Designation: C1453 – 19

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 C1453; 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 test method also is applicable to UO_3 and U_3O_8 powder.

1.4 *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.*

1.5 *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

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets for Light Water Reactors

C859 Terminology Relating to Nuclear Materials

C1267 Test Method for Uranium by Iron (II) Reduction in Phosphoric Acid Followed by Chromium (VI) Titration in the Presence of Vanadium

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

3. Terminology

3.1 For definitions of terms used in this test method but not defined herein, refer to Terminology C859.

4. Summary of Test Method

4.1 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 C696 and C1287.[3,4]

5. Significance and Use

5.1 The test method is designed to show whether or not a material meets the specifications as given in Specifications C753 or C776.

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

6. Interferences

6.1 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.

[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.

[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.

6.2 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.

6.3 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.

7. Apparatus

7.1 *Desiccator,* containing a moisture absorbent.

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

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

7.4 *Platinumware.*

8. Reagents and Materials

8.1 Anhydrous magnesium perchlorate – $Mg(ClO_4)_2$, moisture absorbent, or equivalent.

9. Procedure

9.1 Transfer 2 to 12 g of UO_2 powder or pellets to a tared platinum crucible and weigh to within 0.1 mg.

9.1.1 *UO_2 Powder*—Place the platinum crucible containing the UO_2 powder sample in a muffle furnace and ignite for 3 h at 900 ± 25°C.

9.1.2 *UO_2 Pellets*—Preheat the pellets at 500°C for 3 h, in the muffle furnace, then ignite for 3 h at 900 ± 25°C.

TABLE 1 Oxide Conversion Factors for Impurity Correction

Impurity	Assumed Oxide Form	Oxide Conversion Factor[A]
Al	Al_2O_3	1.89
B	B_2O_3	3.23
Ba	BaO	1.12
Be	BeO	2.78
Bi	Bi_2O_3	1.11
Ca	CaO	1.40
Cd	CdO	1.14
Co	Co_2O_3	1.41
Cr	Cr_2O_3	1.46
Cu	CuO	1.25
Fe	Fe_2O_3	1.43
In[B]	In_2O_3	1.21
Li	Li_2O	2.15
Mg	MgO	1.66
Mn	MnO_2	1.58
Mo	MoO_3	1.50
Na	Na_2O	1.35
Ni	NiO	1.27
P	P_2O_5	2.29
Pb	PbO_2	1.15
Sb	Sb_2O_4	1.26
Si	SiO_2	2.14
Sn	SnO_2	1.27
Ti	TiO_2	1.67
V	V_2O_5	1.79
Zn	ZnO	1.24
Zr	ZrO_2	1.35
Ta	Ta_2O_5	1.22
W	WO_3	1.26

[A] Oxide conversion factor is defined as grams oxide per gram of element.
[B] This element is not required by the UO_2 Specifications C753 and C776 but is included for information only.

TABLE 2 UO_2 Powder Results

Sample Type	Uranium wt % Absolute	Standard Deviation	O/U Ratio	Standard Deviation	No. of Determinations
Sample A	87.796	0.005	2.065	0.001	12
Sample B	86.996	0.004	2.225	0.001	12

9.2 Remove the crucible from the furnace, allow to cool in the air 2 to 3 minutes, then place the crucible in a desiccator and cool to room temperature. Weigh the crucible.

9.3 Repeat the ignition for 3 h at 900°C and repeat step 9.2 until a constant weight of ±0.3 mg is obtained.

9.4 Other ignition and cooling schemes may be used as long as the analyst verifies the precision and the bias of the measurement.

10. Calculation

10.1 *Uranium Content*—Calculate as follows:

$$U,\ wt\% = [(0.8480\,(W - WI)/S)\ X\ 100] - C \quad (1)$$

where:

0.8480 = U_3O_8 to uranium conversion factor for natural uranium. This factor will require adjustment when the uranium isotopic abundance deviates from natural uranium. See Appendix X1.2.
W = Grams of U_3O_8 after ignition.
I = Total of all detected nonvolatile impurities expressed as grams of oxide per gram of ignited U_3O_8. See Table 1 to obtain oxide conversion factors for many common impurity elements encountered. The impurities are determined as described in either Test Method C696 or C1287.
S = Initial sample weight, in g.
C = Total of all nonvolatile impurities analyzed as less than the lower detection limit of the analytical method. The detection limit values shall be taken as the concentration of that element. The total is expressed as percent. These impurities are determined as described in Test Method C696 or C1287. Alternatively, the these impurities can be considered to contribute a total correction of 0.01 % to the uranium percent.

10.2 Oxygen-to-Uranium Ratio—Calculate as follows from the original sample U, wt%:

$$O/U = [(100 - U\ wt\% - Z - m)(A)]/[(15.999)(U\ wt\%)] \quad (2)$$

where:

O = atom % of oxygen
U = atom % of uranium
U wt% = U, weight %, as calculated in 10.1
Z = total non-volatile impurities correction, %, as determined in Test Method C696 or C1287.
m = moisture and volatile impurity content, %, determined in Test Method C696 or C1287.
A = atomic weight of uranium based on isotopic abundance. See X1.1.
15.999 = atomic weight of oxygen