



Standard Test Method for Plutonium Assay by Plutonium (III) Diode Array Spectrophotometry¹

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1. Scope

1.1 This test method describes the determination of total plutonium as plutonium(III) in nitrate and chloride solutions. The technique is applicable to solutions of plutonium dioxide powders and pellets (Test Methods C 697), nuclear grade mixed oxides (Test Methods C 698), plutonium metal (Test Methods C 758), and plutonium nitrate solutions (Test Methods C 759). Solid samples are dissolved using the appropriate dissolution techniques described in Practice C 1168. The use of this technique for other plutonium-bearing materials has been reported (1-5), but final determination of applicability must be made by the user. The applicable concentration range for plutonium sample solutions is 10–200 g Pu/L.²

1.2 *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 697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets³
- C 698 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Mixed Oxides ((U, Pu)O₂)³
- C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable³
- C 758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal³
- C 759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of

- Nuclear-Grade Plutonium Nitrate Solutions³
- C 833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets³
- C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis³

3. Summary of Method

3.1 In a diode array spectrophotometric measurement, as in a conventional spectrophotometric measurement, the substance being determined absorbs light at frequencies characteristic of that substance. The amount of light absorbed at each wavelength is directly proportional to the concentration of the species of interest. The absorption is a function of the oxidation state and the complexation obtained in the solution matrix selected for measurement. Beer's Law permits quantifying the species of interest relative to a traceable standard when both solutions are measured under the same conditions. The array of photosensitive diodes permits the (virtually) simultaneous collection of spectral information over the entire range of the instrument, for example, 190–820 nm (or any selected portion of the range). An entire absorption spectrum can be obtained in 0.1 s; however, optimum precision is obtained from multiple spectra collected over a 4-s period.

3.2 Reduction to plutonium(III) is accomplished by the addition of a measured quantity of reductant solution to the sample aliquant.

3.2.1 For nitrate solutions, ferrous sulfamate is the recommended reductant. Aliquants (1 mL or less) of the sample solution are diluted with 10 mL of a ferrous reductant/matrix solution to 1 g Pu/L, and measured.

3.2.2 For chloride solutions, ascorbic acid is the recommended reductant. Aliquants of the sample solution, each containing 50–100 mg of plutonium, are diluted with 2 mL of zirconium solution to complex fluoride ions, 2 mL ascorbic acid reductant solution, and 1.0 M HCl to a total volume of 25 mL, yielding 2–4 g Pu/L solutions for measurement.

3.3 Plutonium concentration is determined from light absorption measurements taken on the sample solution in the blue-green region from 516 to 640 nm where a broad doublet band is observed. Spectral quantifying software capable of fitting the sample spectrum with spectral information from standard solutions is used to calculate the plutonium concentration. Both commercially available (6) and custom-designed

¹ This test method 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 For solid samples, select the sample size and dissolved solution weight to yield sample solutions in the 10–30 g Pu/L range. With special preparation and spectral analysis techniques, the method has been applied to nitrate solutions in the 0.1–10 g Pu/L range.

³ *Annual Book of ASTM Standards*, Vol 12.01.

(7-12) spectral fitting software have been developed which may be used for plutonium measurements. The users of this procedure are responsible for selecting or customizing, or both, the spectral fitting (and instrument control) software that best meets their individual measurement methodology and needs. Software selection will dictate many of the procedural specifics not included in this procedure. This procedure is intended to address key measurement requirements and to allow users discretion in establishing appropriate procedural details and technique variations. The software package selected should include a feature that indicates the quality of spectral fit, thereby providing information on the measurement reliability and the presence of interferences that absorb light or otherwise alter the plutonium(III) spectrum without requiring supplemental measurements.

4. Significance and Use

4.1 This test method is designed to determine whether a given material meets the purchaser's specification for plutonium content.

5. Interferences

5.1 Materials meeting the applicable material specifications of the ASTM standard for which this procedure was developed, when dissolved and diluted without introduction of interfering contaminants as described in Practice C 1168, will contain no interfering elements or species.

