



# Standard Test Method for Analysis of Isotopic Composition of Uranium in Nuclear- Grade Fuel Material by Quadrupole Inductively Coupled Plasma-Mass Spectrometry<sup>1</sup>

This standard is issued under the fixed designation C1474; 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 is applicable to the determination of the isotopic composition of uranium (U) in nuclear-grade fuel material. The following isotopic weight percentages are determined using a quadrupole inductively coupled plasma-mass spectrometer (Q-ICP-MS):  $^{233}\text{U}$ ,  $^{234}\text{U}$ ,  $^{235}\text{U}$ ,  $^{236}\text{U}$ , and  $^{238}\text{U}$ . The analysis can be performed on various material matrices after acid dissolution and sample dilution into water or dilute nitric ( $\text{HNO}_3$ ) acid. These materials include: fuel product, uranium oxide, uranium oxide alloys, uranyl nitrate (UNH) crystals, and solutions. The sample preparation discussed in this test method focuses on fuel product material but may be used for uranium oxide or a uranium oxide alloy. Other preparation techniques may be used and some references are given. Purification of the uranium by anion-exchange extraction is not required for this test method, as it is required by other test methods such as radiochemistry and thermal ionization mass spectroscopy (TIMS). This test method is also described in ASTM STP 1344<sup>2</sup>.

1.2 The  $^{233}\text{U}$  isotope is primarily measured as a qualitative measure of its presence by comparing the  $^{233}\text{U}$  peak intensity to a background point since it is not normally found present in materials. The example data presented in this test method do not contain any  $^{233}\text{U}$  data. A  $^{233}\text{U}$  enriched standard is given in Section 8, and it may be used as a quantitative spike addition to the other standard materials listed.

1.3 A single standard calibration technique is used. Optimal accuracy (or a low bias) is achieved through the use of a single standard that is closely matched to the enrichment of the samples. The intensity or concentration is also adjusted to within a certain tolerance range to provide good statistical

counting precision for the low-abundance isotopes while maintaining a low bias for the high-abundance isotopes, resulting from high-intensity dead time effects. No blank subtraction or background correction is utilized. Depending upon the standards chosen, enrichments between depleted and 97 % can be quantified. The calibration and measurements are made by measuring the intensity ratios of each low-abundance isotope to the intensity sum of  $^{233}\text{U}$ ,  $^{234}\text{U}$ ,  $^{235}\text{U}$ ,  $^{236}\text{U}$ , and  $^{238}\text{U}$ . The high-abundance isotope is obtained by difference.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. The instrument is calibrated and the samples measured in units of isotopic weight percent (Wt %). For example, the  $^{235}\text{U}$  enrichment may be stated as Wt %  $^{235}\text{U}$  or as g  $^{235}\text{U}$ /100 g of U. Statements regarding dilutions, particularly for ug/g concentrations or lower, are given assuming a solution density of 1.0 since the uranium concentration of a solution is not important when making isotopic ratio measurements other than to maintain a reasonably consistent intensity within a tolerance range.

1.5 *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. Specific precautionary statements are given in Section 9.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder

C776 Specification for Sintered Uranium Dioxide Pellets

C778 Specification for Sand

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

C859 Terminology Relating to Nuclear Materials

<sup>1</sup> 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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<sup>2</sup> Policke, T. A., Bolin, R. N., and Harris, T. L., "Uranium Isotope Measurements by Quadrupole ICP-MS for Process Monitoring of Enrichment," *Symposium on Applications of Inductively Coupled Plasma-Mass Spectrometry to Radionuclide Determinations: Second Volume*, ASTM STP 1344, ASTM, 1998, p. 3.

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.