Designation: D7939 – 15

Standard Test Method for Rapid Radiochemical Determination of Americium-241 in Water[1]

This standard is issued under the fixed designation D7939; 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 test method is specifically for Americium-241 (^{241}Am) in drinking water and other aqueous samples. However, if any isotopes of curium are present in the sample, they will be carried with americium during the analytical separation process and will be observed in the final alpha spectrum. The presence of ^{243}Am in the water sample will bias the results obtained by this test method.

1.2 This test method is applicable to samples in which radioactive contamination is from either known or unknown origins. If any filtration of the sample is performed before starting the analysis, those solids should be analyzed separately. The results from the analysis of these solids should be reported separately (as a suspended activity concentration for the water volume filtered) but identified with the filtrate results.

1.3 This test method is applicable to the determination of soluble ^{241}Am. This test method is not applicable to the determination of ^{241}Am in highly insoluble particulate matter possibly present in water samples contaminated as a result of a radiological dispersal device (RDD) event.

1.4 This test method uses rapid radiochemical separation techniques for determining americium in water samples following a radiological or nuclear incident. Although, with this test method, concentrations of ^{241}Am on the same order of magnitude as methods used for the Safe Drinking Water Act (SDWA) can be detected, this test method is not a substitute for SDWA-approved methods for ^{241}Am.

1.5 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.6 *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]

C1163 Practice for Mounting Actinides for Alpha Spectrometry Using Neodymium Fluoride

C1284 Practice for Electrodeposition of the Actinides for Alpha Spectrometry

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D3370 Practices for Sampling Water from Closed Conduits

D4448 Guide for Sampling Ground-Water Monitoring Wells

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

D6001 Guide for Direct-Push Groundwater Sampling for Environmental Site Characterization

D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *analytical action level, AAL, n*—denotes the value of a quantity that will cause the decision maker to choose an alternative action.

3.2.2 *analytical protocol specifications, APS, n*—output of a directed planning process that contains the project's analytical data needs and requirements in an organized, concise form.

3.2.3 *discrete radioactive particles, DRPs or hot particles, n*—particulate matter in a sample of any matrix in which a high concentration of radioactive material is contained in a tiny particle (micrometre range).

3.2.4 *measurement quality objective, MQO, n*—analytical data requirements of the data quality objectives; project- or program-specific.

3.2.4.1 *Discussion*—They can be quantitative or qualitative. MQOs serve as measurement performance criteria or objectives of the analytical process.

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

Current edition approved Jan. 1, 2015. Published January 2015. DOI: 10.1520/D7939-15.

[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.

3.2.5 *radiological dispersal device, RDD, n*—unconventional weapon constructed to distribute radioactive material(s) into the environment either by incorporating them into a conventional bomb or using sprays, canisters, or manual dispersal, that is, a "dirty bomb."

3.2.6 *required method uncertainty, u_{MR}, n*—target value for the individual measurement uncertainties and an estimate of uncertainty (of measurement) before the sample is actually measured.

3.2.6.1 *Discussion*—The required method uncertainty is applicable below an AAL.

3.2.7 *required relative method uncertainty, φ_{MR}, n*—the value, u_{MR}, divided by the AAL and typically expressed as a percentage.

3.2.7.1 *Discussion*—It is applicable above the AAL.

3.2.8 *sample test source, STS, n*—final form of the sample that is used for nuclear counting.

3.2.8.1 *Discussion*—This form is usually specific for the nuclear counting technique used in this test method, such as a solid deposited on a filter for alpha spectrometry analysis.

3.3 *Acronyms:*

3.3.1 *AAL*—analytical action level

3.3.2 *APS*—analytical protocol specification

3.3.3 *DRPs*—discrete radioactive particles

3.3.4 *IRM*—independent reference material

3.3.5 *MARLAP*—Multi-Agency Radiological Laboratory Analytical Protocols Manual[3]

3.3.6 *MDC*—minimum detectable concentration

3.3.7 *MQO*—measurement quality objective

3.3.8 *NIST*—National Institute of Standards and Technology—United States

3.3.9 *NPL*—National Physical Laboratory—United Kingdom

3.3.10 *RDD*—radiological dispersal device

3.3.11 *STS*—sample test source

4. Summary of Test Method

4.1 This test method is based on a sequence of two chromatographic extraction resins used to concentrate, isolate, and purify americium by removing interfering radionuclides as well as other components of the water matrix to prepare the americium fraction for counting by alpha spectrometry. This test method uses vacuum-assisted flow to improve the speed of the separations. Before the use of the extraction resins, the water sample is filtered as necessary to remove any insoluble fractions, equilibrated with americium-243 (^{243}Am) tracer, and concentrated by evaporation or calcium phosphate precipitation. The sample test source (STS) is prepared by microprecipitation with neodymium(III) fluoride (NdF_3). Standard laboratory protocol for the use of an alpha spectrometer should be used when the sample is ready for counting.

