Designation: ~~C1637 – 13~~ C1637 – 21

Standard Test Method for ~~the~~ Determination of Impurities in Plutonium ~~Metal: Acid~~ Materials—Acid Digestion and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) Analysis[1]

This standard is issued under the fixed designation C1637; 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~~test method covers the determination of ~~58~~ trace elements in plutonium (Pu) ~~metal.~~ materials such as Pu metal, Pu oxides, and Pu/uranium (U) mixed oxides. The Pu sample is dissolved in acid, and the concentration of the trace impurities are determined by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS).

1.2 This ~~Test Method~~test method is specific for the determination of trace impurities ~~in Pu metal. It may be applied to other types of Pu materials, such as Pu oxides, if the samples are dissolved and oxidized to the Pu(IV) state. However,~~ where the samples are dissolved and the oxidation state is adjusted to the Pu(IV) and, if applicable, the U(VI) state. It may be applied to other matrices; however, it is the responsibility of the user to evaluate the performance of other matrices.

1.3 The use of a quadrupole ICP-MS or a high resolution ICP-MS (HR-ICP-MS) can be employed in all applications relevant to this test method. HR-ICP-MS is a better option in many cases since it can reduce or potentially eliminate interferences encountered in the following complex sample matrices.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety ~~concerns~~ concerns, if any, associated with its use. It is the responsibility of the user of this ~~method~~standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and ~~to~~ determine the applicability of regulatory limitations prior to ~~use of this standard.~~use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder for Light Water Reactors

C758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal

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[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.

C759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions
C859 Terminology Relating to Nuclear Materials
C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis
C1432 Test Method for Determination of Impurities in Plutonium: Acid Dissolution, Ion Exchange Matrix Separation, and Inductively Coupled Plasma-Atomic Emission Spectroscopic (ICP/AES) Analysis
D1193 Specification for Reagent Water
E1154 Specification for Piston or Plunger Operated Volumetric Apparatus

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

4. Summary of Test Method

4.1 Samples of plutonium materials are prepared and dissolved in accordance with an appropriate procedure in Practice C1168. These procedures adjust the dissolved Pu to the Pu(IV) oxidation state.

4.2 A sample of Pu metal is weighed and dissolved as described in Practice C1168. An aliquot of the original sample is then taken and diluted with 1 % HNO_3 by volume to a prescribed volume. Aliquots from a second dilution of the original sample are used to prepare run batch dilutions that are analyzed for trace impurities by ICP-MS (Section 10).[3]

4.3 Plutonium oxide powders are weighed and then dissolved in a nitric acid / hydrofluoric acid or ammonium bifluoride solution following the process described in Practice C1168. An aliquot of the dissolved sample is then taken and diluted with 0.1 M HNO_3 by volume to a prescribed volume in order to reach a certain concentration of Pu. In direct analysis mode, a second dilution of the original sample is necessary to prepare run batch dilutions that are analyzed for trace impurities by ICP-MS (Section 11).

4.4 ~~A sample of Pu metal is~~ Mixed oxide powders are weighed and then dissolved in a ~~small volume of 6 M hydrochloric acid (HCl). Then, 10 M nitric acid (HNO~~nitric acid /~~$_3$)/0.03 M hydrofluoric acid (HF) is added to the~~ hydrofluoric acid solution following the process described in Practice C1168~~dissolved Pu to oxidize the Pu to the Pu(IV)~~. Mixed oxide (U, Pu) pellets are first crushed and then dissolved in a nitric acid / hydrofluoric acid solution following the process described in Practice C1168~~state.~~. An aliquot of the ~~original~~dissolved sample is then taken and diluted with ~~1 %~~ 0.5 M HNO_3 by volume to a prescribed ~~volume. Aliquots from a~~ volume in order to reach a certain concentration of Pu. In direct analysis mode, a second dilution of the original sample ~~are used~~is necessary to prepare to run batch dilutions that are analyzed for trace impurities by ICP-MS. For mixed oxide powders and pellets, the standard additions method is recommended where standards at known concentration are added to the diluted dissolved sample and the solutions are analyzed by ICP-MS (Section 12).

