Designation: C1284 – 10

Standard Practice for
Electrodeposition of the Actinides for Alpha Spectrometry[1]

This standard is issued under the fixed designation C1284; 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 practice covers the preparation of separated actinide fractions for alpha spectrometry measurement.[2] It is applicable to any of the actinides that can be dissolved in dilute ammonium sulfate solution. Examples of applicable actinide fractions would be the final elution from an ion exchange separation or the final strip from a solvent extraction separation.

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C1163 Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride
D1193 Specification for Reagent Water

3. Summary of Practice

3.1 Guidance is provided for the electrodeposition of separated actinide fractions onto metal discs. This practice is based on cathodic deposition of the hydrated oxides of the actinides from an acidic medium containing an ammonium salt. The resultant electrodeposited samples are suitable for alpha spectrometry measurements.

4. Significance and Use

4.1 The determination of actinide elements by alpha spectrometry measurement is an essential part of many environmental research, bioassay, and monitoring programs. Alpha spectrometry measurements identify and quantify the alpha-emitting actinide elements. A variety of separation methods will typically precede the electrodeposition of a sample for alpha spectrometry measurements. In addition to the electrodeposition procedure presented in this practice, the scientific literature contains other procedures for actinide electrodeposition.

NOTE 1—An alternate method for mounting actinides for alpha spectrometry measurements by coprecipitation with neodymium fluoride is described in Practice C1163.

5. Interferences

5.1 Any element present in the separated fraction which is capable of cathodic electrodeposition will be present on the metal disc. In particular ^{210}Po (5.30 MeV) deposited on the disc would interfere with the yield determination of ^{232}U (5.32 MeV) or ^{243}Am (5.28 MeV) tracers used in the determination of isotopic uranium and ^{241}Am (5.15 MeV), respectively.

5.2 Incomplete separation of rare earth elements or incomplete wet ashing for the removal of organic material will decrease the efficiency of the electrodeposition and may result in a thick deposit unsuitable for alpha spectrometry measurement.

5.3 The quantity of actinide should be such that <5 μg cm^{-2} are electrodeposited on the metal disc. Thicker deposits are typically unsuitable for measurement by alpha spectrometry due to the resulting attenuation and decrease in energy resolution.

6. Apparatus

6.1 *Electrodeposition Power Supply*—Constant current, adjustable from 0 to 2 A with indicating meter.

6.2 *Electrodeposition Cell*—Disposable cells are recommended. The cells should have a minimum volume capacity of 25 mL.

6.3 *Metal Discs*—Stainless steel disc, or other metal disc such as platinum, polished to a mirror finish on one side. The

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[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] Based on Talvitie, N. A., "Electrodeposition of Actinides for Alpha Spectrometric Determination," *Analytical Chemistry*, Vol 44, 1972, pp. 280–283.

[3] 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.

diameter of the disc is determined by diameter of the electrodeposition cell. The current density should be approximately 0.5 A cm^{-2} of the disc area.

6.4 *Electrodeposition Anode*—The exact dimensions of the anode will be determined by the cross-sectional area and depth of the electrodeposition cell. For example, a 1.5 mm diameter by 100 mm long platinum wire with loop facing the cathode (stainless steel disc). The anode loop should be just slightly smaller than the cathode.

6.5 *Insulated Base,* to support the cell and provide the cathode connection.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals must be used in all procedures. Unless otherwise noted, all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,[4] if such specifications are available. Other grades of reagents may be used if it is ascertained by the user that the reagent is of sufficiently high purity to permit its use without reducing the accuracy of the determination. All reagents should be stored in plastic bottles.

7.2 *Reagent Blanks*—Reagent blanks should be analyzed to determine their contribution to the sample result. The analysis of reagent blanks as part of the overall analytical method should be considered.

7.3 *Purity of Water*—Unless otherwise indicated, water is defined as reagent water as described in Specification D1193, Type III.

7.4 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (15 M NH_4OH).

7.5 *Ammonium Sulfate Solution (1 M)*—Dissolve 132 g of $(NH_4)_2SO_4$ in water and dilute to 1 L.

7.6 *Electrolyte Solution*—1 M $(NH_4)_2SO_4$ solution adjusted to pH 3.5 with concentrated H_2SO_4 and concentrated NH_4OH.

7.7 *Ethyl Alcohol Solution*—95 %.

7.8 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (16 M HNO_3).

7.9 *Sodium Hydrogen Sulfate (0.36 M in 1 M H_2SO_4)*—Dissolve 10 g of $NaHSO_4 \cdot H_2O$ in 88 mL of water and add 112 mL of 1.8 M H_2SO_4.

7.10 *Sulfuric Acid (1.8 M)*—Mix 100 mL of concentrated sulfuric acid with water and dilute to 1 L.

8. Precautions

8.1 Adequate laboratory facilities such as fume hoods and controlled ventilation, along with safe techniques must be used in performing this procedure. Use of safety glasses or goggles is recommended.

8.2 The electrodeposition cell should be tested for leaks prior to use.

9. Electrodeposition Procedure

9.1 Add 2 mL of 0.36 M $NaHSO_4$ in 1 M H_2SO_4 to the separated actinide fraction in the beaker. Add 5 mL of concentrated HNO_3 to the beaker, swirl to mix, and evaporate the solution to dryness but do not bake.

9.2 Add 5 mL of electrolyte solution to the beaker and warm to dissolve the residue.

9.3 Transfer the sample solution to the electrodeposition cell. Rinse the beaker with 5 to 10 mL of electrolyte solution and add the rinsings to the electrodeposition cell. Add pH indicator and adjust the pH.

9.4 Connect the cathode (−) to base of the electrodeposition cell.

9.5 Lower the platinum anode (+) to about 1 cm above the stainless steel disc in the electrodeposition cell.

9.6 Turn on the power supply and, for a 2.5 cm metal disc, adjust the current to 1.2 A. Electrodeposit at a constant current for 2 h.

9.7 After 2 h, add 1 mL of concentrated NH_4OH to the cell and continue to electrodeposit for 1 min.

9.8 Turn off the current and remove the anode. Empty and discard the electrolyte solution.

9.9 Disassemble the cell and rinse the disc with ethyl alcohol solution. Touch the edge of the disc with a tissue to absorb the excess alcohol.

9.10 Place the disc on a 200 to 250°C hot plate to dry and fix the electrodeposited species. Air drying of the electroplated disc has also been successfully used.

9.11 Submit the sample for alpha spectrometry measurement.

10. Keywords

10.1 actinides; alpha spectrometry; electrodeposition

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[4] *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.

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