5. Significance and Use

5.1 This test method is considered a rapid method when compared to other classical methods for the determination of ^{241}Am in aqueous solutions. During the method validation of this method, a test batch of fourteen test samples plus quality control samples was chemically processed in ~7.5 hours. Additional time for counting the samples depends on the measurement quality objectives.

5.2 This test method is specific for Americium-241 (^{241}Am) in drinking water and other aqueous samples. However, if any isotopes of curium are present in the sample, they will be carried with americium during the analytical separation process and will be observed in the final alpha spectrum.

5.3 This test method is capable of achieving a required method uncertainty for ^{241}Am of 0.070 Bq/L at an analytical action level of 0.555 Bq/L. This test method is capable of achieving a required relative method uncertainty, φ_{MR}, 13 % above 0.555 Bq/L. This test method is capable of achieving a "required" minimum detectable concentration (MDC) of 0.055 Bq/L.

5.4 To attain these stated measurement quality objectives (MQOs), a sample volume of approximately 200 mL and count time of at least 1 to 3 hours are recommended. The sample turnaround time and throughput may vary based on additional project MQOs, the time for analysis of the final counting form, and initial sample volume. This test method should be validated before use following the protocols provided in Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities.[4]

5.5 This test method is intended to be used for water samples that are similar in composition to drinking water. This method was evaluated following the guidance presented for "Level E Method Validation: Adapted or Newly Developed Methods, Including Rapid Methods" in Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities and Chapter 6 of MARLAP, 2004.[3] Multi-radionuclide analysis using sequential separation may be possible using this test method in conjunction with other rapid methods.

6. Interferences

6.1 *Radiological*—Alpha-emitting radionuclides with irresolvable alpha energies, such as ^{241}Am (5.48 MeV), ^{238}Pu (5.50 MeV), and ^{228}Th (5.42 MeV), shall be chemically separated to enable radionuclide-specific measurements. This test method separates these radionuclides effectively. The significance of peak overlap will be determined by the individual detector's alpha energy resolution characteristics and the quality of the final precipitate that is counted.

6.2 *Tracer Impurity*—The presence of ^{241}Am may be found in ^{243}Am tracer solutions. Newly acquired ^{243}Am tracer solutions shall be analyzed before use to verify the absence of ^{241}Am activity.

[3] *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP), EPA 402-B-1304 04-001A, Vol I, Chaps. 6, 7, and 20, Glossary; Vols II and III, Appendix G, July 2004, www.epa.gov/radiation/ marlap/index.html

[4] *Method Validation Guide for Radiological Laboratories Participating in Incident Response Activities*, Revision 0, U.S. Environmental Protection Agency, Office of Air and Radiation, Washington, DC, EPA 402-R-09-006, June 2009, www.epa.gov/narel/incident_guides.html and www.epa.gov/erln/radiation.html.

6.3 *Non-Radiological*—Very high levels of competing higher valence anions (greater than divalent such as phosphates) will lead to lower yields when using the evaporation option because of competition with active sites on the resin. If higher valence anions are present, the phosphate precipitation option may need to be used initially in place of evaporation. If calcium phosphate coprecipitation is performed to collect americium (and other potentially present actinides) from large-volume samples, the amount of phosphate added to coprecipitate the actinides (in 12.1.4.1) should be reduced to accommodate the sample's high phosphate concentration.

7. Apparatus

7.1 *Analytical balance,* with a 10^{-4}-g readability or better.

7.2 *Centrifuge,* able to accommodate 225- to 250-mL flasks.

7.3 *Centrifuge tubes,* plastic, 15-, 25-, and 50-mL capacity, or equivalent.

7.4 *Centrifuge flasks,* 225- to 250-mL capacity.

7.5 *Filter,* 0.45-µm membrane.

7.6 *pH Paper,* 1.0 to 4.0.

