Designation: C1430 – 18

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.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazards statements, see Section 9.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 for Light Water Reactors

C859 Terminology Relating to Nuclear Materials

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. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to the nuclear fuel cycle, refer to Terminology C859.

4. Summary of Test Method

4.1 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.

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

5. Significance and Use

5.1 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.

[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.

6. Interferences

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

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

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

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

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

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

6.7 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 11.2).

7. Apparatus

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

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

7.3 *Fused Quartz Sample Boats.*

7.4 *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).

7.5 *Gas Pressure Gage and Regulator.*

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

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

8. Standard Materials

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

9. Hazards and Precautions

9.1 Take proper safety precautions for handling uranium.

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

9.3 Exercise appropriate caution when working with compressed gasses.

10. Procedure

10.1 Analyze samples as whole pellets. No preparation is required. The nominal sample size is 5–10-g pellet. Smaller

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

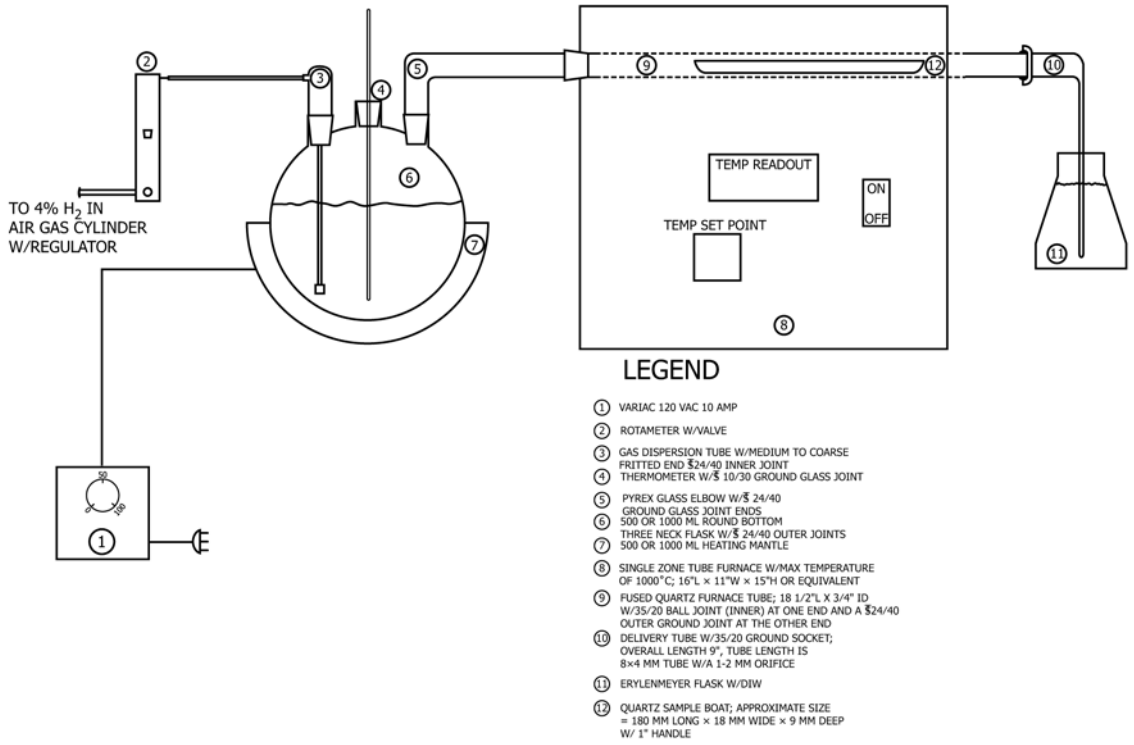

FIG. 1 Assorted Connectors, Tubing, Flasks, Stoppers, and Delivery Tubes

pellets may need to be composited (two pellets/test) to maintain minimum weight. Avoid using chipped or cracked pellets.

