**Designation: C 1625 – 05**

# Standard Test Method for
# Uranium and Plutonium Concentrations and Isotopic Abundances by Thermal Ionization Mass Spectrometry[1]

This standard is issued under the fixed designation C 1625; 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 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 *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]

C 753 Specification for Nuclear Grade, Sinterable Uranium Dioxide Powder

C 757 Standard Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable

C 776 Specification for Nuclear Grade, Sinterable Uranium Dioxide Pellets

C 833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets

C 1008 Standard Specification of Sintered (Uranium-Plutonium) Dioxide Pellets – Fast Reactor Fuel

C 1068 Guide for Qualification of Measurement Methods by a Laboratory Within the Nuclear Industry

C 1156 Guide for Establishing Calibration for a Measurement Method Used to Analyze Nuclear Fuel Cycle Materials

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C 1347 Practice for Preparation and Dissolution of Uranium Materials for Analyis

C 1411 Practice for the Ion Exchange Separation of Uranium and Plutonium Prior to Isotopic Analysis

C 1415 Standard Test Method for $^{238}$Pu Isotopic Abundance by Alpha Spectrometry

D 3084 Practice for Alpha-Particle Spectrometry of Water

E 137 Practice for Evaluation of Mass Spectrometers for Quantitative Analysis from a Batch Inlet

2.2 *Other Documents*

International Target Values 2000 for Measurement Uncertainties in Safeguarding Nuclear Materials[3]

## 3. Summary of Test Method

3.1 The uranium and plutonium are separated from each other and purified from other elements by selective extraction, anion exchange (such as in C 1411) or extraction chromatography. The uranium and plutonium fractions are individually mounted on rhenium filaments and analyzed by thermal ionization mass spectrometry to determine the relative abundance of the isotopes. If a known $^{233}$U or $^{242}$Pu (or $^{244}$Pu) spike is added prior to chemical separation the corresponding elemental concentration may also be determined by isotope dilution mass spectrometry (IDMS).

## 4. Significance and Use

4.1 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 C 757, C 833, C 753, C 776, C 1008, or as specified by the purchaser. The uranium and/or plutonium concentration and isotopic abundances are measured by mass spectrometry following this test method.

4.2 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.

## 5. Interferences

5.1 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.

5.2 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 8$M$ nitric acid as a reagent blank and calculating the amount of uranium it contains.

5.3 When [238]Pu is present in low abundance it may be necessary to measure it by alpha-spectrometry following C 1415 or D 3084.

## 6. Apparatus

6.1 *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 and in Practice E 137. The mass spectrometer used should possess the following characteristics:

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

6.1.2 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.

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

6.1.4 A mechanism for changing samples.

6.1.5 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.

6.1.6 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.

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

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

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

6.3 Filament preheating and degassing unit for cleaning unloaded filaments.

## 7. Materials and Reagents

7.1 *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.

7.2 *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.

7.3 *Certified Reference Materials (CRM)*—of varying isotopic composition, traceable to a national standard body[4], for use as calibration and quality control standards.

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

## 8. Instrument Calibration

8.1 The measurement method may be qualified following C 1068 and calibrated following C 1156.

8.2 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.

8.2.1 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.

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

8.3 *Mass Discrimination*—Use a traceable isotopic standard to determine the mass discrimination. The deviation from the certified value of the 235/238 ratio (for U) or the 239/242 ratio (for Pu) is a measure of the mass discrimination of the mass spectrometer.

8.3.1 Calculate the elemental mass discrimination bias factor, $B$, as follows:

$$B = (1/c)[(aR_{i/j}/R_s)-1] \qquad (1)$$

where:
$B$ = mass discrimination factor,
$aR_{i/j}$ = average measured atom ratio of isotope $i$ to isotope $j$
$R_s$ = certified atom ratio value of the CRM
$c$ = $\Delta$ mass/mass. The values for $c$ for various ratios and ion species include:

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

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| ratio | $U^+$ or $Pu^+$ |
|---|---|
| $^{235}U/^{238}U$ | +3/238 |
| $^{236}U/^{235}U$ | -1/235 |
| $^{233}U/^{238}U$ | +5/238 |
| $^{234}U/^{235}U$ | +1/235 |
| $^{242}Pu/^{239}Pu$ | -3/239 |
| $^{240}Pu/^{239}Pu$ | -1/239 |
| $^{241}Pu/^{239}Pu$ | -2/239 |
| $^{238}Pu/^{239}Pu$ | +1/239 |

8.3.2 Correct every measured ratio, $R_{i/j}$, for mass discrimination as follows:

$$R_{i/j} = a R_{i/j}/(1+cB) \qquad (2)$$

where $R_{i/j}$ is the corrected atom ratio of isotope $i$ to isotope $j$

8.4 *Dead Time Correction*—Required for counting detectors, at high count rates. Use laboratory protocols for this correction with high count rate samples.