NOTE 1—Fluoride, if present, would interfere if the zirconium, routinely added to the sample solution aliquant for the chloride matrix, were omitted from the procedure. Zirconium may be added to the nitrate matrix. Ferrous-Reductant Solution to handle fluorides if present. Zirconium, when used, should be added to all samples, blanks, and standards to obtain a consistent matrix. Refer to Specifications C 833 and C 757.

5.2 Interferences are caused by: 1) materials that absorb light in the region of the plutonium absorption, 2) undissolved solids that cause light scattering, 3) strong oxidizing or complexing agents that prevent complete reduction of the plutonium to the plutonium(III) oxidation state, and 4) anions that shift the spectrum.

5.2.1 Absorption of light in the region of interest by another species is a potential interference. Identification of potentially interfering species and inclusion of their spectra in the spectral curve fitting process will significantly reduce their effect. At a minimum, sample measurements should be flagged when the higher than normal fitting error occurs, resulting from the presence of unidentified absorbing species. Enhancement of the spectral curve fitting capabilities of the DAS can be achieved by taking double derivatives of the spectrum collected. The spectral curve fitting software of the DAS is then used to quantitate the mathematically manipulated spectrum.

NOTE 2—Care must be taken in the choice of the preprocessing methods (derivatives, mean centering, autoscaling and/or channel selection) as these may affect the robustness of the final model, particularly with regard to unknown interferences. Use of residual analysis will not always detect unknown interferences and results will vary depending on the preprocessing methods and models employed.

5.2.2 This spectrophotometric assay method should not be used on turbid (cloudy) solutions or solutions containing

undissolved material. In addition to visual or turbidity meter measurements, or both, the presence of undissolved solids may be identified by the resulting shifts in the spectral baseline and by elevated spectral fitting errors.

NOTE 3—Plutonium oxides, mixed oxides, and plutonium metals meeting the material specifications for which this test method is intended, will dissolve when procedures in Practice C 1168 are followed. Failure to achieve dissolution is an indication that the material does not meet the specifications, and the application of this test method for plutonium assay must be verified by the user. The user and customer are cautioned: when undissolved solids that persist after exhaustive dissolution efforts are to be removed by filtration through filter paper or other inert material of appropriate porosity, the subsequent plutonium assay measurements require close scrutiny. While filtration of undissolved solids may permit the reliable measurement of the concentration of plutonium in the filtrate, the resulting analysis may not be representative of their source material. Solids may indicate incomplete dissolution of the plutonium in the sample material, not necessarily a plutonium-free refractory residue. When this technique is utilized in support of reprocessing operations, process solutions containing solids may be an indication of incomplete dissolution of the plutonium-bearing material being processed or of an error in process operations. In addition to process control considerations, the undissolved solids may represent accountability and criticality control problems.

5.2.3 Strong oxidizing agents and complexing agents in sufficient concentration to prevent complete reduction typically are not present in plutonium nitrate samples. Appreciable concentrations of fluoride and sulfate anions have been found to interfere. The concentration of hydrofluoric acid, added to catalyze dissolution of oxides, may be removed by evaporation prior to measurement to ensure that the zirconium effectively complexes the traces of fluoride ion. Changes in the plutonium spectrum from incomplete reduction due to oxidizing agents and shifts in the spectrum due to complexing agents are also indicated by increases in the spectral curve fitting error.

5.2.4 Anion identity and concentration will shift the location and alter the shape of the absorption curve. The system calibration must include the anion shift effect by encompassing the expected range of anion identities and concentrations or by using appropriate spectral fitting features that identify and correct for the effect.

5.3 A study was conducted at the Los Alamos National Laboratory to determine the immunity of the Pu(III) spectrophotometric assay method to a diverse species of potential interferences. The elements studied were element numbers 1, 9, 11–13, 17, 19, 22–31, 35, 42, 44–46, 48, 50, 53, 57, 58, 60, 62, 73, 74, 76, 77, 79, 83, 90, 92, 93, and 95. Potential interference from nitrate, phosphate, sulfate, and oxalic acid is also documented (13).

