



# 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<sup>1</sup>

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 ( $\epsilon$ ) 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  $\text{UO}_3$  and  $\text{U}_3\text{O}_8$  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<sup>2</sup>

C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder<sup>2</sup>

C 776 Specification for Sintered Uranium Dioxide Pellets<sup>2</sup>

C 1267 Test Method for Uranium by Iron II Reduction in Phosphoric Acid Followed by Chromium VI Titration in the Presence of Vanadium<sup>2</sup>

C 1287 Test Method for Determination of Impurities in Uranium Dioxide by Inductively Coupled Plasma Mass Spectrometry<sup>2</sup>

## 3. Summary of Test Method

3.1 A weighed portion of  $\text{UO}_2$  is converted to  $\text{U}_3\text{O}_8$  by

repeated ignition at  $900^\circ\text{C}$  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.<sup>3,4</sup>

## 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  $\text{U}_3\text{O}_8$  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  $\pm 0.1$  mg.

6.4 *Platinumware*.

## 7. Reagents and Materials

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

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 12.01.

<sup>3</sup> 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.

<sup>4</sup> Petit, G.D. and Keinberger, C.A., "Preparation of Stoichiometric  $\text{U}_3\text{O}_8$ ," *Analytical Chemistry*, *ANCHA* Vol 25, 1961, p. 579.

## 8. Procedure

8.1 Transfer 2 to 12 g of  $\text{UO}_2$  powder or pellets to a tared platinum crucible and weigh to within 0.1 mg.

8.1.1 *UO<sub>2</sub> Powder*—Place the platinum crucible containing the  $\text{UO}_2$  powder sample in a muffle furnace and ignite for 3 h at  $900 \pm 25^\circ\text{C}$ .

8.1.2 *UO<sub>2</sub> Pellets*—Preheat the pellets at  $500^\circ\text{C}$  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  $900^\circ\text{C}$  and repeat step 8.2 until a constant weight of  $\pm 0.3$  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:

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

where:

0.8480 =  $\text{U}_3\text{O}_8$  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  $\text{U}_3\text{O}_8$  after ignition.

I = Total of all detected nonvolatile impurities expressed as grams of oxide per gram of ignited  $\text{U}_3\text{O}_8$ . See Table 1 to obtain oxide conversion factors for many common impurity elements encountered. The impurities are determined as described in either Test Method C 696 or C 1287.

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 C 696 or C 1287. 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%:

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

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 C 696 or C 1287.

m = moisture and volatile impurity content, %, determined in Test Method C 696 or C 1287.

A = atomic weight of uranium based on isotopic abundance. See X1.1.

15.999 = atomic weight of oxygen

TABLE 1 Oxide Conversion Factors for Impurity Correction

Impurity	Assumed Oxide Form	Oxide Conversion Factor <sup>A</sup>
Al	$\text{Al}_2\text{O}_3$	1.89
B	$\text{B}_2\text{O}_3$	3.23
Ba	BaO	1.12
Be	BeO	2.78
Bi	$\text{Bi}_2\text{O}_3$	1.11
Ca	CaO	1.40
Cd	CdO	1.14
Co	$\text{Co}_2\text{O}_3$	1.41
Cr	$\text{Cr}_2\text{O}_3$	1.46
Cu	CuO	1.25
Fe	$\text{Fe}_2\text{O}_3$	1.43
In <sup>B</sup>	$\text{In}_2\text{O}_3$	1.21
Li	$\text{Li}_2\text{O}$	2.15
Mg	MgO	1.66
Mn	$\text{MnO}_2$	1.58
Mo	$\text{MoO}_3$	1.50
Na	$\text{Na}_2\text{O}$	1.35
Ni	NiO	1.27
P	$\text{P}_2\text{O}_5$	2.29
Pb	$\text{PbO}_2$	1.15
Sb	$\text{Sb}_2\text{O}_4$	1.26
Si	$\text{SiO}_2$	2.14
Sn	$\text{SnO}_2$	1.27
Ti	$\text{TiO}_2$	1.67
V	$\text{V}_2\text{O}_5$	1.79
Zn	ZnO	1.24
Zr	$\text{ZrO}_2$	1.35
Ta	$\text{Ta}_2\text{O}_5$	1.22
W	$\text{WO}_3$	1.26

<sup>A</sup> Oxide conversion factor is defined as grams oxide per gram of element.