5. Significance and Use

5.1 This test method may be run together with Test Method C1432 to analyze for trace impurities in Pu metal. Using the technique described in this test method and the technique described in Test Method C1432 will provide the analyst with a more thorough verification of the impurity concentrations contained in the Pu metal sample. In addition, Test Method C1432 can be used to determine impurity concentrations for analytes such as Ca, Fe, Na, and Si, which have not been determined using this test method.

5.2 This test method can be used on Pu matrices in nitrate solutions.

5.3 This test method has been validated for use on materials that meet the specifications described in Specification C757 and Test Methods C758 and C759.

5.4 This test method has been validated for all elements listed in Table 1.

6. Interferences

6.1 Ions from doubly charged (2+) species are formed in the ICP-MS. The actinide related ~~spectral~~isobaric interferences are from

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[3] "Inductively Coupled Plasma – Mass Spectrometry Using the VG Elemental Plasma Quad," *Actinide Analytical Chemistry ~~Procedures~~Procedures, LA-UR-05-7605*, Los Alamos National Laboratory, ~~ANC102 R.1.2, LA-UR-05-7605, 200~~2004~~.~~ .

TABLE 1 Impurity Elements, Mean Percent Recoveries and Percent Relative Standard Deviations

~~Element~~	~~N~~	~~Mean R, %~~	~~RSD, %~~
~~Lithium~~	~~22~~	~~93.65~~	~~7.26~~
~~Beryllium~~	~~22~~	~~96.46~~	~~8.14~~
~~Boron~~	~~22~~	~~98.48~~	~~6.97~~
~~Magnesium~~	~~22~~	~~98.30~~	~~7.58~~
~~Aluminium~~	~~22~~	~~99.66~~	~~8.62~~
~~Phosphorus~~	~~22~~	~~99.43~~	~~8.96~~
~~Titanium~~	~~22~~	~~99.25~~	~~2.44~~
~~Vanadium~~	~~22~~	~~94.44~~	~~7.38~~
~~Chromium~~	~~22~~	~~97.29~~	~~3.90~~
~~Manganese~~	~~22~~	~~95.48~~	~~3.46~~
~~Cobalt~~	~~22~~	~~95.92~~	~~4.35~~
~~Nickel~~	~~22~~	~~96.78~~	~~3.98~~
~~Zinc~~	~~22~~	~~94.24~~	~~4.12~~
~~Copper~~	~~22~~	~~96.66~~	~~3.70~~
~~Germanium~~	~~22~~	~~98.16~~	~~4.54~~
~~Arsenic~~	~~22~~	~~101.38~~	~~8.67~~
~~Selenium~~	~~22~~	~~101.15~~	~~8.00~~
~~Rubidium~~	~~22~~	~~100.24~~	~~5.36~~
~~Strontium~~	~~22~~	~~98.89~~	~~4.16~~
~~Yttrium~~	~~22~~	~~98.07~~	~~3.81~~
~~Zirconium~~	~~22~~	~~98.10~~	~~3.41~~
~~Niobium~~	~~22~~	~~96.92~~	~~3.65~~
~~Molybdenum~~	~~22~~	~~97.82~~	~~3.81~~
Molybdenum[A]	*21*	*98.36*	*2.90*
~~Ruthenium~~	~~22~~	~~98.32~~	~~2.14~~
~~Palladium~~	~~22~~	~~97.69~~	~~2.49~~
~~Silver~~	~~22~~	~~105.14~~	~~7.88~~
Silver[A]	*21*	*106.56*	*4.26*
~~Cadmium~~	~~22~~	~~96.03~~	~~3.72~~
~~Indium~~	~~22~~	~~98.01~~	~~3.57~~
~~Tin~~	~~22~~	~~97.25~~	~~3.94~~
~~Antimony~~	~~22~~	~~95.05~~	~~6.21~~
~~Tellurium~~	~~22~~	~~100.10~~	~~6.86~~
~~Caesium~~	~~22~~	~~101.81~~	~~6.93~~
~~Barium~~	~~22~~	~~97.99~~	~~3.68~~
~~Lanthanum~~	~~22~~	~~98.31~~	~~3.84~~
~~Cerium~~	~~22~~	~~97.57~~	~~3.72~~
~~Praeseodymium~~	~~22~~	~~97.32~~	~~3.00~~
~~Neodymium~~	~~22~~	~~97.22~~	~~3.56~~
~~Samarium~~	~~22~~	~~98.39~~	~~3.34~~
~~Europium~~	~~22~~	~~97.43~~	~~3.02~~
~~Gadolinium~~	~~22~~	~~100.04~~	~~2.78~~
~~Terbium~~	~~22~~	~~97.62~~	~~2.72~~
~~Dysprosium~~	~~22~~	~~98.18~~	~~2.20~~
~~Holmium~~	~~22~~	~~98.61~~	~~2.21~~
~~Erbium~~	~~22~~	~~98.05~~	~~2.29~~
~~Ytterbium~~	~~22~~	~~99.59~~	~~2.43~~
~~Lutetium~~	~~22~~	~~97.06~~	~~5.00~~
Lutetium[A]	*21*	*97.79*	*3.72*
~~Hafnium~~	~~22~~	~~100.32~~	~~3.95~~
~~Tantalum~~	~~22~~	~~93.42~~	~~3.21~~
Tantalum[A]	*21*	*93.89*	*2.43*
~~Tungsten~~	~~22~~	~~96.29~~	~~3.54~~
~~Rhenium~~	~~22~~	~~99.75~~	~~3.28~~
~~Iridium~~	~~22~~	~~99.88~~	~~3.70~~
~~Platinum~~	~~22~~	~~100.57~~	~~3.93~~
~~Gold~~	~~22~~	~~101.20~~	~~5.35~~
Gold[A]	*21*	*100.41*	*3.96*
~~Thallium~~	~~22~~	~~100.09~~	~~5.02~~
~~Lead~~	~~22~~	~~101.58~~	~~5.54~~
~~Bismuth~~	~~22~~	~~100.70~~	~~5.43~~
~~Thorium~~	~~22~~	~~103.30~~	~~6.89~~
~~Uranium~~	~~22~~	~~104.14~~	~~9.11~~