6. Apparatus

6.1 *Diode Array Spectrophotometer (DAS)*—wavelength range 190–820 nm; wavelength accuracy ± 2 nm; wavelength reproducibility ± 0.05 nm; full dynamic range 0.0022 to 3.3; photometric accuracy at 1 AU with a NBS 931 filter at 512 nm is ± 0.005 AU; baseline flatness < 0.0013 AU; noise at 500 nm is 0.0002 AU RMS; stray light measured with a Hoya 056 filter

at 220 nm <0.05 %;⁴

6.2 *Analytical Balance*—readability of 0.1 mg; linearity 0.1 mg over any 10 g range and 0.2 mg over 160 g full scale.

6.3 *Solution Density Meter*—readability of 0.1 mg/mL; precision of 0.3 mg/mL; linearity and accuracy 0.5 mg/mL over the range 0 to 2.0 g/mL.

6.4 *Adjustable, Fixed-Volume Pipetters*—calibrated to deliver the desired range of volumes for sample and matrix-reductant solutions.

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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ Other grades may 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 distilled or deionized water.

7.3 *Ascorbic Acid-Reductant Solution* (C₆H₈O₆, aminoguanidine bicarbonate (CH₆N₄H₂CO₃), 0.4 M in each reagent)—Prepare fresh daily by dissolving 7 g of ascorbic acid and 5.5 g aminoguanidine bicarbonate in 80 mL of 1 M HCl, then dilute to a final volume of 100-mL 1 M HCl.

NOTE 4—The ascorbic acid is stabilized by the addition of aminoguanidine (Guanylhydrazine:HN:C(NH₂)NHNH₂). The stabilized reductant solution has been found to be effective when ascorbic acid stability problems are encountered.

7.4 *Ferrous-Reductant Solution* (ferrous sulfamate, 0.05 M; sulfamic acid, 0.25 M; nitric acid, 1.0 M)—Prepare fresh weekly by adding 12 mL of freshly prepared ferrous sulfamate (2 M) to 90 mL of sulfamic acid (1.5 M). Stir, then add 175 mL of nitric acid (3.0 M) and dilute to 500 mL with water.

7.5 *Ferrous Sulfamate* (Fe(NH₂SO₃)₂, 2.0 M)—Prepare fresh for the preparation of the ferrous-reductant solution. Add 220 g of solid sulfamic acid to 450 mL of water, stir, and heat at 70–80°C until dissolved. Continue stirring and heating, while adding approximately 0.5-g portions of iron metal powder (Fe⁰) until 56 g of iron have been dissolved in the heated sulfamic acid. Filter the solution while hot; allow to cool; then dilute to a final volume of 50 mL.

NOTE 5—The dissolution of the sulfamic acid need not be quantitative before beginning the addition of the iron powder. Excessive heating

⁴ The optical specifications listed in 6.1 have been found to provide satisfactory results. In addition to these specifications, an acceptable spectrometer system should also provide multicomponent spectral quantitating software, computer control, and fiber optic capabilities. systems which meet these specifications include, but are not limited to, the Hewlett Packard 8451A and 8452A, the Agilent 8453, and the TIDAS II. Fiber Optic Coupler, Fibers, and 1, 3, and 4-cm Flow Cell/Pump System have been developed and reported on (2, 3, 5). Specifications listed are those of the HP-8451A and the HP-8452A. Although the wavelength range for plutonium requires only a fraction of the 190–820 nm range specified (plutonium absorption spectrum is measured over the 520–634 nm region) spectrophotometers with significantly smaller range would be of little general use to the purchaser.

