



Standard Practice for Removal of Uranium or Plutonium, or both, for Impurity Assay in Uranium or Plutonium Materials¹

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1. Scope

1.1 This practice covers instructions for using an extraction chromatography column method for the removal of plutonium or uranium, or both, from liquid or digested oxides or metals prior to impurity measurements. Quantification of impurities can be made by techniques such as inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma atomic emission spectrometry (ICP-AES) or atomic absorption spectrometry (AAS.)

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:²

- C 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder
- C 757 Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable
- C 776 Specification for Sintered Uranium Dioxide Pellets
- C 787 Specification for Uranium Hexafluoride for Enrichment
- C 788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals
- C 996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ²³⁵U
- C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C 1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry

C 1347 Practice for Preparation and Dissolution of Uranium Materials for Analysis

C 1432 Test Method for Determination of Impurities in Plutonium: Acid Dissolution, Ion Exchange Matrix Separation, and Inductively Coupled Plasma-Atomic Emission Spectroscopic (ICP/AES) Analysis

C 1517 Test Method for Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy

D 1193 Specification for Reagent Water

3. Summary of Practice

3.1 An aliquot of liquid sample or dissolved solid sample is adjusted as needed to 8M nitric acid for plutonium/uranium removal using extraction chromatography. Uranium and plutonium are retained on the resin and trace impurities are collected in the column effluent. The impurities can be measured by a variety of techniques.

4. Significance and Use

4.1 This practice can be used to separate uranium or plutonium, or both, prior to the impurity analysis by various techniques. The removal of uranium and plutonium prior to quantification can improve the detection limits by minimizing the signal suppression caused by uranium or plutonium when using ICP techniques. Detection limits of ~1–10 part-per-billion (PPB) may be obtainable by matrix removal. Also, removal of the uranium and plutonium may allow the impurities analysis to be performed on a non-glove box enclosed instrument.

4.2 Other test methods exist to determine impurities in uranium or plutonium. Test Method C 1517 is able to determine many impurities in uranium at detection levels of ~1–10 part-per-million (ppm) by DC-Arc Spectrometry. Test Method C 1287 is able to determine impurities in uranium at detection levels of ~100 ppb by ICP-MS. Test Method C 1432 provides

¹ This practice 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.

an alternative technique to remove plutonium by ion exchange prior to analysis of the impurities by ICP-AES.

4.3 This practice can be used to demonstrate compliance with nuclear fuel specifications, for example, Specifications [C 753](#), [C 757](#), [C 776](#), [C 787](#), [C 788](#), and [C 996](#).

5. Interferences

5.1 Zirconium, hafnium, niobium, and tantalum are retained on the diamyl, amyolphosphonate resin unless hydrofluoric acid is added to the nitric acid load/rinse solution. The addition of hydrofluoric acid to recover zirconium, hafnium, niobium, and tantalum reduces uranium and plutonium retention. For this reason, hydrofluoric acid levels must be minimized (typically <0.05M HF) in the load/rinse solution to prevent uranium or plutonium, or both, from eluting from the column into the trace metal fraction.

6. Apparatus

6.1 *Large Columns*, >13 mL capacity (inner diameter = 1.5 cm has been found acceptable) and reservoirs.

6.2 *Plastic Collection Tubes*, 50 mL.

6.3 *Column Rack*, used for gravity flow systems.

6.4 *Polyethylene Frits* for columns, 20 μm .

6.5 *Vacuum Box*—The use of a vacuum-assisted flow system permits the use of higher eluent flow rates. Gravity flow systems may be used instead.

7. Reagents

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.³ 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. High purity acids may be used to reduce reagent blanks and to achieve lower detection limits.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification [D 1193](#) as Type I.

7.3 *Hydrofluoric Acid* (28 M)—Concentrated hydrofluoric acid (sp gr 1.2).

7.4 *Nitric Acid* (16 M)—Concentrated HNO_3 (sp gr 1.42).

7.5 *Nitric Acid Solution* (8 M)—Add 500 mL of concentrated nitric acid (sp gr 1.42) to 300 mL of water and dilute to 1 L with water.

7.6 *Nitric Acid* (8M)—*Hydrofluoric Acid* (0.05M)—Add 500 mL concentrated nitric acid and 1.8 mL concentrated hydrofluoric acid to 250 mL water and dilute to 1 liter with water.

³ *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

7.7 *Diamyl, Amyolphosphonate Resin*,⁴ 50–100 μm particle size resin for use with vacuum-assisted flow systems; 100 to 150 μm or larger if using gravity flow systems.

7.8 *Polymethacrylate Resin*,⁵ 100–150 μm particle size resin.

8. Hazards

8.1 Refer to the laboratory's chemical hygiene plan and other applicable guidance for handling chemical and radioactive materials and for the management of radioactive, mixed, and hazardous waste.