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Phosphorus	22	99.43	8.96
Titanium	22	99.25	2.44

Vanadium	22	94.44	7.38
Chromium	22	97.29	3.90
Manganese	22	95.48	3.46
Cobalt	22	95.92	4.35
Nickel	22	96.78	3.98
Zinc	22	94.24	4.12
Copper	22	96.66	3.70
Germanium	22	98.16	4.54
Arsenic	22	101.38	8.67
Selenium	22	101.15	8.00
Rubidium	22	100.24	5.36
Strontium	22	98.89	4.16
Yttrium	22	98.07	3.81
Zirconium	22	98.10	3.41
Niobium	22	96.92	3.65
Molybdenum	22	97.82	3.81
Molybdenum[A]	*21*	*98.36*	*2.90*
Ruthenium	22	98.32	2.14
Palladium	22	97.69	2.49
Silver	22	105.14	7.88
Silver[A]	*21*	*106.56*	*4.26*
Cadmium	22	96.03	3.72
Indium	22	98.01	3.57
Tin	22	97.25	3.94
Antimony	22	95.05	6.21
Tellurium	22	100.10	6.86
Caesium	22	101.81	6.93
Barium	22	97.99	3.68
Lanthanum	22	98.31	3.84
Cerium	22	97.57	3.72
Praeseodymium	22	97.32	3.00
Neodymium	22	97.22	3.56
Samarium	22	98.39	3.34
Europium	22	97.43	3.02
Gadolinium	22	100.04	2.78
Terbium	22	97.62	2.72
Dysprosium	22	98.18	2.20
Holmium	22	98.61	2.21
Erbium	22	98.05	2.29
Ytterbium	22	99.59	2.43
Lutetium	22	97.06	5.00
Lutetium[A]	*21*	*97.79*	*3.72*
Hafnium	22	100.32	3.95
Tantalum	22	93.42	3.21
Tantalum[A]	*21*	*93.89*	*2.43*
Tungsten	22	96.29	3.54
Rhenium	22	99.75	3.28
Iridium	22	99.88	3.70
Platinum	22	100.57	3.93
Gold	22	101.20	5.35
Gold[A]	*21*	*100.41*	*3.96*
Thallium	22	100.09	5.02
Lead	22	101.58	5.54
Bismuth	22	100.70	5.43
Thorium	22	103.30	6.89
Uranium	22	104.14	9.11

[A] Without ~~Outlying Value~~outlying value.

actinide 2+ and actinide-oxide 2+. The ~~spectral~~isobaric interferences are observed at 120.5 and 127.5 atomic mass unit ~~(amu),~~(AMU), when analyzing plutonium-239.