⁵ “Reagent Chemicals, American Chemical Society Specifications,” *American Chemical Society*, Washington, D.C.

beyond the time required to achieve the dissolution of the sulfamic acid/iron powder or at temperatures above 80°C will cause excessive decomposition of the sulfamic acid

7.6 *Hydrochloric Acid* (HCl, 12 M)—Concentrated, sp gr 1.19.

7.7 *Hydrochloric Acid* (HCl, 1.0 M)—Add 84 mL of hydrochloric acid (sp gr 1.19) to approximately 500 mL of water. Stir, then dilute to a final volume of 1 L.

7.8 *Nitric Acid* (HNO₃, 15.8 M)—Concentrated, sp gr 1.42.

7.9 *Nitric Acid* (1.0 M)—Add 63 mL of nitric acid (sp gr 1.42) to approximately 500 mL of water. Stir, then dilute to a final volume of 1 L.

7.10 *Nitric Acid* (3.0 M)—Add 190 mL of nitric acid (sp gr 1.42) to approximately 500 mL of water. Stir, then dilute to a final volume of 1 L.

7.11 *Plutonium Standard Solutions*—Prepare standards traceable to a national measurement system, which cover the range of concentrations over which sample measurements will be performed.

7.12 *Sulfamic Acid* (NH₂SO₃H, 1.5 M)—Dissolve 145 g of solid sulfamic acid in 900 mL of water with stirring. Filter, then dilute with water to a final volume of 1 L.

7.13 *Zirconium Reagent* (ZrOCl₂·8H₂O, 0.75 M)—Dissolve 120.5 g zirconium chloride octahydrate in 450 mL of 1.0 M HCl; dilute to a final volume of 500 mL of 1.0 M HCl.

8. Calibration and Standardization of Instrument

8.1 Calibrate the system prior to each use. To calibrate, prepare several aliquants of at least two different plutonium standard solutions in the same concentration range as the samples to be measured in accordance with the preparation procedure described in Section 9. At least one of the standard solutions prepared will be used independently to ensure the accuracy of the calibration and should not be used in generating the calibration curve.

8.2 Following spectral referencing, measure each of the aliquants from one or more of the standard solutions. Quantitate each of the resulting spectra using appropriate software fitting routines; establish the calibration curve; and test the curve to ensure that all results and parameters meet the control limits previously established by the user.

8.3 Depending upon the choice of spectral curve fitting technique, blank solutions may be required for calibration and subsequent sample measurements.

9. Procedure

9.1 Sample preparation:

9.1.1 Dissolve all solid samples in accordance with Practices C 1168 or equivalent practices.

9.1.2 Dissolve metal with hydrochloric acid.

9.1.3 Dissolve all oxides by acid digestion in either sealed reflux tubes or in beakers. If hydrofluoric acid was added for dissolution, convert the dissolved sample solution to either a nitrate or chloride matrix by evaporation, ensuring removal of hydrofluoric acid.

9.1.4 Plutonium nitrate sample solutions are measured directly.

9.1.5 Inspect all solutions to be measured for unusual appearance or properties.

9.2 Measurement of nitrate/sulfamate matrix samples and standards.

9.2.1 Take aliquants of the sample solution, each containing 7–13 mg of plutonium by weight. Add a 10-mL portion of the ferrous-reductant solution by weight to each aliquant. Seal each container and agitate to ensure complete mixing. Table 1 prescribes the recommended dilutions to achieve 1 g Pu/L.

NOTE 6—For this method variation, the sample aliquant size should be selected to achieve less than 2 g Pu/L and to achieve less than 2 M acid and nitrate concentrations with a minimum dilution factor of ten. When the final plutonium concentration is less than 2 g/L, the reduction of plutonium with excess ferrous ion in dilute nitric/sulfamic acid results in complete and rapid reduction. With proper care, technique, and equipment calibration, the dilution of the sample may be performed volumetrically.

9.2.2 If the spectrophotometer has not been referenced recently (within an hour), re-reference using the blank solution described in 8.3. confirm that the light intensity at this stage is approximately 80–80% of the dynamic range of the detector response.

