



Designation: C1647 – 20

# Standard Practice for Removal of Uranium or Plutonium, or both, for Impurity Assay in Uranium or Plutonium Materials<sup>1</sup>

This standard is issued under the fixed designation C1647; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[C753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder](#)

[C757 Specification for Nuclear-Grade Plutonium Dioxide Powder for Light Water Reactors](#)

[C776 Specification for Sintered Uranium Dioxide Pellets for Light Water Reactors](#)

[C787 Specification for Uranium Hexafluoride for Enrichment](#)

[C788 Specification for Nuclear-Grade Uranyl Nitrate Solution or Crystals](#)

[C859 Terminology Relating to Nuclear Materials](#)

[C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % <sup>235</sup>U](#)

[C1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis](#)

[C1287 Test Method for Determination of Impurities in Nuclear Grade Uranium Compounds by Inductively Coupled Plasma Mass Spectrometry](#)

[C1347 Practice for Preparation and Dissolution of Uranium 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](#)

[C1517 Test Method for Determination of Metallic Impurities in Uranium Metal or Compounds by DC-Arc Emission Spectroscopy](#)

[D1193 Specification for Reagent Water](#)

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method but not defined herein, refer to Terminology [C859](#).

## 4. Summary of Practice

4.1 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.

## 5. Significance and Use

5.1 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

<sup>1</sup> 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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<sup>2</sup> 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.

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.

5.2 Other test methods exist to determine impurities in uranium or plutonium. Test Method **C1517** is able to determine many impurities in uranium at detection levels of ~1–10 part-per-million (ppm) by DC-Arc Spectrometry. Test Method **C1287** is able to determine impurities in uranium at detection levels of ~100 ppb by ICP-MS. Test Method **C1432** provides an alternative technique to remove plutonium by ion exchange prior to analysis of the impurities by ICP-AES.

5.3 This practice can be used to demonstrate compliance with nuclear fuel specifications, for example, Specifications **C753**, **C757**, **C776**, **C787**, **C788**, and **C996**.

## 6. Interferences

6.1 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.

## 7. Apparatus

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

7.2 *Plastic Collection Tubes*, 50 mL.

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

7.4 *Polyethylene Frits* for columns, 20 µm.

7.5 *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.

## 8. Reagents

8.1 *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.<sup>3</sup> 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.

<sup>3</sup> *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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification **D1193** as Type I.

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

8.4 *Nitric Acid (16 M)*—Concentrated HNO<sub>3</sub> (sp gr 1.42).

8.5 *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.

8.6 *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.

8.7 *Diamyl, Amyolphosphonate Resin*,<sup>4,5</sup> 50–100 µm particle size resin for use with vacuum-assisted flow systems; 100 to 150 µm if using gravity flow systems.

8.8 *Polymethacrylate Resin*,<sup>6</sup> 100–150 µm particle size resin.

## 9. Hazards

9.1 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.

9.2 Hydrofluoric acid is a highly corrosive and toxic acid that can severely burn skin, eyes, and mucous membranes. 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. Familiarization and compliance with the Safety Data Sheet is essential.

## 10. Procedure

### 10.1 Column Preparation and Conditioning:

NOTE 1—If prepackaged columns are available from the resin supplier, skip to **10.1.3**.

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.

10.1.1 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.

<sup>4</sup> The sole source of supply of the UTEVA Resin known to the committee at this time is Eichrom Technologies LLC, Lisle, IL, USA and Triskem International, Bruz, France. 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,<sup>1</sup> which you may attend.

<sup>5</sup> 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.

<sup>6</sup> The sole source of supply of the Pre-filter material known to the committee at this time are Eichrom Technologies LLC, Lisle, IL, USA and Triskem International, Bruz, France. 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,<sup>1</sup> which you may attend.