6.2 ~~Spectral~~Isobaric interferences occur from the argon plasma and the acid used to transport the sample to the plasma. These ~~spectral~~isobaric interferences occur between 12 and 80 ~~amu.~~ AMU.

6.3 Ions from plutonium cause a matrix related signal suppression. Signal suppression increases as the Pu concentration increases. In order to minimize signal suppression effects from Pu, samples are diluted so that the concentration of Pu in the analyzed aliquot is less than 500 µg/mL. Three internal standards are added to samples to correct for matrix related signal suppression and signal drift. Scandium, rhodium and thulium are used as internal standards. Analytes at the low end of the mass range (below 75 ~~amu)~~AMU) are referenced to scandium. Rhodium is a reference for analytes at the middle of the mass range ~~(76-138)~~ (76 to 138) and all analytes at the high end of the mass range are referenced to thulium ~~(139-238 amu).~~ (139 to 238 AMU).

6.4 The use of a collision/reaction system, or similar technology, with a quadrupole ICP-MS, or the use of a high-resolution ICP-MS, may reduce or eliminate some of these interferences.

7. Apparatus

7.1 *ICP-MS Instrument—*~~An ICP-MS instrument with a quadrupole mass spectrometer and a electron multiplier that operates at 1 amu resolution is used for this determination. The instrument can also be a magnetic sector instrument or a time of flight instrument.~~ One of the following:

7.1.1 An ICP-MS instrument with a quadrupole mass spectrometer and an electron multiplier that operates at 1 AMU resolution can be used. A collision/reaction system, or similar technology, can be used to reduce interferences.

7.1.2 A high-resolution (also known as magnetic sector) ICP-MS.

7.1.3 A time-of-flight mass spectrometer.

7.2 The ICP-MS ~~is interfaced to a glovebox. The~~ torch box, and the analyzer region of the mass spectrometer are glovebox ~~enclosed,~~ enclosed in an appropriate containment unit, since Pu containing materials come in direct contact with these sections of the instrument. Methods for enclosing plasma spectroscopic sources so that hazardous materials can be analyzed safely are described in ASTM STP 951.[4]

7.3 Graduated 14 mL disposable plastic round bottom tubes and caps or similar.

7.4 *Piston-operated Volumetric Pipettors and Dispensers,* ~~Electronic pipettes.~~ complying with the requirements of Specification E1154, for pipetting and dispensing of solutions, acids, and so forth.

8. Reagents and Materials

8.1 Ultra high purity acids (such as spectroscopic grade) shall be used for sample dissolution and calibration standards preparation unless otherwise noted.

8.2 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specification are available.[5]

8.3 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean laboratory accepted demineralized or deionized water as described by Type I of Specification D1193.

~~NOTE 1—The molarity of ultra high purity acids may vary from standard ACS specifications for concentrated acids.~~

NOTE 1—All reagents are prepared and stored in polytetrafluoroethylene (PTFE) containers.

8.4 *Hydrochloric Acid* (HCl, 11.3 M), concentrated HCl.

8.5 *Hydrochloric Acid* (HCl, 6 M)— ~~(HCl, 6 M),~~ Add 531 mL of concentrated HCl (11.3 M) to less than 450 mL of water and dilute to 1 liter with water.

8.6 *Nitric Acid* (HNO_3, 15.8 ~~M)—~~ M), concentrated nitric acid.

