Designation: C 1432 – 03

Standard Test Method for Determination of Impurities in Plutonium: Acid Dissolution, Ion Exchange Matrix Separation, and Inductively Coupled Plasma-Atomic Emission Spectroscopic (ICP/AES) Analysis[1]

This standard is issued under the fixed designation C 1432; 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 25 elements in plutonium (Pu) materials. The Pu is dissolved in acid, the Pu matrix is separated from the target impurities by an ion exchange separation, and the concentrations of the impurities are determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES).

1.2 This test method is specific for the determination of impurities in 8 M HNO_3 solutions. Impurities in other plutonium materials, including plutonium oxide samples, may be determined if they are appropriately dissolved (see Practice C 1168) and converted to 8 M HNO_3 solutions.

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

2. Referenced Documents

2.1 *ASTM Standards:*

C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable[2]

C 758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal[2]

C 759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions[2]

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis[2]

D 1193 Specification for Reagent Water[3]

3. Summary of Test Method

3.1 A sample of plutonium metal is dissolved in a small volume of 6 M hydrochloric acid (HCl). Then, 10 M (HNO_3)/0.03 M hydrofluoric acid (HF) is added to the dissolved plutonium to oxidize the plutonium to the Pu (IV) state. The sample solution is loaded onto a nitrate anion exchange resin and eluted with 8 M HNO_3/0.006 M HF. The rinses contain the target metallic impurities and less than 15 µg/mL Pu. The plutonium is stripped from the anion exchange resin with 0.1 M HCl. The rinses containing the metallic impurities are analyzed by ICP-AES.

4. Significance and Use

4.1 This test method can be used on plutonium matrices in nitrate solutions.

4.2 This test method has been validated for all elements listed in Test Methods C 757 except sulfur (S) and tantalum (Ta).

4.3 This test method has been validated for all of the cation elements measured in Table 1. Phosphorus (P) requires a vacuum or an inert gas purged optical path instrument.

5. Interferences

5.1 Plutonium concentrations of less than 50 µg/mL in the final aqueous phase do not significantly affect the analytical results for most elements. Interference studies should be made to determine the degree of Pu and other elemental interferences on the target analytes; background and interelement corrections may be required.

6. Apparatus

6.1 An ICP-AES equipped with a Charge Injection Device (CID) detector or an ICP-AES with a spectral bandpass of 0.05 nm or less is required to provide the necessary spectral resolution.[4] The spectrometer may be either a simultaneous multielement or a sequential spectrometer. The spectrometer may be either an inert gas-path or vacuum instrument; the appropriate spectral lines should be selected for each specific instrument. Either an analog or digital readout system may be used.

[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] *Annual Book of ASTM Standards*, Vol 12.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] A Thermo Jarrell Ash PolyScan Iris spectrometer (Thermo Electron Spectroscopy, Franklin, MA), or an Applied Research Laboratories 3580 ICP-AES instrument (Dearborn, MI) have been found to be acceptable.

TABLE 1 Percent Recovery and Repeatability Standard Deviation for Sixteen Spiked Samples

Element	Wavelength/Order (nm)	Actual Conc (µg/mL)	Mean Conc (µg/mL)	Average (%R)	R.S.D. (%)
Aluminum	Al 396.152 {67}	2.5	2.4	95	6
Barium	Ba 455.403 {58}	2.5	2.4	95	5
Beryllium	Be 313.042 {84}	2.5	2.3	94	6
Boron	B 249.773 {106}	2.5	2.5	100	7
Cadmium	Cd 226.502 {116}	2.5	2.5	101	12
Calcium	Ca 396.847 {66}	2.5	2.6	104	20
Chromium	Cr 283.563 {93}	2.5	2.3	92	8
Cobalt	Co 228.616 {115}	2.5	2.5	101	6
Copper	Cu 324.754 {81}	2.5	2.4	97	6
Iron	Fe 259.940 {101}	2.5	2.5	101	12
Lead	Pb 220.353 {120}	2.5	3.1	122	12
Lithium	Li 670.784 {39}	2.5	2.2	87	6
Magnesium	Mg 280.270 {94}	2.5	2.4	95	6
Manganese	Mn 257.610 {102}	2.5	2.5	98	5
Molybdenum	Mo 202.030 {130}	2.5	2.6	103	10
Nickel	Ni 231.604 {114}	2.5	2.5	100	11
Silicon	Si 251.612 {104}	2.5	2.3	92	16
Sodium	Na 588.995 {45}	25.0	24.7	97	16
Strontium	Sr 421.552 {62}	2.5	2.4	95	5
Tin	Sn 189.989 {139}	2.5	2.7	109	19
Titanium	Ti 334.941 {79}	2.5	2.5	102	8
Tungsten	W 207.911 {127}	2.5	2.5	99	11
Vanadium	V 292.402 {90}	2.5	2.0	82	7
Zinc	Zn 213.856 {123}	2.5	2.5	100	8
Zirconium	Zr 339.198 {78}	2.5	2.5	101	10

6.2 The ICP-AES is interfaced to a glovebox. The torch box is glovebox enclosed, since plutonium containing materials come in direct contact with the torch. This setup is described in ASTM STP 951.[5]

6.3 Vacuum manifold set at approximately 23 cm Hg (9 in. Hg) is optional.[6] A gravity system is also acceptable.

6.4 15 mL plastic disposable ion exchange columns.[7]

6.5 50 mL plastic vials.

6.6 Plastic micro and macro pipettes.

6.7 1000 mL plastic volumetric flasks.

7. Reagents and Materials

7.1 *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 specifications are available.[8] Other grades could be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean laboratory accepted demineralized or deionized water as described by Type 1 of Specification D 1193.

7.3 Ultra high purity acids shall be used for sample dissolution and calibration standards preparation unless otherwise noted.

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 ultra high purity[9] HCl.

7.5 *Hydrochloric Acid* (HCl, 6 M)—Add 531 mL of concentrated ultra high purity HCl (11.3 M) to less than 450 mL of water and dilute to 1 L with water.

7.6 *Hydrochloric Acid* (HCl, 0.1 M)—Add 8.8 mL of concentrated ultra high purity HCl (11.3 M) to water, while stirring, and dilute to 1 L with water. (Reagent grade HCl can be used in preparing this reagent.)

7.7 *Hydrofluoric Acid* (HF, 28.3 M), concentrated ultra high purity[9] HF.

7.8 *Nitric Acid* (HNO_3, 15.8 M), concentrated ultra high purity[9] nitric acid.

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

7.10 *Nitric Acid-Hydrofluoric Acid Mixture*, 8 M HNO_3/0.006 M HF—Add 0.21 mL of concentrated ultra high purity HF (28.3 M) to water; using a plastic pipette, while stirring, add 506 mL of concentrated ultra high purity HNO_3 (15.8 M) and dilute to 1 L with water.

[5] 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 STP 951*, ASTM, 1986.

[6] Eichrom Technologies Vacuum Box System (Part # AC-24-BOX), Eichrom Technologies Inc., Darien. IL, has been found to be acceptable.

[7] Ion exchange columns from either Applied Separation or Bio-Rad Inc., have been found to be acceptable.

[8] *Reagent Chemicals, American Chemical Society Specification*, Am. Chem. Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Reagents Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., New York, NY and the *United States Pharmacopeia*.

[9] The Ultrex (J. T. Baker, Inc.) and Seastar brands of ultra high purity acids have been found to be acceptable.