8.2 Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid is similar to other acids in that the initial extent of a burn depends on the concentration, the temperature and the duration of contact with the acid. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Due to the serious consequences of hydrofluoric acid burns, prevention of exposure or injury of personnel is the primary goal. Utilization of appropriate laboratory controls (hoods) and wearing adequate personal protective equipment to protect from skin and eye contact is essential.

9. Procedure

9.1 Column Preparation and Conditioning:

NOTE 1—If prepackaged columns are available from the resin supplier, skip to [9.1.3](#).

NOTE 2—Each column shall contain 10 mL diamyl, amyolphosphonate resin. This resin amount can remove approximately 250 mg combined plutonium/uranium to enable impurity assay by ICP-AES, ICP-MS, or AAS.

9.1.1 Add approximately 3 mL of polymethacrylate resin to the bottom of a column. Rinse the column walls with water. Place a frit on top of the resin.

9.1.2 Add approximately 10 mL of diamyl, amyolphosphonate resin to the column. Rinse the column walls with water and place another frit on top of the resin.

9.1.3 Place the columns on a vacuum box if using a vacuum-assisted flow system or in a column rack if using a gravity flow system.

NOTE 3—If not using a vacuum box, the larger particle size resin should be used.

⁴ The sole source of supply of the UTEVA resin known to the committee at this time is Eichrom Technologies, Inc. 8205 S. Cass Ave Suite 106 Darien, IL 60561. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend. This resin is described in Horwitz, E. P., et al, "Separation and Preconcentration of Uranium from Acidic Media by Extraction Chromatography," *Analytica Chimica Acta*, 266, 1992, pp. 25-37.

⁵ The sole source of supply of the pre-filter material known to the committee at this time is Eichrom Technologies, Inc. 8205 S. Cass Ave Suite 106 Darien, IL 60561. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

9.1.4 Add 10 mL 8M nitric acid to each column to condition the resin. Turn on vacuum and adjust flow rate to 2–4 mL/min if using a vacuum-assisted flow system. Allow each column to drain completely and turn off the vacuum if using the vacuum assisted flow system.

9.1.5 Discard the eluted conditioning solutions.

9.2 Column Loading and Separation:

9.2.1 Procedures for dissolution of plutonium and uranium materials are found in Practices **C 1168** and **C 1347**.

9.2.2 Adjust the solution resulting from dissolution so that the nitric acid concentration is 8M.

9.2.3 Record the sample aliquot weight (g) or volume (mL) and sample pre-dilution factor (weight/volume or volume/volume).

9.2.4 Place clean, labeled 50 mL plastic collection tubes below each column in the vacuum extraction system.

9.2.5 Pipet 10 mL of sample aliquot prepared in **9.2.2** into the appropriate column. (the resin capacity is 250 mg U and Pu combined. If the concentration of the sample is greater than 25 mg/mL, then less than 10 mL should be added to avoid exceeding the resin capacity).

9.2.6 Turn on vacuum and adjust flow rate to 2–4 mL/min if using a vacuum-assisted flow system. Allow each column to drain completely.

9.2.7 Pipet 10 mL 8M nitric acid to each column and allow to drain completely.

9.2.8 Repeat step **9.2.7**.

NOTE 4—This second column elution step may be performed with hydrofluoric added (20 mL 8M HNO₃ – 0.05M HF) to completely elute zirconium, hafnium, tantalum and niobium (see **5.1**). This is done in a second elution rinse to minimize hydrofluoric acid in the initial column elution so that silicon background at the ICP-AES is minimized and silicon can be measured in the first elution. A small amount of zirconium, hafnium, tantalum and niobium may be in the first elution so results from both elutions must be added to get a final result. If silicon is not of interest, this step can be avoided by making the 8M nitric acid load solution approximately 0.05M hydrofluoric acid.

9.2.9 After columns have drained completely, increase the vacuum to remove residual liquid from columns, if using the vacuum-assisted flow system.

9.2.10 Turn off vacuum if using the vacuum-assisted flow system and remove collection tubes from vacuum system. Adjust collection tube volume to 30 mL with 8M nitric acid, if needed.

9.2.11 Submit collected fractions for analysis by the selected technique such as ICP-MS, ICP-AES, or AAS.

9.3 While not strictly within the scope of this practice, plutonium or uranium, or both, retained on the column may be eluted at this point, if desirable for the purposes of the laboratory performing this analysis. Guidance for accomplishing this may be found in Horwitz, et al.⁴

10. Calculations

10.1 Calculate the Metal Impurity, *I*:

$$I = (S - B) \times D \quad (1)$$

where:

S = metallic impurity measured in final solution, µg/mL,

B = blank measurement for metallic impurity in final solution, µg/mL, and

D = sample dilution factor.

$$D = \frac{SV \times P}{SA} \quad (2)$$

where:

SV = column removal volume, mL (typically 30 mL, step **9.2.10**),

SA = sample aliquot used in column separation, mL (typically 10 mL, step **9.2.5**), and

P = pre-dilution factor (step **9.2.3**).

11. Keywords

11.1 AAS; extraction chromatography; ICP-AES; ICP-MS; impurities; plutonium; resin; separation; uranium

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