8.5 *Linearity*—The linearity of the mass spectrometer may be determined over the working ratio range by measuring the $^{235}U/^{238}U$ ratio, under identical conditions, of appropriate CRMs. The ratio of the certified $^{235}U/^{238}U$ ratio to the experimental $^{235}U/^{238}U$ ratio is independent of isotopic ratio if the system is linear. Under ideal conditions, any deviation from a constant value greater than 4 in 10,000 is likely to be nonlinearity. Uranium CRMs are used because the range of isotopic ratios of existing plutonium CRMs is not adequately large.

## 9. Procedure

9.1 *Sample Preparation*

9.1.1 *Sample Dissolution*—Dissolve an appropriate sample to obtain the desired filament loading for the mass spectrometric analysis. See C 1347 for the dissolution of uranium or C 1168 for plutonium. Add the appropriate amount of spike, by weight or volume, as appropriate, if concentration is to be determined by isotope dilution methods.

NOTE 2—Spike addition and equilibration must be performed prior to chemical purification if determining concentration by IDMS.

9.1.2 *Sample Purification*—Use C 1411 or similar procedure to separate the uranium and plutonium from each other and from other impurities.

9.2 *Filament Loading*—Samples may either be directly loaded by evaporation, electroplated, or loaded onto a resin bead for mounting on the filament. Samples and standards should be prepared for analysis by the same method at similar mass loadings.

9.3 *Sample Heating and Isotopic Ratio Measurement*:

9.3.1 Insert the filament assembly into the mass spectrometer. If the instrument contains a turret to allow loading of several samples, load at least one QC sample (blank or CRM) per wheel.

9.3.2 Seal the source and evacuate to a pressure of less than 400 μPA ($3 \times 10^{-6}$ torr).

9.3.3 Slowly begin heating the sample filament. If not done previously, hold the sample filament at a dull, red glow (500-700°C) for 5-30 minutes to permit outgassing (this may be performed in a separate system to reduce contamination of the mass spectrometry source). When outgassing has ceased, increase the ionizing filament temperature to emit ions. Typical emitting temperatures are 1450-1650°C for plutonium and 1650-1850°C for uranium.

9.3.4 Locate the uranium or plutonium spike peak, or the $^{238}U$ peak or the $^{239}Pu$ peak, if analyzing unspiked samples. Focus the major isotope beam by adjusting the magnetic field, the accelerating voltage, and any electrical or mechanical controls available.

9.3.5 The intensity of the major beam is adjusted until stable emission of the desired intensity is achieved. The emission rate should be constant or at least increase or decrease slowly and evenly.

9.3.6 When acceptable ion emission is reached, measure the relative intensities of the ion peaks of interest. Multiple measurements of isotope pairs are made to provide quality control parameters.

9.3.7 When sufficient data are collected to obtain the desired precision, turn off the filament current and discontinue the analysis.

9.3.8 Record and correct (see section 8) the isotopic ratios of the $i$th to the $j$th species for the unspiked sample ($R_{i/j}$), for the spike, ($S_{i/j}$) and for the sample-plus-spike mixture ($M_{i/j}$). The symbols for the isotopes $^{233}U$, $^{234}U$, $^{235}U$, $^{236}U$, $^{238}U$, $^{238}Pu$, $^{239}Pu$, $^{240}Pu$, $^{241}Pu$, and $^{242}Pu$ are abbreviated to 3, 4, 5, 6, 8, P8, 9, 0, 1, and 2, respectively (see section 10); note that these symbols do not include every isotope that may be measured. In this nomenclature, the observed ratios of $^{238}U$ to $^{233}U$ in the sample, the spike, and the sample-plus-spike mixture ($R_{i/j}$, $S_{i/j}$ and $M_{i/j}$) become $R_{8/3}$, $S_{8/3}$ and $M_{8/3}$, respectively.

