Designation: C 1414 – 01 (Reapproved 2007)

Standard Practice for The Separation of Americium from Plutonium by Ion Exchange[1]

This standard is issued under the fixed designation C 1414; 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 describes the use of an ion exchange technique to separate plutonium from solutions containing low concentrations of americium prior to measurement of the ^{241}Am by gamma counting.

1.2 This practice covers the removal of plutonium, but not all the other radioactive isotopes that may interfere in the determination of ^{241}Am.

1.3 This practice can be used when ^{241}Am is to be determined in samples in which the plutonium is in the form of metal, oxide, or other solid provided that the solid is appropriately sampled and dissolved (See Test Methods C 758, C 759, and C 1168).

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 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 758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Metal

C 759 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis

C 1268 Test Method for Quantitative Determination of Americium 241 in Plutonium by Gamma-Ray Spectrometry

D 1193 Specification for Reagent Water

3. Summary of Practice

3.1 Plutonium is adsorbed from a nitric acid (HNO_3) solution (8 *M*) onto an anion exchange resin. Under these conditions, a negligible amount of americium is adsorbed onto the resin and may be determined by gamma counting of the eluate using Test Method C 1268.

4. Significance and Use

4.1 This practice is applicable when small amounts of ^{241}Am are present in plutonium samples (see Test Methods C 758 and C 759). An example is the determination of ^{241}Am in a ^{238}Pu sample. The high specific activity of ^{238}Pu presents a safety hazard that precludes its presence in a counting facility. Therefore, it is necessary to remove the ^{238}Pu prior to the determination of ^{241}Am.

4.2 When a plutonium solution contains fission or activation products, this practice does not separate all radionuclides that interfere in the determination of ^{241}Am, such as the rare earths.

5. Interferences

5.1 The presence of other gamma-ray emitting radionuclides similar in energy to ^{241}Am or that interfere with gamma counting make the determination of ^{241}Am less accurate. Most +4 valence actinides are adsorbed on the resin. The distribution coefficient for Am on this resin in nitric acid is less than 1, indicating insignificant adsorption. Therefore, this practice will separate many elements that might interfere with gamma counting of ^{241}Am.

5.1.1 The elements thorium, neptunium (IV), gold, platinum, iridium, and palladium are not quantitatively separated from plutonium by this procedure.

6. Apparatus

6.1 *Anion exchange resin column (100-200 mesh)*, containing quaternary ammonium functional groups (basic resin-chloride ionic form).[3]

6.2 *Bottles*, polyethylene, 30 mL.

6.3 *Sample beaker*, 30 mL, borosilicate glass.

[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.

[3] Prefilled columns packed with AG 1-X8, available from Bio-Rad, Richmond, CA, have been found to be acceptable.

6.4 *Hot plate*.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals should 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.[4] Other grades may be used, provided that the reagent is first demonstrated to be 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 (Specification D 1193).

7.3 *Nitric acid (HNO_3)*, concentrated (sp gr 1.42).

7.4 *Hydrochloric acid (HCl)*, concentrated (sp gr 1.19).

7.5 *Nitric acid, 0.1 M*. Add 6 mL of concentrated HNO_3 (sp gr 1.42) to 950 mL of water and dilute to 1 L.

7.6 *Nitric acid 8 M*. Add 500 mL of concentrated HNO_3 (sp gr 1.42) to 400 mL of water and dilute to 1 L.

7.7 *Hydrofluoric acid (HF)*, concentrated (sp. gr. 1.18).

7.8 *Strip solution, 0.1 M HCl/0.01 M HF*. Add 8.3 mL of concentrated HCl (sp gr. 1.19) and 0.4 mL (6 to 7 drops) of concentrated HF to 950 mL of water and dilute to 1 L.

8. Procedure

8.1 Prepare a plutonium solution by following the procedure in Practice C 1168 or by using another suitable dissolution technique. Transfer an aliquot of the plutonium solution to a 30 mL beaker. The amount of plutonium must be less than the adsorption capacity of the ion exchange resin. A maximum of 50 mg of plutonium is suggested for the prefilled columns.

8.2 Evaporate the sample to dryness on a hot plate. Add 3-4 mL of 8 *M* HNO_3 and take to dryness again. Cool the sample to room temperature and repeat the dissolution and evaporation once more before proceeding to 8.3.

8.3 Condition a prefilled anion exchange column by adding 3-5 mL of 8 *M* HNO_3 and allow to drain. Discard the eluant.

8.4 Position a clean 1 oz polyethylene bottle beneath the column to collect the effluent. Dissolve the plutonium sample in beaker containing 3-4 mL of 8 *M* HNO_3. Transfer contents of the beaker to the preconditioned ion exchange column.

8.5 Allow solution to drain into the bottle. Rinse beaker with 3-4 mL of 8 *M* HNO_3. Transfer the rinse from the beaker to a column and allow the solution to drain into a bottle. Repeat this process twice more, allowing column to drain between additions before proceeding to 8.6.

8.6 Add 10 mL of 8 *M* HNO_3 directly to the column for the final rinse and allow to drain. Remove the bottle and add sufficient 8 *M* HNO_3 to make a total volume equal to 25 ± 2 mL.

8.7 Survey the bottle for external contamination.

8.7.1 If bottle exterior is found to be contaminated, clean to acceptable levels of activity and transfer to a counting facility.

8.7.2 If no contamination is found, transfer the bottle to a counting facility and determine the activity of gamma counting according to Test Method C 1268.

8.8 Strip the plutonium from the column with three 5 mL aliquots of 0.1 *M* HNO_3 or 1.0 *M* HCl/0.01 *M* HF.[5] Discard the column and place the plutonium in the appropriate waste stream, or keep for further analysis.

9. Keywords

9.1 americium; gamma counting; ion exchange; plutonium solutions

[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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[5] 0.1 *M* HCl/0.01 *M* HF is used when a more complete removal of plutonium from the ion exchange resin is desired.