7.7 *Filter apparatus,* 25-mm-diameter, polysulfone filtration chimney, stem support, and stainless steel support, or equivalent. A single-use (disposable) filter funnel/filter combination may be used to avoid cross-contamination.

7.8 *Filter, 25-mm membrane,* 0.1-µm pore size or equivalent, polypropylene, polycarbonate, or equivalent.

7.9 *Stainless steel planchets,* or other sample mounts able to hold the 25-mm filter.

7.10 *Tweezers.*

7.11 *Pipette,* 100-µL or equivalent and appropriate plastic tips.

7.12 *Reservoirs,* with male Luer connector, 10 or 20-mL syringe style.

7.13 *Vacuum box system,* compatible with standard Luer-tipped SPE columns and appropriate connectors.

7.13.1 *Vacuum box and lid,* capable of being thoroughly cleaned, including eluate ports.

7.13.2 *Disposable eluate connectors/liners,* specific to the box lid.

7.13.3 *Inner rack,* with 30-mm diameter holes (for 50-mL centrifuge tubes).

7.14 *Vortex mixer.*

7.15 *Vacuum source.*

7.16 *Miscellaneous laboratory ware,* plastic or glass, 25, 50, 100, 250, and 350 mL.

8. Reagents and Standards

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.[5] Other grades may be used, provided that that the reagent is of sufficient high purity to permit its use without increasing the background of the measurement. Some reagents, even those of high purity, may contain naturally occurring radioactivity, such as isotopes of uranium, radium, actinium, thorium, rare earths and potassium compounds, or artificially produced radionuclides, or any combination thereof. Consequently, when such reagents are used in the analysis of low-radioactivity samples, the activity of the reagents shall be determined under analytical conditions that are identical to those used for the sample. The activity contributed by the reagents should be considered to be a component of background and applied as a correction when calculating the test sample result. The increased background may reduce the sensitivity of the measurement.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 of Specification D1193. All solutions used in microprecipitation should be prepared with water filtered through a 0.45-µm (or better) filter.

8.3 *Am-243 Tracer Solution*—Add 0.22 to 0.37 Bq ^{243}Am per aliquant; activity added known to at least 5 % (combined standard uncertainty ≤ 5 %).

8.4 *Ammonium Hydrogen Phosphate [$(NH_4)_2HPO_4$] (3.2M)*—Dissolve 106 g of $(NH_4)_2HPO_4$ in 200 mL of water, heat gently to dissolve, and dilute to 250 mL with water.

8.5 *Ammonium Hydroxide (NH_4OH)(15M)*—Concentrated, available commercially.

8.6 *Ammonium Thiocyanate (NH_4SCN) Indicator (1M)*—Dissolve 7.6 g of NH_4SCN in 90 mL of water and dilute to 100 mL with water. An appropriate quantity of sodium thiocyanate (8.1 g) or potassium thiocyanate (9.7 g) may be substituted for NH_4SCN.

8.7 *Ascorbic Acid ($C_6H_8O_6$) (1M)*—Dissolve 17.6 g of $C_6H_8O_6$ in 90 mL of water and dilute to 100 mL with water. Prepare weekly.

8.8 *Calcium Nitrate (0.9M)*—Dissolve 53 g of calcium nitrate tetrahydrate ($Ca(NO_3)_2{\cdot}4H_2O$) in 100 mL of water and dilute to 250 mL with water.

8.9 *Ethanol, 100 %*—Anhydrous C_2H_5OH, available commercially.

8.9.1 *Ethanol (~80 % v/v)*—Mix 80-mL 100 % ethanol and 20-mL water.

8.10 *Ferrous Sulfamate (0.6M)*—Add 57 g of sulfamic acid (NH_2SO_3H) to 150 mL of water and heat to 70°C. Slowly add 7 g of iron powder (<100-mesh size) while heating and stirring with a magnetic stirrer until dissolved (may take as long as 2 h). Filter the hot solution using a qualitative filter, transfer to flask, and dilute to 200 mL with water. Prepare fresh weekly.

[5] *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.

8.11 *Hydrochloric Acid (HCl) (12M)*—Concentrated HCl, available commercially.

8.11.1 *Hydrochloric Acid (9M)*—Add 750 mL of concentrated HCl to 100 mL of water and dilute to 1 L with water.

8.11.2 *Hydrochloric Acid (4M)*—Add 333 mL of concentrated HCl to 500 mL of water and dilute to 1 L with water.