10.2 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.

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

10.4 Create a boat map to maintain sample identity.

10.5 Use a pair of tweezers and carefully weigh the pellet. Rezero the balance and repeat the pellet weighing until a consistent weight is obtained. Carefully place the pellet in the quartz sample boat. Repeat for each pellet.

10.6 Include one or two equilibrated standard control pellets with each sample batch.

10.7 Carefully place the loaded boat into the sample tube. Position the boat as close to center of the furnace tube as possible.

10.8 Fit the purge gas connection to end of tube and clamp. Make certain that the water in the humidifying flask is at 35 ± 10°C (± 5°C is optimal) and check the gas cylinder pressure to verify there is sufficient gas to complete the cycle.

10.9 Turn on the gas flow and allow the chamber to purge for approximately five minutes.

NOTE 2—The flow rate of the purge gas and the length of the purge cycle will vary with the size of the furnace tube. A purge of greater than or equal to three furnace volumes/minute is the recommended minimum. The flow rate must be adequate to maintain a positive pressure inside the sample chamber.

10.10 Attach a Pyrex® delivery tube with ground glass fitting to the exit end of the furnace, and place the end in a container of water to verify and monitor the gas flow.

10.11 Turn on the furnace and bring the temperature to 800°C.

10.12 After temperature is reached, allow the pellets to equilibrate for a minimum of 4 h. Monitor the system occasionally during the run to ensure constant temperature and gas flow.

10.13 At the end of the 4-h cycle, turn the furnace down to 50°C and allow the samples to cool. The purge gas flow must be maintained until the samples reach 50°C. Then, turn off the carrier gas and allow the pellets to cool to room temperature.

NOTE 3—If the samples are allowed to cool to room temperature while the purge gas is flowing, the water in the purge gas will begin to condense inside the tube and on the pellets. A temperature of 50°C is high enough to prevent condensation but low enough to prevent oxidation by room air.

10.14 Remove the sample boat and reweigh the pellets immediately. Use multiple weighings as necessary to obtain a consistent weight.

11. Calculation

11.1 *O/U (UO_2 Pellets):*

$$= 2.000 - \frac{(W_2 - W_1)}{(W_2)\,[(AW_o)/(AW_u + 2\,AW_o)]} \quad (1)$$

$$= 2.000 - \frac{(W_2 - W_1)}{(W_2)\,(0.0593)}$$

where:

W_1 = Weight of sample before equilibration, g,
W_2 = Weight of sample after equilibration, g,
AW_o = Atomic weight of oxygen,
AW_u = Atomic weight of uranium, and
0.0593 = $\frac{(AW_o)}{(AW_u + 2\,AW_o)}$.

11.2 *O/M (UO_2-Gd_2O_3 Pellets):*

$$\text{Pellet O/M} = 2.000 - \Delta\ \text{O/M} \quad (2)$$

$$= 2.000 - \frac{(W_2 - W_1)}{(W_2)\,[0.0593 + (\%\ Gd_2O_3 \times 0.00026)]}$$

where:

W_1 = Weight of sample before equilibration, g,
W_2 = Weight of sample after equilibration, g,
% Gd_2O_3 = Measured Gd, expressed as stoichiometric weight % Gd_2O_3, and
(% Gd_2O_3)(0.00026) = Correction factor for weight gain due to formation of oxygen-rich UO_2-Gd_2O_3 solid solution during sintering. For processes that do not produce a 100 % solid solution, this factor should be evaluated to determine if modification is necessary (see Appendix X1).

11.3 *Percent Uranium:*

11.3.1 *Percent Uranium, Based on Sample Weight:*

$$\%U = \frac{\left[\frac{(100 - G - \%I)}{15.9994}\right] - \left[\frac{O/M \times G}{157.25}\right]}{\left[\frac{O/M}{AW_u}\right] + \left[\frac{1}{15.9994}\right]} \quad (3)$$