



Standard Test Method for Uranium and Plutonium Concentrations and Isotopic Abundances by Thermal Ionization Mass Spectrometry¹

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1. Scope

1.1 This test method covers the determination of the concentration and isotopic composition of uranium and plutonium in solutions. The purified uranium or plutonium from samples ranging from nuclear materials to environmental or bioassay matrices is loaded onto a mass spectrometric filament. The isotopic ratio is determined by thermal ionization mass spectrometry, the concentration is determined by isotope dilution.

1.2 The values stated in SI units are to be regarded as the standard. Values in parentheses are for information only.

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 safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder
- C757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable
- C776 Specification for Sintered Uranium Dioxide Pellets
- C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets
- C859 Terminology Relating to Nuclear Materials
- C1008 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets—Fast Reactor Fuel
- C1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry
- C1156 Guide for Establishing Calibration for a Measure-

ment Method Used to Analyze Nuclear Fuel Cycle Materials

C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis

C1411 Practice for The Ion Exchange Separation of Uranium and Plutonium Prior to Isotopic Analysis

C1415 Test Method for ²³⁸Pu Isotopic Abundance By Alpha Spectrometry

C1614 Practice for the Determination of ²³⁷Np, ²³²Th, ²³⁵U and ²³⁸U in Urine by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) and Gamma Ray Spectrometry

D3084 Practice for Alpha-Particle Spectrometry of Water

2.2 Other Documents

International Target Values 2010 for Measurement Uncertainties in Safeguarding Nuclear Materials³

3. Terminology

3.1 For definitions of pertinent terms not listed here, see Terminology C859.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *isotope dilution mass spectrometry (IDMS)*—isotope ratio measurements, using mass spectrometry, of samples spiked with accurately known weights of individual low abundance isotopes (adapted from Practice C1614).

4. Summary of Test Method

4.1 The uranium and plutonium are separated from each other and purified from other elements by selective extraction, anion exchange (such as in C1411) or extraction chromatography. The uranium and plutonium fractions are individually mounted on filaments of rhenium, tungsten, or tantalum, and are analyzed by thermal ionization mass spectrometry to determine the relative abundance of the isotopes. If a known ²³³U or ²⁴²Pu (or ²⁴⁴Pu) spike is added prior to chemical separation the corresponding elemental concentration may also be determined by isotope dilution mass spectrometry (IDMS).

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 K. Zhao et. al., "International Target Values 2010 for Measurement Uncertainties in Safeguarding Nuclear Materials," International Atomic Energy Agency STR-368, 2010.

5. Significance and Use

5.1 Uranium and plutonium oxides can be used as a nuclear-reactor fuel in the form of pellets. In order to be suitable for use as a nuclear fuel the starting material must meet certain specifications, such as found in Specifications [C757](#), [C833](#), [C753](#), [C776](#), [C1008](#), or as specified by the purchaser. The uranium and/or plutonium concentration and isotopic abundances are measured by mass spectrometry following this test method.

5.2 The separated heavy element fractions placed on mass spectrometric filaments must be very pure. The quantity required depends upon the sensitivity of the instrument detection system. If an electron multiplier detector is to be used, only a few nanograms are required. If a Faraday cup is used, a few micrograms are needed. Chemical purity of the sample becomes more important as the sample size decreases, because ion emission of the sample is suppressed by impurities.

6. Interferences

6.1 Uranium-238 and ^{238}Pu interfere in the measurement of each other, and ^{241}Am interferes with the measurement of ^{241}Pu , thereby requiring chemical separation. Removal of impurities provides uniform ionization of uranium or plutonium, hence improved precision, and reduces the interference from molecular species of the same mass number as the uranium or plutonium isotopes being measured. Isotopic analysis of plutonium should be completed within a reasonable time period (approximately 20 days) after separation from americium to minimize interference of ^{241}Am ingrowth from ^{241}Pu .

6.2 Extreme care must be taken to avoid contamination of the sample by environmental uranium. The level of uranium contamination should be measured by analyzing an aliquot of 8M nitric acid as a reagent blank and calculating the amount of uranium it contains.

6.3 When ^{238}Pu is present in low abundance it may be necessary to measure it by alpha-spectrometry following Test Method [C1415](#) or Practice [D3084](#).

7. Apparatus

7.1 *Mass Spectrometer*—The suitability of mass spectrometers for use with this test method of analysis shall be evaluated by means of performance tests described in this test method. The mass spectrometer used should possess the following characteristics:

7.1.1 A thermal ionization source with single or multiple filaments of rhenium, tungsten or tantalum.

7.1.2 An analyzer radius sufficient to resolve adjacent masses in the mass-to-charge range being studied, that is, $m/z = 233$ to 238 for U^+ or 238 to 244 for Pu^+ . Abundance sensitivity must be great enough to detect one part of ^{236}U in 400 parts ^{235}U .

7.1.3 A minimum of one stage of magnetic deflection. Since the resolution is not affected, the angle of deflection may vary with the instrument design.

7.1.4 A mechanism for changing samples.

7.1.5 A direct-current (Faraday cup) or electron multiplier detector, as a single detector system or, several detectors in a multi collector design, followed by a current measuring device.

7.1.6 A pumping system to attain a vacuum of less than 400 μPa (3×10^{-6} torr) in the source, the analyzer, and the detector regions.

7.1.7 A mechanism to scan masses by means of varying the magnetic field or the accelerating voltage.

7.1.8 A computer to collect and process data produced by the instrument.

7.2 An *Optical Pyrometer* should be available to determine the filament temperature.

7.3 Filament preheating and degassing unit for cleaning unloaded filaments.

8. Materials and Reagents

8.1 *Purity of Reagents*—all reagents used in the final purification and filament loading steps should be of the highest purity available. Other grades may be used if they are determined not to affect the final result.

8.2 *Filaments*—high purity, the size and configuration are instrument dependent. Filaments should be degassed, and maybe carbon saturated, prior to use.

NOTE 1—The purity of the filaments should be confirmed with each batch received. Zone refined filaments should be used for low-level analyses.

8.3 *Certified Reference Materials (CRM)*—of varying isotopic composition, traceable to a national standard body⁴, for use as calibration and quality control standards.

8.4 *Spikes*—Materials, preferably CRMs, for use in the determination of elemental concentration by IDMS.

9. Instrument Calibration

9.1 The measurement method may be qualified following Guide [C1068](#) and calibrated following Guide [C1156](#).

9.2 The measurement and correction for mass discrimination and dead time are critical factors in obtaining precise and accurate results. Equally critical to the accuracy of the measurement is the linearity of the total measuring circuit including the collector. Calibration of the mass spectrometer is based on the assumption that these are the only sources of significant (>1 in 10^4) systematic error in the measurement. Thus, accurate calibration is made by analyzing standards of known isotopic composition under conditions in which cross-contamination between samples does not occur.

9.2.1 For multi-collector systems, the bias between collectors may also be an important factor in the systematic error and thus must also be evaluated prior to making measurements.

9.2.2 For very low-level samples, or samples with extreme ratios, other corrections may need to be made, for example, dark count data/dark current.

9.3 *Mass Discrimination*—Use a traceable isotopic standard to determine the mass discrimination. The deviation from the

⁴ Available from USDOE New Brunswick Laboratory, Argonne, IL, or other equivalent source.