Designation: C 1432 – 99

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). The elements determined are listed in Table 2.

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

1.3 Plutonium bearing materials are radioactive and toxic. Adequate laboratory facilities, glove boxes, and fume hoods, 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.

1.4 *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 method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use of this standard.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powers and Pellets[2]

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]

TABLE 1 ICP-AES Operating Conditions[A]

Parameter	Value
Forward rf power	1.4 kW
Reflected rf power	<10 W
Outer argon flow	15 L/min
Auxiliary argon flow	0.8 L/min
Carrier argon flow	0.7 L/min
Observation height	15 mm above load coil
Nebulizer	Cross flow type
Solution uptake rate	1.4 mL/min

[A] These conditions are typical for an ARL #3580.

TABLE 2 Repeatability Standard Deviation for Three Spike Recovery Experiments

Element	Wavelength (nm)	Actual Conc (µg/g)	Mean Conc (µg/g)	Experimental Determinations Avg % Recovery	RSD %
Aluminum	308.22	6.0	6.9	114	2.7
Barium	493.41	3.0	3.3	109	1.7
Beryllium	313.04	1.5	1.7	112	1.7
Cadmium	226.5	6.0	6.5	109	4.6
Calcium	393.37	3.0	3.3	109	1.8
Chromium	267.72	6.0	7.0	117	5.1
Cobalt	237.86	6.0	6.9	115	1.6
Copper	324.75	3.0	3.5	118	2.0
Hafnium	232.25	6.0	6.4	106	1.1
Iron	259.94	6.0	6.8	113	1.6
Lead	220.35	6.0	6.9	114	3.6
Lithium	670.78	3.0	3.4	112	1.8
Magnesium	279.55	3.0	3.5	116	1.8
Manganese	257.61	3.0	3.4	112	1.8
Molybdenum	202.03	6.0	4.5	75	22
Nickel	231.6	6.0	6.8	114	1.6
Niobium	316.34	6.0	5.9	98.9	2.2
Phosphorus	178.29	6.0	6.4	106	2.3
Potassium	766.49	30.0	33	110	1.8
Strontium	421.55	3.0	3.3	109	1.7
Titanium	368.52	3.0	3.3	111	1.1
Vanadium	292.4	6.0	6.8	113	1.9
Yttrium	371.03	3.0	3.3	109	1.7
Zinc	206.2	3.0	2.3	75.4	7.5
Zirconium	349.62	6.0	6.5	109	1.5

[1] This guide is under the jurisdiction of ASTM Committee C-26 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.

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 Pu metal is dissolved in a small volume of 6 M hydrochloric acid (HCl). Then, 1 mL of 10 M (HNO_3)/0.2 M hydrofluoric acid (HF) is added to the dissolved Pu to oxidize the Pu to the Pu (IV) state. The sample solution is loaded onto a nitrate anion exchange resin and eluted with 8 M HNO_3/0.2 M HF. The rinses contain the target metallic impurities and less than 15 µg/mL Pu. The Pu 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 measures all elements listed in Specification C 757, except sulfur (S) and tantalum (Ta).

4.2 This test method measures all of the cation elements measured in Test Methods C 697, except silver (Ag), gold (Au), and bismuth (Bi). Phosphorus (P) requires a vacuum 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 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.

6.2 The ICP-AES is interfaced to a glovebox. The torchbox is glovebox enclosed since Pu containing materials come in direct contact with the torch. The torchbox offers several safety features, such as a shielded window for observing the plasma, which allows the operator to view the plasma without risking damage to the eyes. The torchbox is equipped with an interlock that shuts off high voltage power to the torchbox when the torchbox door is open. The interlock prevents the operator from being exposed to high voltages during routine cleaning. This setup is described in ASTM STP 951.[5]

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

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

6.5 30-mL plastic vials.

6.6 Plastic micro and macro pipettes.

6.7 A 500-mL fritted column.

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 where such specifications are available.[8] Other grades should 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 describe 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—All reagents are prepared and stored in polytetrafluoroethylene (PTFE) containers.

7.4 *Hydrochloric Acid* ((HCl) (sp gr 1.18)), concentrated ultra high purity[9] HCl.

7.5 *Hydrochloric Acid* (HCl, 6 M)—Add 500 mL of concentrated ultra high purity HCl (sp gr 1.18) to less than 500 mL of water and dilute to 1 L with water.

7.6 *Hydrochloric Acid* (HCl, 0.1 M)—Add 8.3 mL of concentrated ultra high purity HCl (sp gr 1.18) 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) (sp gr 1.15)), concentrated ultra high purity[9] HF.

7.8 Nitric Acid ((HNO_3) (sp gr 1.42)), concentrated ultra high purity[9] HNO_3.

7.9 *Nitric Acid-Hydrofluoric Acid Mixture*, 10 M HNO_3/0.2 M HF—Add 7.2 mL of concentrated ultra high purity HF (sp gr 1.15) to water, using a plastic pipet, while stirring; add 637-mL concentrated ultra high purity HNO_3 (sp gr 1.42); and dilute to 1 L with water.

7.10 *Nitric Acid-Hydrofluoric Acid Mixture*, 8 M HNO_3/0.2 M HF—Add 7.2 mL of concentrated ultra high purity HF (sp gr 1.15) to water, using a plastic pipet, while stirring; add 510 mL of concentrated ultra high purity HNO_3 (sp gr 1.42); and

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

[4] An Applied Research Laboratories 3580 ICP-AES instrument (Fisons Instruments, Dearborn, MI) has been found to be acceptable. The ARL 3580 is a combination Pashen-Runge type spectrometer containing a 58 channel simultaneous spectrometer and a sequential spectrometer, both with a 1-m focal length and capable of operating in the 165 to 800-nm range.

[5] Edelson, 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] Speed Mate 10 Vacuum Extraction System, Applied Separations, Bethlehem, PA, 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 Specifications*, American Chemical Society, Washington, D.C. 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.

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