Designation: C 1453 – 00

Standard Test Method for the Determination of Uranium by Ignition and the Oxygen to Uranium (O/U) Atomic Ratio of Nuclear Grade Uranium Dioxide Powders and Pellets[1]

This standard is issued under the fixed designation C 1453; 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 uranium and the oxygen to uranium atomic ratio in nuclear grade uranium dioxide powder and pellets.

1.2 This test method does not include provisions for preventing criticality accidents or requirements for health and safety. Observance of this test method does not relieve the user of the obligation to be aware of and conform to all international, national, or federal, state and local regulations pertaining to possessing, shipping, processing, or using source or special nuclear material.

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.*

1.4 This test method also is applicable to UO_3 and U_3O_8 powder.

2. Referenced Documents

2.1 *ASTM Standards:*

C 696 Test Methods for Chemical, Mass Spectrometric and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets[2]

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

C 776 Specification for Sintered Uranium Dioxide Pellets[2]

C 1267 Test Method for Uranium by Iron II Reduction in Phosphoric Acid Followed by Chromium VI Titration in the Presence of Vanadium[2]

C 1287 Test Method for Determination of Impurities in Uranium Dioxide by Inductively Coupled Plasma Mass Spectrometry[2]

3. Summary of Test Method

3.1 A weighed portion of UO_2 is converted to U_3O_8 by repeated ignition at 900°C in air, to a constant weight. Corrections are made for nonvolatile and volatile impurities including moisture, based on independent determinations described in Test Methods C 696 and C 1287.[3,4]

4. Significance and Use

4.1 The test method is designed to show whether or not a material meets the specifications as given in Specifications C 753 or C 776.

4.2 The powder's stoichiometry is useful for predicting the oxide's sintering behavior in the pellet production process.

5. Interferences

5.1 The moisture content must be determined and a correction must be made for the moisture content otherwise a high bias will occur for the O/U ratio.

5.2 A nonvolatile impurity correction must be made otherwise a high bias will occur for the uranium value. An extended ignition time may be required if significant amounts of anions that are difficult to decompose are present.

5.3 The U_3O_8 to uranium conversion factor and the uranium atomic weight will require adjustment for nonnatural isotopic concentrations otherwise a bias will be present.

6. Apparatus

6.1 *Desiccator*, containing a moisture absorbent.

6.2 *Muffle Furnace*, capable of maintaining and controlling temperatures to 900 ± 25°C.

6.3 *Analytical Balance*, capable of weighing to ± 0.1 mg.

6.4 *Platinumware*.

7. Reagents and Materials

7.1 Anhydrous magnesium perchlorate − $Mg(ClO_4)_2$, moisture absorbent, or equivalent.

[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.

[3] Jones, R.J., Ed., "Selected Measurement Methods for Plutonium and Uranium in the Nuclear Fuel Cycle," *USAEC Document TID-7029*, 1963, AERDB, pp. 91–93.

[4] Petit, G.D. and Keinberger, C.A., "Preparation of Stoichiometric U_3O_8," *Analytical Chemistry, ANCHA* Vol 25, 1961, p. 579.

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