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[4] Edellson, M. ~~C., and Daniel, J. Leland,~~ C. and Leland, D. J., "Plasma Spectroscopy of the Analysis of Hazardous Materials: Design and Application of Enclosed Plasma Sources," *Conference Proceedings, ASTM 951*, ~~ASTM,~~ ASTM International, 1986.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.7 *Nitric ~~Acid,~~Acid* (1 % by Volume),~~1 % by volume— One~~ one volume of concentrated nitric acid (HNO_3, 15.8 M) brought to one hundred volumes with water.

8.8 *Hydrofluoric Acid* (HF, 28.3 M), concentrated HF.

8.9 *Nitric ~~Acid-Hydrofluoric~~Acid-hydrofluoric Acid Mixture,* 10 M HNO_3 / 0.03 M ~~HF— Add~~ HF—Add 1 mL of concentrated HF (28.3 M) to water; using a plastic pipette, while stirring, add 633 mL concentrated HNO_3 (15.8 M) and dilute to 1 L with water.

8.10 Stock solutions, traceable to a national standards organization, of multielement spike solutions are available from commercial vendors. The stock solutions of mulielement spike solutions can also be prepared in-house.

8.10.1 *Spike Solution 1 ~~(SS-1)—~~(SS-1),* contains 500 µg/mL of Al, As, Ba, Be, Bi, Cs, In, Li, Mg, Rb, Se, and Sr in 0.8 M HNO_3.

8.10.2 *Spike Solution 2 ~~(SS-2)—~~(SS-2),* contains 500 µg/mL of B, Ge, Hf, Mo, Nb, P, Re, Sb, Sn, Ta, Ti, W, and Zr in 0.8 M HNO_3.

8.10.3 *Spike Solution 3 ~~(SS-3)—~~(SS-3),* contains 500 µg/mL of Ag, Cd, Cr, Co, Cu, Mn, Ni, Pb, Tl, V, and Zn in 0.8 M HNO_3.

8.10.4 *Spike Solution 4 ~~(SS-4)—~~(SS-4),* contains 500 µg/mL of Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pr, Sm, Tb, Th, U, Yb, and Y in 0.8 M HNO_3.

8.10.5 *Spike Solution 5 ~~(SS-5)—~~(SS-5),* contains 500 µg/mL of Au, Ir, Pd, Pt, Ru, and Te in 0.8 M HNO_3.

8.11 Stock solutions, traceable to a national standards organization, of multielement impurity standards are available from commercial vendors. The stock solutions of multielement standards can also be prepared in-house.

8.11.1 *Inductively Coupled Plasma Calibration Standard-One ~~(ICPCS-1)~~(ICPCS-1),* contains 10 µg/mL of Ag, Al, As, Be, B, Cr, Co, Cu, Ge, Li, Mg, Mn, Ni, P, Pr, Rb, Se, Ti, V, and Zn in 10 % HNO_3 by volume and ~~< 0.5 %~~ <0.5 % HF by volume.

8.11.2 *Inductively Coupled Plasma Calibration Standard-Two ~~(ICPCS-2)~~(ICPCS-2),* contains 10 µg/mL of Ba, Bi, Cd, Cs, Ce, Dy, Er, Eu, Gd, Ho, La, Lu, Nd, Pb, Re, Sm, Sr, Tb, Tl, Th, U, Yb, and Y in 10 % HNO_3 by volume.

8.11.3 *Inductively Coupled Plasma Calibration Standard-Three ~~(ICPCS-3)~~(ICPCS-3),* contains 10 µg/mL of Au, Hf, In, Ir, Mo, Nb, Pd, Pt, Ru, Ta, Te, Sb, Sn, W, and Zr in 10 % HCl by volume, 2 % HF by volume and ~~< 0.5 %~~ <0.5 % HNO_3 by volume.

8.12 Stock solutions, traceable to a national standards organization, of single element internal standard are available from commercial vendors.

8.12.1 *Internal Standard-One (IS-1),* contains 10 µg/mL of Sc in 2 % HNO_3 by volume.

8.12.2 *Internal Standard-Two (IS-2),* contains 10 µg/mL of Rh in 2 % HCl by volume.

8.12.3 *Internal Standard-Three (IS-3),* contains 10 µg/mL of Tm in 2 % HNO_3 by volume.