## 10. Calculation

10.1 Calculate atom fraction $^{235}U$, $A_5$, on the unspiked U as follows:

$$A_5 = R_{5/8}/(R_{4/8} + R_{5/8} + R_{6/8} + R_{8/8}) \qquad (3)$$

where $R_{8/8}$ (which equals 1) is retained for clarity. Next, calculate atom fraction $^{238}U$, $A_8$, as follows:

$$A_8 = R_{8/8}/(R_{4/8} + R_{5/8} + R_{6/8} + R_{8/8}) \qquad (4)$$

In these equations, $^{238}U$ is assumed to be the principal isotope. For highly enriched U where $^{235}U$ is the principal isotope, obtain the ratio of each isotope to $^{235}U$ instead of to $^{238}U$ by using $R_{4/5}$, $R_{5/5}$, $R_{6/5}$, $R_{8/5}$ in place of $R_{4/8}$, $R_{5/8}$, $R_{6/8}$, and $R_{8/8}$. Finally, calculate the atom % $N_5$ and $N_8$ as follows:

$$N_5 = 100A_5 \qquad (5)$$

$$N_8 = 100A_8 \qquad (6)$$

If desired, calculate $N_4$ and $N_6$ similarly by dividing the corresponding atom ratio by the same sum of four ratios as shown in Eq 3 and Eq 4 and by multiplying the resultant atom fraction by 100 to obtain percent as shown in Eq 5 and Eq 6.

10.2 Calculate the corresponding atom fraction $^{239}Pu$, $A_9$, and atom percent $^{239}Pu$, $N_9$, on the unspiked Pu fraction as follows:

$$A_9 = R_{9/9} / (R_{9/9} + R_{0/9} + R_{1/9} + R_{2/9}) \qquad (7)$$

$$N_9 = 100A_9 \qquad (8)$$

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where $R_{9/9}$ (which equals 1) is retained for clarity. If required, include $R_{P8/9}$ determined by either mass spectrometry (this test method) or alpha spectrometry (C 1415). If desired, calculate $N_{P8}$, $N_0$, $N_1$, and $N_2$ similarly by dividing the corresponding atom ratio by the same sum of four ratios shown in Eq 7 and by multiplying by 100 to obtain percent as shown in Eq 8.

10.3 For accountability, it may be necessary to report isotopic abundances in weight percent. Calculate weight percent $^{235}$U and $^{239}$Pu as follows:

$$\text{Weight percent } ^{235}U = (W_5 \times 100)/(W_4 + W_5 + W_6 + W_8) \quad (9)$$

where:
$W_4 = A_4 \times 234.04$,
$W_5 = A_5 \times 235.04$,
$W_6 = A_6 \times 236.05$, and
$W_8 = A_8 \times 238.05$.

$$\text{Weight percent } ^{239}Pu = (W_9 \times 100)/(W_9 + W_0 + W_1 + W_2) \quad (10)$$

where:
$W_{P8} = A_{P8} \times 238.05$,
$W_9 = A_9 \times 239.05$,
$W_0 = A_0 \times 240.05$,
$W_1 = A_1 \times 241.06$, and
$W_2 = A_2 \times 242.06$.

10.3.1 If desired, calculate the weight percent $^{234}$U, $^{236}$U, and $^{238}$U similarly by dividing $W_4$, $W_6$, and $W_8$ in turn by ($W_4$+ $W_5$+ $W_6$+ $W_8$) and by multiplying the resultant weight fraction by 100 to obtain percent. The weight percent $^{238}$Pu, $^{240}$Pu, $^{241}$Pu, and $^{242}$Pu can be found similarly by dividing $W_{P8}$, $W_0$, $W_1$, and $W_2$ by ($W_{P8}$+ $W_9$+ $W_0$+ $W_1$+ $W_2$) and by multiplying the resulting weight fraction by 100 to obtain percent.