NOTE 7—Care must be taken to ensure that the measurement cell does not contain any of the species of interest. Any analyte present in the cell during referencing will act as a blank or offset for subsequently collected spectra.

9.2.3 Measure the aliquants from the sample solutions and the aliquants from the standard solutions not used for instrument calibration in 8.2 as follows:

9.2.3.1 Rinse the measurement flow cell with the solution to be measured.

9.2.3.2 Collect and store a spectrum from each aliquant of the plutonium standard and sample solutions.

9.2.3.3 Compute the concentration of each of the solutions measured as described in Section 10. Always use the volumetric dilution factor, which is calculated from gravimetric data using the densities of the sample and the matrix-reductant solutions, to obtain the plutonium concentration in the sample solution.

9.2.4 Evaluate each of the resulting spectra using the appropriate software fitting routine and the calibration curve obtained in 8.2. Examine the results from the standards and the samples to ensure that they meet the control limits previously established by the user.

9.3 Measurement of chloride matrix samples and standards:

9.3.1 Take aliquants of the sample solution, each containing 50–100 mg of plutonium by weight. Add two mL of zirconium

solution to each aliquant and mix. Add 2 mL ascorbic acid-reductant solution and remix. Dilute the solution with 1.0 M HCl to 25 mL in a tared flask to yield a 2–4 g Pu/L solution for measurement. Measure the total weight of the solution in the tared flask. Stopper and mix the solution, then measure the solution density to compute the volume. With proper care, technique, and equipment calibration, the dilution of the sample may be performed volumetrically. The recommended dilutions to achieve 2–4 g Pu/L are prescribed in Table 2.

9.3.2 Process one blank with each series of reference materials aliquants or group of samples measured.

9.3.3 Prepare and reference the spectrophotometer for use as recommended by the manufacturer.

NOTE 8—Care must be taken to ensure that the measurement cell does not contain any of the species of interest. Any analyte present in the cell during referencing will act as a blank or offset for subsequently collected spectra.

9.3.4 Measure the aliquants from the sample solutions and the aliquants from the standard solutions not used for instrument calibration in 8.2 as follows:

9.3.4.1 Rinse the measurement flow cell with the solution to be measured.

9.3.4.2 Collect and store a spectrum from each aliquant of the plutonium standard and sample solutions.

9.3.4.3 Compute the concentration of each of the solutions measured as described in Section 10. Always use the volumetric dilution factor, which is calculated from gravimetric data using the densities of the sample and the matrix-reductant solutions to obtain the concentration of plutonium in the sample solution.

9.3.5 Evaluate each of the resulting spectra using the appropriate software fitting routine and the calibration curve obtained in 8.2. Examine the results from the standards and the samples to ensure that they meet the control limits previously established by the user.

10. Calculations

10.1 Calculate the plutonium concentration of the dissolved sample solution as follows:⁶

$$g \text{ Pu/L} = R \times C \times D_v \quad (1)$$

⁶ Depending upon the software selected, the product of $R \times C$ may be generated automatically, because the system calibration is a part of the spectral quantitating system.

TABLE 1 Recommended Dilution of the Sample Solution (Nitrate Matrix) Based Upon the Estimated Plutonium Concentration

Estimated Sample Concentration Range, g Pu/L	Recommended Dilution		Resulting Measurement Concentration Range, g Pu/L
	Sample Volume, μL	Matrix Volume, mL	
10–12	1000	10	0.90–1.09
13–15	800	10	0.89–1.11
16–21	600	10	0.91–1.19
22–29	400	10	0.81–1.12
30–41	300	10	0.87–1.19
42–58	200	10	0.82–1.14
59–81	150	10	0.87–1.20
82–134	100	10	0.81–1.33
135–200	50	10	0.67–1.00

TABLE 2 Recommended Dilution of the Sample Solution (Chloride Matrix) Based Upon the Estimated Plutonium Concentration

Estimated Sample Concentration Range, g Pu/L	Recommended Dilution		Resulting Measurement Concentration Range, g Pu/L
	Sample Volume, mL	Final Volume, mL	
10–20	5	25	2.0–4.0
21–30	3	25	2.5–3.6
31–40	2	25	2.5–3.2

where:

R = result obtained from the spectral fitting software¹³ (prior to application of the system calibration). If a significant blank was observed from 9.4.3.2, the result must be corrected for the measured blank.

