and Regulator.*

6.6 *Purge Gas* (4 % hydrogen, 96 % argon or 4 % hydrogen and 96 % nitrogen. Gas purity of 99.995 % has been found to perform satisfactorily.

6.7 *Purge Gas Humidifier,* with heater and controller capable of maintaining water temperature at 35 ± 10°C.

7. Standard Materials

7.1 NBL[3], NBL-traceable, or equivalent, uranium dioxide pellets. Analyze at least one standard pellet per batch.

8. Hazards and Precautions

8.1 Take proper safety precautions for handling uranium.

8.2 The furnace, sample tube and sample boats are heated to 800°C. Care must be taken to avoid burns.

8.3 Exercise appropriate caution when working with compressed gasses.

9. Procedure

9.1 Analyze samples as whole pellets. No preparation is required. The nominal sample size is 5–10-g pellet. Smaller pellets may need to be composited (two pellets/test) to maintain minimum weight. Avoid using chipped or cracked pellets.

9.2 Place a small weighing tray or watch glass on the balance pan. Tare the balance and check to ensure that the balance is stable. If the balance will not stabilize, do not proceed.

NOTE 1—The extremely small weight changes that are being measured in this test method make it critical that the balance is working properly.

9.3 Weigh a check weight at least daily to confirm that the analytical balance is operating correctly.

[3] Available from the New Brunswick Laboratory, 9800 S. Cass Ave., Argonne, IL.

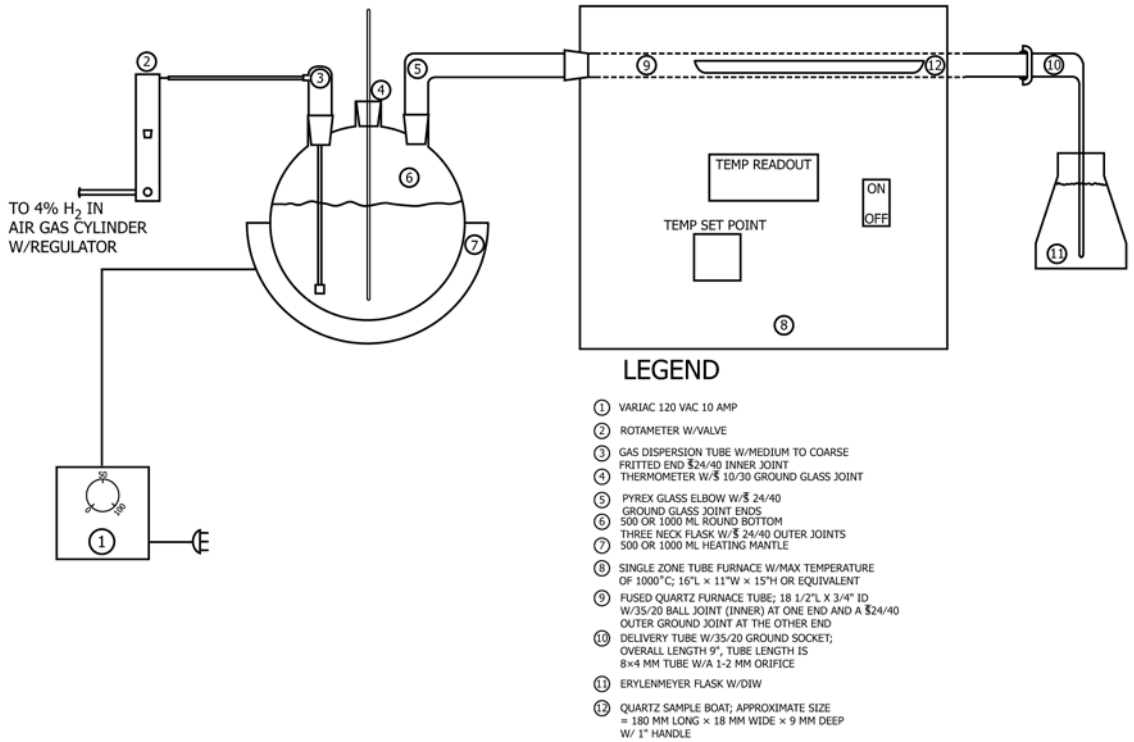

FIG. 1 Assorted Connectors, Tubing, Flasks, Stoppers, and Delivery Tubes