10.4 Calculate the U concentration in atoms per milliliter or gram $U$, for low- and high- $^{235}$U-enrichment samples as follows:

$$U = (\text{mL spike/mL sample}) \times (S_3/A_8)\{(M_{8/3} - S_{8/3})/[1 - (M_{8/3}/R_{8/3})]\} \quad (11)$$

$$U = (\text{mL spike/mL sample}) \times (S_3/A_5)\{(M_{5/3} - S_{5/3})/[1 - (M_{5/3}/R_{5/3})]\} \quad (12)$$

Calculate the Pu concentration in atoms per milliliter or gram, $Pu$, as follows:

$$Pu = (\text{mL spike/mL sample}) \times (S_2/A_9)\{(M_{9/2} - S_{9/2})/[1 - (M_{9/2}/R_{9/2})]\} \quad (13)$$

Calculate the weight concentration of U by multiplying the atoms U/mL, $U$, by the millimolecular weight of the U under test (that is, $W_4$+ $W_5$+ $W_6$+ $W_8$) and dividing by the number of atoms in a millimole as follows:

$$\text{mg U/mL} = U \frac{\text{millimolecular weight of U}}{6.022 \times 10^{20} \text{ atoms/millimole}} \quad (14)$$

Similarly for Pu,

$$\text{mg Pu/mL} = Pu \frac{\text{millimolecular weight of Pu}}{6.022 \times 10^{20} \text{ atoms/millimole}} \quad (15)$$

## 11. Precision and Bias

11.1 An interlaboratory program, the Safeguards Measurement Evaluation Program, in which USDOE New Brunswick Laboratory provides materials to participating facilities for analysis, was used to provide interlaboratory comparisons of analysis of the same materials. Data from these analyses of both uranium and plutonium materials were used as the basis for the statistical evaluation of the test method. The data, accumulated over several analytical periods that extend over more than a year's time, are summarized in Tables 1-5 for laboratories whose performance is within, or close to, International Target Values. Percent relative difference (%RD), defined as 100*(Measured Value-Reference Value)/Reference Value, is an indicator of bias. The standard deviation (sd) is an indicator of precision.

11.2 Isotopic Abundance Measurements: The mean %RDs for high-enriched (90 %) uranium $^{235}$U measurements are ≤ 0.006, with sds of ≤0.008 %. The mean %RDs for low-enriched (4 %) uranium $^{235}$U measurements are ≤0.07, with sds of ≤0.11 %. The mean %RDs for $^{239}$Pu measurements are ≤0.018, with sds of ≤0.016 %. Results for the other uranium and plutonium isotopic abundances are also included in Tables 1-3, for information only.

11.3 IDMS Measurements: The mean %RDs for uranium IDMS measurements on several different material types are ≤0.05, with sds of ≤0.12 %. The mean %RDs for plutonium IDMS measurements on two different material types are ≤0.14, with sds of ≤0.13 %. See Table 4 and Table 5, respectively.

**TABLE 1 Uranium Isotopic Results, High-Enrichment Materials (90 % enriched)**

| | Lab A | Lab F | Lab J | | Lab A | Lab F | Lab J |
|---|---|---|---|---|---|---|---|
| **Material 42 (UNH solution)** | | | | **Material 43 (UNH solution)** | | | |
| N | 12 | 12 | 19 | N | 16 | 4 | 8 |
| Mean % RD, U-234 | 0.025 | -0.010 | 0.068 | Mean % RD, U-234 | -0.038 | -0.091 | 0.064 |
| sd | 0.215 | 0.082 | 0.038 | sd | 0.175 | 0.062 | 0.028 |
| Mean % RD, U-235[A] | 0.002 | 0.004 | 0.004 | Mean % RD, U-235[B] | 0.003 | 0.006 | 0.006 |
| sd | 0.007 | 0.007 | 0.006 | sd | 0.008 | 0.006 | 0.004 |
| Mean % RD, U-236 | -0.32 | -0.13 | -0.16 | Mean % RD, U-236 | -0.29 | -0.10 | -0.11 |
| sd | 0.21 | 0.04 | 0.09 | sd | 0.19 | 0.03 | 0.07 |
| Mean % RD, U-238 | -0.008 | -0.036 | -0.043 | Mean % RD, U-238 | -0.016 | -0.049 | -0.063 |
| sd | 0.077 | 0.070 | 0.063 | sd | 0.090 | 0.075 | 0.049 |

[A] 95 % confidence interval of Material 42 U-235 abundance is ±0.003 %.
[B] 95 % confidence interval of Material 43 U-235 abundance is ±0.004 %.