8.11.3 *Hydrochloric Acid (1M)*—Add 83 mL of concentrated HCl to 500 mL of water and dilute to 1 L with water.

8.12 *Hydrofluoric Acid (HF) (28M)*—Concentrated HF, available commercially.

8.12.1 *Hydrofluoric Acid (0.58M)*—Add 20 mL of concentrated HF to 980 mL of filtered demineralized water and mix. Store in a plastic bottle.

8.13 *Isopropyl Alcohol,* 95 %.

8.14 *Neodymium Standard Solution (1000 µg/mL)*—May be purchased from a supplier of standards for atomic spectroscopy. Cerium may be substituted for neodymium.

8.15 *Neodymium Carrier Solution (0.50 mg/mL)*—Dilute 10 mL of the neodymium standard solution (8.14) to 200 mL with filtered demineralized water. This solution is stable.

8.16 *Neodymium Fluoride Substrate Solution (10 µg/mL)*—Pipette 5 mL of neodymium standard solution (8.14) into a 500-mL plastic bottle. Add 460 mL of 1*M* HCl to the plastic bottle. Cap the bottle and shake to mix. Measure 40 mL of concentrated HF in a plastic graduated cylinder and add to the bottle. Recap the bottle and shake to mix thoroughly. This solution is stable for up to six months.

8.17 *Nitric Acid (HNO_3) (16M)*—Concentrated HNO_3, available commercially.

8.17.1 *Nitric Acid (3M)*—Add 191 mL of concentrated HNO_3 to 700 mL of water and dilute to 1 L with water.

8.17.2 *Nitric Acid (2M)*—Add 127 mL of concentrated HNO_3 to 800 mL of water and dilute to 1 L with water.

8.17.3 *Nitric Acid (0.5M)*—Add 32 mL of concentrated HNO_3 to 900 mL of water and dilute to 1 L with water.

8.18 *Nitric Acid (2M)-Sodium Nitrite (0.1M) Solution*—Add 32 mL of concentrated HNO_3 (8.17) to 200 mL of water and mix. Dissolve 1.7 g of sodium nitrite ($NaNO_2$) in the solution and dilute to 250 mL with water. Prepare fresh daily.

8.19 *Nitric Acid (3M)-Aluminum Nitrate [$Al(NO_3)_3$] (1.0M) Solution*—Dissolve 213 g of anhydrous $Al(NO_3)_3$ in 700 mL of water. Add 190 mL of concentrated HNO_3 (8.17) and dilute to 1 L with water. Alternatively $Al(NO_3)_3$ nonahydrate (375 g) may be substituted for anhydrous $Al(NO_3)_3$.

8.20 *Phenolphthalein Solution*—Dissolve 1 g of phenolphthalein in 100-mL 95 % isopropyl alcohol and dilute with 100 mL of water.

8.21 *CMPO Resin*[6,7]—Cartridge, 2 mL, 50- to 100-µm mesh size, solid extractant.

8.22 *DAAP Resin*[8,9]—Cartridge, 2 mL, 50- to 100-µm mesh size, solid extractant.

9. Hazards

9.1 *General:*

9.1.1 Refer to the laboratory's safety manual for concerns of contamination control, personal exposure monitoring, and radiation dose monitoring.

9.1.2 Refer to the laboratory chemical hygiene plan (or equivalent) for general safety rules regarding chemicals in the workplace.

9.2 *Radiological:*

9.2.1 *Hot Particles (DRPs):*

9.2.1.1 Hot particles, also termed "discrete radioactive particles (DRPs)," will be small on the order of 1 mm or less. Typically, DRPs are not evenly distributed in the media, and their radiation emissions are not uniform in all directions (anisotropic). Filtration using a 0.45-µm or finer filter will minimize the presence of these particles in the filtrate.

9.2.1.2 Care should be taken to provide suitable containment for filter media used in the pretreatment of samples that may have DRPs because the particles become highly statically charged as they dry out and will "jump" to other surfaces causing contamination.

9.2.1.3 Filter media should be individually surveyed for the presence of these particles, and this information should be reported with the final sample results.

9.2.2 For samples with detectable activity concentrations of ^{241}Am, laboratory ware should be used only once because of the potential for cross contamination.

