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Designation: C1637 - 06 C1637 - 13

Standard Test Method for the Determination of Impurities in Plutonium Metal: Acid Digestion and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) Analysis¹

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 (HNO₃)/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 % HNO₃ 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

 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. Current edition approved Jan. 1, 2006Jan. 1, 2013. Published February 2006January 2013. Originally approved in 2006. Last previous edition approve in 2006 as D1637
 – 06. DOI: 10.1520/C1637-06.

 $^{^{2}}$ 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.

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.

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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.

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 μ g/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

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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

7.3 *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 2-All reagents are prepared and stored in polytetrafluoroethylene (PTFE) containers.

7.4 Hydrochloric Acid (HCl, 11.3 M), concentrated HCl.

7.5 *Hydrochloric Acid* (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.

7.6 Nitric Acid (HNO₃, 15.8 M)— concentrated nitric acid.

7.7 *Nitric Acid*, 1 % by volume— One volume of concentrated nitric acid (HNO₃, 15.8 M) brought to one hundred volumes with water.

7.8 Hydrofluoric Acid (HF, 28.3 M), concentrated HF.

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."

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.

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.

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

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^A Without Outlying Value

7.9 *Nitric Acid-Hydrofluoric Acid Mixture*, 10 M HNO₃ / 0.03 M HF— Add 1 mL of concentrated HF (28.3 M) to water; using a plastic pipette, while stirring, add 633 mL concentrated HNO₃ (15.8 M) and dilute to 1 L with water.

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