9. Hazards

9.1 Plutonium bearing materials are radioactive and toxic. Adequate laboratory facilities, gloveboxes and fumehoods along with safe techniques, must be used in handling samples containing these materials. A detailed discussion of all the precautions necessary is beyond the scope of this test method; however, personnel who handle these materials should be familiar with such safe handling practices.

9.2 Extreme care should be exercised in using concentrated acids.

9.3 (**Warning—**~~H~~ydrofluoric acid is a highly corrosive and toxic acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid ~~is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature, and the duration of contact with the acid. Hydrofluoric acid~~ differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left ~~unattended. Due to the serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personal protective equipment to protect from skin and eye contact is essential. Acute exposure to HF can cause painful and severe burns upon skin contact that require special medical attention. Chronic or prolonged exposure to low levels on the skin may cause fluorosis.~~ untreated. Familiarization and compliance with the Safety Data Sheet is essential.)

10. Procedure

10.1 ~~Sample Dissolution~~*Sample Dissolution:*

10.1.1 Obtain an aliquot of Pu metal of approximately 0.25 g for every sample that will be analyzed. For other plutonium materials, obtain an aliquot whose Pu content is approximately 0.25 g.

10.1.2 ~~Obtain an aliquot of Pu metal of approximately 0.25 g for every sample that will be analyzed.~~ For one of the samples that will be analyzed obtain a second aliquot to be analyzed as a ~~"spiked sample." A "spiked sample"~~spiked sample. A spiked sample should be analyzed with each batch of samples.

10.1.3 Dissolve each sample of Pu material using an appropriate procedure from Practice C1168.

10.1.4 For the "spiked sample", add 0.1 mL each of the SS-1, SS-2, SS-3, SS-4, and SS-5 spike solutions.

10.1.5 The final concentration from steps 10.1.1 – 10.1.4 should be 0.25 g of Pu per 3.0 mL of solution.

10.1.6 Carry a blank, consisting of the solution used for dissolution with no sample added, through the same process used in 10.1.3.

10.1.7 Label (using the sample identification) a clean 14 mL plastic tube for each sample ~~to be~~dissolved in 10.1.1 – 10.1.6~~analyzed. Label two additional tubes as follows: 1) "spiked sample" (include the identification of the Pu sample that will be used) and 2) "blank". These tubes will be used as "dissolution tubes". Weigh and record the weight of each of the Pu metal aliquots to ±0.0001 g. Place the weighed Pu metal aliquot into the appropriately labeled plastic tube.~~ , including the blank and spiked sample.

~~9.1.3 Pipette 0.1 mL each of the SS-1, SS-2, SS-3, SS-4 and SS-5 into the tube labeled "SPIKED SAMPLE".~~

~~9.1.4 Pipette 1 mL of 6 M HCl into each of the "dissolution tubes", including the one labeled "blank" (see Practice C1168).~~

~~Note 3—Addition of the dissolution acid (6 M HCl) to the samples should be performed by slowly adding a few drops at a time and swirling the sample tube; if the reaction becomes too vigorous the solution may bubble out of the plastic tube. Do not cap and do not vigorously shake the tubes containing samples.~~

~~9.1.5 Add enough 10 M HNO_3 / 0.03 M HF so that the final volume is 3.0 mL and mix thoroughly.~~

~~9.1.6 Label another set of tubes as in 9.1.2.~~

10.1.8 ~~Pipette~~Pipet 0.3 mL from each ~~of the~~dissolved sample, ~~dissolution tubes~~spiked sample, ~~into each of the identically labeled tubes in step~~ and blank into the corresponding 14 mL plastic tube.~~9.1.6.~~

10.1.9 Dilute to 5 mL with 1 % HNO_3 by volume and mix thoroughly (the samples will be analyzed from further dilutions of this second dilution).

~~Note 4—The remainder of the sample in the dissolution tubes may be used for analysis using Test Method C1432 or can be disposed of using the appropriate laboratory protocol.~~

Note 2—The remainder of the sample in the dissolution tubes may be used for analysis using Test Method C1432 or can be disposed of using the appropriate laboratory protocol.

10.2 ~~Internal Standard and Calibration Standard Preparation~~*Internal Standard and Calibration Standard Preparation:*