9.3 *Procedure-Specific Non-Radiological Hazards*—Particular attention should be paid to the use of HF. HF is an extremely dangerous chemical used in the preparation of some

[6] CMPO resin, also known as TRU[7] resin in the radiochemistry community, is octylphenyl_N-N-di-isobutyl carbamoylphosphine oxide (CMPO) dissolved in tri-n-butyl phosphate (TBP); Eichrom Parts TR-R50-S, or equivalent. The sole source of supply of the resins known to the committee at this time is Eichrom Technologies LLC, 1955 University Lane, Lisle, IL 60532. 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.

[7] TRU is a trademark of Eichrom Technologies LLC.

[8] DAAP resin, also known as UTEVA[9] resin, is diamyl amylphosphonate, Eichrom Parts UT-R50-S6 or equivalent. The sole source of supply of the resins known to the committee at this time is Eichrom Technologies LLC, 1955 University Lane, Lisle, IL 60532. 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] UTEVA is a trademark of Eichrom Technologies LLC.

of the reagents and the microprecipitation procedure. Appropriate personal protective equipment (PPE) should be used in strict accordance with the laboratory safety program specification.

10. Sample Collection, Preservation, and Storage

10.1 Collect a sample in accordance with Guides D4448 and D6001 and Practice D3370 or other documented procedures as appropriate.

10.2 No sample preservation is required if sample is delivered to the laboratory within three days of sampling date/time.

10.3 If the dissolved concentration of americium is sought, the insoluble fraction shall be removed by filtration before preserving with acid.

10.4 If the sample is to be held for more than three days, concentrated HNO_3 shall be added to achieve a pH < 2.

11. Calibration and Standardization

11.1 Set up the alpha spectrometry system according to the manufacturer's recommendations. The energy range of the spectrometry system should at least include the region between 3 and 8 MeV.

11.2 Calibrate each detector used to count samples according to appropriate section of Practice D7282.

11.3 Continuing instrument quality control testing shall be performed according to the applicable sections of Practice D7282.

12. Procedure

12.1 *Water Sample Preparation:*

12.1.1 As required, filter the 100- to 200-mL sample aliquant through a 0.45-µm filter and collect the sample in an appropriate size beaker.

12.1.2 Acidify the sample with concentrated HNO_3 to a pH < 2.0 by adding enough HNO_3. This usually requires about 2 mL of HNO_3 per 1000 mL of sample.

12.1.3 Add 0.22 to 0.37 Bq of ^{243}Am as a tracer following laboratory protocol.

NOTE 1—For a sample approximately 100 mL or less, the evaporation option is recommended. Proceed to 12.1.5. Otherwise, go to 12.1.4.

12.1.4 *Calcium Phosphate Coprecipitation Option:*

12.1.4.1 Add 0.5 mL of 0.9*M* $Ca(NO_3)_2$ to the beaker.

12.1.4.2 Place the beaker on a hot plate, cover with a watch glass, and heat until boiling.

12.1.4.3 Once the sample boils, take the watch glass off the beaker and lower the heat.

12.1.4.4 Add two to three drops of phenolphthalein indicator and 200 µL of 3.2*M* $(NH_4)_2HPO_4$ solution.

12.1.4.5 Add enough concentrated NH_4OH with a squeeze bottle to reach the phenolphthalein end point (pH ~8.4) and form $Ca_3(PO_4)_2$ precipitate. The NH_4OH should be added very slowly. Stir the solution with a glass rod. Allow the sample to heat gently to digest the precipitate for another 20 to 30 min.

12.1.4.6 If the sample volume is too large to centrifuge the entire sample, allow precipitate to settle until solution can be decanted (30 min to 2 h).

12.1.4.7 If the volume is small enough to centrifuge, decant or aspirate supernatant solution and discard to waste.

12.1.4.8 Transfer the precipitate to a 50 mL or 250-mL centrifuge container, completing the transfer with a few millilitres of water, and centrifuge the precipitate for approximately 10 minutes at 2000 rpm.

12.1.4.9 Decant supernatant solution and discard to waste.

12.1.4.10 Wash the precipitate with an amount of water approximately twice the volume of the precipitate. Mix well using a stirring rod, breaking up the precipitate if necessary. Centrifuge for 5–10 minutes at 2000 rpm. Discard the supernatant solution.

