



Designation: C1453 – 00 (Reapproved 2006)

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 Pellets¹

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 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: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

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

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.

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 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}

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 **C753** or **C776**.

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 $900 \pm 25^\circ\text{C}$.

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

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.

6.4 Platinumware.

7. Reagents and Materials

7.1 Anhydrous magnesium perchlorate – $\text{Mg}(\text{ClO}_4)_2$, moisture absorbent, or equivalent.

8. Procedure

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

8.1.1 *UO₂ Powder*—Place the platinum crucible containing the UO_2 powder sample in a muffle furnace and ignite for 3 h at $900 \pm 25^\circ\text{C}$.

8.1.2 *UO₂ Pellets*—Preheat the pellets at 500°C for 3 h, in the muffle furnace, then ignite for 3 h at $900 \pm 25^\circ\text{C}$.

8.2 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.

8.3 Repeat the ignition for 3 h at 900°C and repeat step 8.2 until a constant weight of ± 0.3 mg is obtained.

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

9. Calculation

9.1 *Uranium Content*—Calculate as follows:

$$U, \text{ wt\%} = [(0.8480 (W - I)/S) \times 100] - C \quad (1)$$

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, these impurities can be considered to contribute a total correction of 0.01 % to the uranium percent.

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

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

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.

where:

O = atom % of oxygen

U = atom % of uranium

U wt% = U, weight %, as calculated in 9.1

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

10. Precision and Bias

10.1 *UO₂ Powder*—The precision for the O/U ratio for UO_2 powder is shown in Table 2. The bias was not determined as there are no standards due to the relative reactivity of the powder. The data in Table 2 were determined by one analyst over a three-day period.

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

10.2 UO₂ Pellets: