



# Standard Practice for The Separation of Americium from Plutonium by Ion Exchange<sup>1</sup>

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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 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  $^{241}\text{Am}$  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  $^{241}\text{Am}$ .

1.3 This practice can be used when  $^{241}\text{Am}$  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:

C 758 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear and Radiochemical Analysis of Nuclear-Grade Plutonium Metal<sup>2</sup>

C 759 Test Methods for Chemical, Mass Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Plutonium Nitrate Solutions<sup>2</sup>

C 1168 Practice for Preparation and Dissolution of Plutonium Materials for Analysis<sup>2</sup>

C 1268 Test Method for the Quantitative Determination of Americium 241 in Plutonium by Gamma-Ray Spectrometry<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

## 3. Summary of Practice

3.1 Plutonium is adsorbed from a nitric acid ( $\text{HNO}_3$ ) solution (8 M) onto an anion exchange resin. Under these condi-

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

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

## 5. Interferences

5.1 The presence of other gamma-ray emitting radionuclides similar in energy to  $^{241}\text{Am}$  or that interfere with gamma counting make the determination of  $^{241}\text{Am}$  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  $^{241}\text{Am}$ .

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).<sup>4</sup>

6.2 *Bottles*, polyethylene, 30 mL.

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

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

<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.

Current edition approved January 10, 2001. Published March 2001. Originally published as C 1414-99. Last previous edition C 1414-99.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 12.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>4</sup> Prefilled columns packed with AG 1-X8, available from Bio-Rad, Richmond, CA, have been found to be acceptable.

Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</sup> 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<sub>3</sub>)*, 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<sub>3</sub> (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<sub>3</sub> (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<sub>3</sub> 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<sub>3</sub> 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<sub>3</sub>. 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<sub>3</sub>. 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<sub>3</sub> directly to the column for the final rinse and allow to drain. Remove the bottle and add sufficient 8 M HNO<sub>3</sub> 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<sub>3</sub> or 1.0 M HCl/0.01 M HF.<sup>6</sup> 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

<sup>5</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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

<sup>6</sup> 0.1 M HCl/0.01 M HF is used when a more complete removal of plutonium from the ion exchange resin is desired.

*The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.*

*This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.*

*This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).*