C = calibration factor for the calibration curve¹³ obtained in 8.2.

D_v = volumetric dilution factor, V_f/V_a .

V_f = final volume of the diluted sample aliquant. If the final solution was taken gravimetrically, then the volume must be computed using the solution density of the final solution (volume = weight/density). For the nitrate method variation where the aliquant and matrix-reductant solutions are each added by separate weight additions, $V_f = V_a + V_r$.

V_a = volume of aliquant taken for measurement. If the aliquant was taken gravimetrically, then the volume must be computed using the solution density of the sample or standard (volume = weight/density).

V_r = volume of reductant-matrix solution used to dilute the aliquant taken. If the reductant solution was taken gravimetrically, then the volume must be computed using the solution density of the reductant-matrix solution (volume = weight/density).

10.2 For solid samples, calculate the weight percent plutonium in the material as follows:

$$\text{Wt. \% Pu} = (\text{g Pu/L}) \times 100 / (W_s/V_s) \quad (2)$$

where:

W_s = weight of solid sample taken. Buoyancy corrections should be made as appropriate for the type of material being sampled.

V_s = volume of the dissolved sample solution.

11. Precision and Bias

11.1 The nitrate/sulfamate matrix method evaluation was performed as follows: The spectrophotometric determination of plutonium(III) was calibrated using standard solutions traceable to the national measurement system through NBL-CRM 126 Plutonium Metal and the Faraday (using controlled-potential coulometry). With gravimetric preparation, an estimated precision of 0.2 % relative standard deviation and a

mean recovery percent of 99.9 % (versus the consensus values for the materials) was obtained from measurements performed at the Savannah River Site Analytical Laboratories on Rocky Flats Plutonium Metal Sample Exchange Materials (Lots A84, B84, C84, and A86) during six of the quarterly exchanges. These lots of plutonium-bearing materials vary in their impurity content. These materials were selected because their composition is representative of the range of materials for which this test method is intended. These data indicated that no statistically significant bias was detected when the measurement precision, the uncertainty in the calibration standard, and the uncertainty in the consensus values of the plutonium metals were considered.⁷ Spectra collected during the evaluation of this test method were obtained using an HP-8451A Diode Array Spectrophotometer. Spectral quantitating was performed using commercially available Hewlett-Packard Spectral Analysis Software in multicomponent/second derivative mode.

11.2 The chloride matrix method evaluation was performed as follows: The spectrophotometric determination of plutonium(III) was calibrated using NBL-CRM 126 Plutonium Metal. The precision and bias of the method were determined from 45 assays performed at the Los Alamos National Laboratory over 18 months on a control standard of PuO₂. This control standard was also assayed 140 times during the same time period using controlled-potential coulometry. With volumetric preparation, the precision of the spectrophotometric method, based on the 45 assays, was 0.15 % relative standard deviation. No statistically significant bias was found relative to controlled-potential coulometry (Coulometry gave a value of 88.05 % Pu).⁷ Spectra collected during the evaluation of this method were obtained using an HP-8452A Diode Array Spectrophotometer. Spectral quantitating was performed using LANL Spectral Analysis Software (3).

11.3 This test method has been used only on plutonium oxide materials greater than 86.0% and high purity plutonium metal standards. Its use on less pure plutonium samples may degrade the accuracy and precision of the method.

12. Keywords

12.1 diode array spectrophotometry; plutonium assay; spectrophotometry (of plutonium (III))

⁷ These data are on file with ASTM.

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