<sup>B</sup> This element is not required by the  $\text{UO}_2$  Specifications C 753 and C 776 but is included for information only.

## 10. Precision and Bias

10.1 *UO<sub>2</sub> Powder*—The precision for the O/U ratio for  $\text{UO}_2$  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  $\text{UO}_2$  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 *UO<sub>2</sub> Pellets*:

10.2.1 The precision for the O/U ratio for  $\text{UO}_2$  pellets is shown in Table 3.<sup>5</sup> The data were determined by two different laboratories. The data for Laboratory A were determined over several days by one analyst. The data for laboratory B were determined over seven days by four analysts using three furnaces.

10.2.2 The precision for the uranium content for  $\text{UO}_2$  pellets is shown in Table 4.<sup>5</sup> The data were determined as described for Table 3.

10.2.3 The reference value for G-2 working pellet standard was obtained from the weighted average of eight ferrous sulphate type titration measurements and nine ignition type measurements. The measurements were standardized against

<sup>5</sup> Supporting data for Tables 2-X1.1 are available from ASTM Headquarters.

NBL 125 standard. One analyst performed the measurements over two weeks. The determined uranium value was 88.103 % with a standard deviation of 0.053 %. The value is not statistically different from the values determined by this test method. The data does not indicate any statistically significant bias at the 88.103 % level.

TABLE 3 Comparison of Interlab O/U Results

Laboratory	Sample Type	O/U Ratio	Standard Deviation	No. of Determinations
A	Sintered depleted UO <sub>2</sub> pellet G-2	2.001	0.001	4
B	Sintered depleted UO <sub>2</sub> pellet G-2	2.000	0.002	18

TABLE 4 Comparison of Interlab Uranium Results

Method	Laboratory	Sample Type	Uranium wt% Absolute	Standard Deviation	No. of Determinations
Gravimetric	A	Sintered depleted UO <sub>2</sub> pellet G-2	88.106	0.003	4
Gravimetric	B	Sintered depleted UO <sub>2</sub> pellet G-2	88.114	0.010	18
Reference value, see 10.2.3	B	Sintered depleted UO <sub>2</sub> pellet G-2	88.103	0.053	See 10.2.3

## 11. Keywords

11.1 gravimetric; ignition; O/U ratio; oxygen; oxygen to uranium ratio; uranium

## APPENDIX

### (Nonmandatory Information)

#### X1. ADDITIONAL CALCULATIONS AND INFORMATION

X1.1 The atomic weight of uranium based on the mass fraction of the individual isotopes is calculated as follows:

$$A = \sum Fi Ai \quad (X1.1)$$

where:

A = Atomic weight of uranium;

Fi = Mass fraction of uranium isotope i; and

Ai = Atomic weight of uranium isotope i.

X1.2 The conversion factor for U<sub>3</sub>O<sub>8</sub> to uranium is calculated as follows:

$$\text{Conversion Factor} = \frac{3A}{3A + 8O} \quad (X1.2)$$

where:

A = Atomic weight of uranium;

O = Atomic weight of oxygen (15.9994).

For natural isotopic abundance uranium the conversion factor is 0.8480.

X1.3 *Uranium Trioxide*—The uranium content of UO<sub>3</sub> can be determined using this test method.<sup>3</sup> (See Note X1.1). The results for the UO<sub>3</sub> were determined by a single set of comparative data. (See Table X1.1.)

NOTE X1.1—Sulphur, which is commonly present in UO<sub>3</sub> as UO<sub>2</sub>SO<sub>4</sub> is not completely volatilized in the conversion to U<sub>3</sub>O<sub>8</sub> if the ignition time is less than 3 h.<sup>3</sup>

TABLE X1.1 Uranium Content of UO<sub>3</sub>

Sample	Gravimetric Uranium %	Titrimetric <sup>A</sup> Uranium %	
UO <sub>3</sub>	82.78	Technician A 82.74	Technician B 82.81

<sup>A</sup> The titrimetric method refers to the Davies-Gray method in C 1267.

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