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**TABLE 2 Uranium Isotopic Results, Low-Enrichment Material**
**(4% enriched)**

|  | Lab F | Lab M | Lab P | Lab T |
|---|---|---|---|---|
| **CRM 125-A**[A]**(UO₂ pellet)** |  |  |  |  |
| **N** | 18 | 32 | 20 | 36 |
| **Mean % RD, U-234** | 0.05 | 0.047 | -0.29 | 0.27 |
| **sd** | 0.35 | 1.04 | 0.99 | 2.38 |
| **Mean % RD, U-235** | -0.029 | -0.015 | -0.071 | 0.059 |
| **sd** | 0.032 | 0.111 | 0.095 | 0.034 |
| **Mean % RD, U-236**[B] |  |  |  |  |
| **Mean % RD, U-238** | 0.001 | 0.001 | 0.002 | -0.004 |
| **sd** | 0.001 | 0.005 | 0.004 | 0.005 |

[A]95 % confidence interval of U-235 abundance is ±0.07 %.
[B]U-236 abundance is too low for meaningful measurements.

**TABLE 3 Plutonium Isotopic Results**

|  | Lab F | Lab T |  | Lab F | Lab T |
|---|---|---|---|---|---|
| **CRM 136**[A] **Pu-239=85 %** |  |  | **CRM 137**[B] **Pu-239=78 %** |  |  |
| **N** | 11 | 10 | **N** | 12 | 10 |
| **Mean % RD, Pu-238** | 0.37 | 1.92 | **Mean % RD, Pu-238** | 0.07 | 0.94 |
| **sd** | 0.14 | 1.18 | **sd** | 0.15 | 0.55 |
| **Mean % RD, Pu-239** | 0.005 | 0.011 | **Mean % RD, Pu-239** | 0.005 | 0.018 |
| **sd** | 0.006 | 0.007 | **sd** | 0.016 | 0.010 |
| **Mean % RD, Pu-240** | -0.038 | -0.086 | **Mean % RD, Pu-240** | -0.010 | -0.060 |
| **sd** | 0.043 | 0.031 | **sd** | 0.056 | 0.037 |
| **Mean % RD, Pu-241** | 0.11 | -0.11 | **Mean % RD, Pu-241** | 0.04 | -0.09 |
| **sd** | 0.21 | 0.11 | **sd** | 0.16 | 0.05 |
| **Mean % RD, Pu-242** | -0.25 | -0.21 | **Mean % RD, Pu-242** | -0.21 | -0.31 |
| **sd** | 0.15 | 0.28 | **sd** | 0.21 | 0.07 |

[A]95 % confidence intervals for Pu-239 abundance is ±0.018 %.
[B]95 % confidence intervals for Pu-239 abundance is ±0.028 %.

**TABLE 4 Uranium IDMS Results**

|  | Lab A |  | Lab J |  | Lab A |
|---|---|---|---|---|---|
| **Normal-Enrichment UNH Solutions**[A] |  | **High-Enrichment UNH Solutions**[B] |  | **Low-Enriched UO₃ powder**[C] |  |
| **N** | 88 | **N** | 37 | **N** | 32 |
| **Mean % RD** | 0.000 | **Mean % RD** | -0.049 | **Mean % RD** | -0.030 |
| **sd** | 0.120 | **sd** | 0.067 | **sd** | 0.123 |

[A]95 % confidence interval of U assay of normal-enrichment solutions is approximately ±0.02 %.
[B]95 % confidence interval of U assay of high-enrichment solutions is approximately ± 0.01 %.
[C]95 % confidence interval of U assay of low-enrichment UO₃ powder is ±0.012 %.

## 12. Keywords

12.1 thermal ionization mass spectrometry; uranium, plutonium; isotope ratio; atom percent; mass percent; isotope dilution mass spectrometry

**TABLE 5 Plutonium IDMS Results**

|  | Lab F | Lab G |  | Lab F | Lab G |
|---|---|---|---|---|---|
| **CRM 122 oxide**[A] **(Pu-239=87 %)** |  |  | **CRM 126 metal**[B] **(Pu-239=99+%)** |  |  |
| **N** | 8 | 8 | **N** | 11 | 8 |
| **Mean % RD** | 0.057 | -0.144 | **Mean % RD** | 0.044 | -0.012 |
| **sd** | 0.105 | 0.041 | **sd** | 0.068 | 0.130 |

[A]95 % confidence interval of CRM 122 plutonium assay is ± 0.044 %.
[B]95 % confidence interval of CRM 126 plutonium assay is ±0.018 %.

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