Designation: C1637 – 13

Standard Test Method for the Determination of Impurities in Plutonium Metal: 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 covers the determination of 58 trace elements in plutonium (Pu) metal. 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 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, it is the responsibility of the user to evaluate the performance of other matrices.

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable

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

C759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions

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

3. Summary of Test Method

3.1 A sample of Pu metal is dissolved in a small volume of 6 M hydrochloric acid (HCl). Then, 10 M nitric acid (HNO_3)/ 0.03 M hydrofluoric acid (HF) is added to the dissolved Pu to oxidize the Pu to the Pu(IV) state. An aliquot of the original sample is 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.[3]

4. Significance and Use

4.1 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.

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

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

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

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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.

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

[A] Without Outlying Value

5. Interferences

5.1 Ions from doubly charged (2+) species are formed in the ICP-MS. The actinide related spectral interferences are from actinide 2+ and actinide-oxide 2+. The spectral interferences are observed at 120.5 and 127.5 atomic mass unit (amu), when analyzing plutonium-239.

5.2 Spectral interferences from the argon plasma and the acid used to transport the sample to the plasma. These spectral interferences occur between 12 and 80 amu.

5.3 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) are referenced to scandium. Rhodium is a reference for analytes at the middle of the mass range (76-138) and all analytes at the high end of the mass range are referenced to thulium (139-238 amu).

6. Apparatus

6.1 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.

6.2 The ICP-MS is interfaced to a glovebox. The torch box, and the analyzer region of the mass spectrometer are glovebox enclosed, 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]

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

6.4 Electronic pipettes.

7. Reagents and Materials

7.1 Ultra high purity acids shall be used for sample dissolution and calibration standards preparation unless otherwise noted.[5]

7.2 *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.[6]

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

[5] "The ULTREX II (J. T. Baker) and INSTRUMENT QUALITY (Seastar Chemicals) lines of ultra high purity acids have been found satisfactory for this purpose."

[6] *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.