12.1.4.11 Dissolve precipitate in approximately 5-mL concentrated HNO_3.

12.1.4.12 Transfer the solution to a 100-mL beaker.

12.1.4.13 Rinse the centrifuge tube with 2 to 3 mL of concentrated HNO_3 and transfer to the same beaker.

12.1.4.14 Evaporate the solution to dryness and go to 12.2.

12.1.5 *Evaporation Option to Reduce Volume and to Digest Organic Components:*

12.1.5.1 Evaporate the sample to less than 50 mL and transfer to a 100-mL beaker.

NOTE 2—For some water samples, $CaSO_4$ formation may occur during evaporation. If this occurs, use the $Ca_3(PO_4)_2$ precipitation option in 12.1.4.

12.1.5.2 Gently evaporate the sample to dryness and redissolve in approximately 5 mL of concentrated HNO_3.

12.1.5.3 Repeat 12.1.5.2 two more times, evaporate to dryness, and go to 12.2.

12.2 *Actinide Separations Using Extraction Resins:*

12.2.1 Redissolve the $Ca_3(PO_4)_2$ residue or evaporated water sample.

12.2.1.1 Dissolve either residue with 10 mL of 3*M* HNO_3-1.0*M* $Al(NO_3)_3$.

NOTE 3—An additional 5 mL may be necessary if the residue volume is large.

12.2.1.2 Add 2 mL of 0.6*M* ferrous sulfamate to each sample solution. Swirl to mix.

NOTE 4—If the additional 5 mL was used to dissolve the sample in 12.2.1.1, add a total of 3 mL of ferrous sulfamate solution.

12.2.1.3 Add one drop of 1*M* NH_4SCN indicator to each sample and mix.

NOTE 5—The color of the solution turns deep red because of the presence of soluble ferric thiocyanate complex.

12.2.1.4 Add 1 mL of 1*M* ascorbic acid to each solution, swirling to mix. Wait for 2 to 3 minutes.

NOTE 6—The red color should disappear, which indicates reduction of Fe^{+3} to Fe^{+2}. If the red color still persists, then additional ascorbic acid solution has to be added dropwise with mixing until the red color disappears.

NOTE 7—If particles are observed suspended in the solution, centrifuge the sample. The supernatant solution will be transferred to the column in 12.2.3.1. The precipitates will be discarded.

12.2.2 *Setup of and Conditioning DAAP and CMPO Cartridges:*

12.2.2.1 Place the inner tube rack into the vacuum box with 50-mL centrifuge tubes in the rack.

12.2.2.2 Fit the lid to the vacuum system box.

12.2.2.3 Place appropriate connectors into the lid openings.

12.2.2.4 Securely connect a DAAP cartridge to the top of a CMPO cartridge.

12.2.2.5 Securely attach a reservoir to the top of the DAAP cartridge.

12.2.2.6 Securely connect the CMPO cartridge to the vacuum box.

12.2.2.7 Connect the vacuum box to the vacuum source.

12.2.2.8 Seal all unused openings with manifold caps, disposable pipets, a strip of tape, or otherwise as recommended by the box manufacturer.

12.2.2.9 Apply vacuum and ensure proper fitting of the lid.

12.2.2.10 Add 5 mL of 3*M* HNO_3 to the reservoir to precondition the DAAP and CMPO cartridges.

12.2.2.11 Adjust the vacuum pressure to achieve a flow rate of ~1 mL/min.

12.2.2.12 Label the tandem setup with the sample identifier.

NOTE 8—Unless otherwise specified in the procedure, use a flow rate of ~1 mL/min (1 drop/s).

NOTE 9—Unless otherwise specified, allow each addition to the reservoir to reach the top of the cartridge before adding the next solution to the reservoir.

12.2.3 *Preliminary Purification of the Americium Fraction Using DAAP and CMPO Resins:*

12.2.3.1 Transfer the solution from 12.2.1.4 into the reservoir by pouring or using a plastic transfer pipette.

12.2.3.2 Add 5 mL of 3*M* HNO_3 to the beaker (from 12.2.1.4) as a rinse and transfer the rinse solution into the reservoir. The flow rate can be adjusted to ~3 mL/min for this step.

12.2.3.3 Add 5 mL of 3*M* HNO_3 to the reservoir as a second column rinse (flow rate ~3 mL/min).

12.2.3.4 Separate the DAAP cartridge from the CMPO cartridge.

12.2.3.5 Discard the DAAP cartridge and the load and rinse effluent collected so far.

12.2.3.6 Return the collection tube to the rack or place a clean tube in the rack.

12.2.3.7 Place a clean reservoir on the CMPO cartridge.

12.2.4 *Final Americium Separation Using the CMPO Cartridge:*

NOTE 10—If the sample is known not to contain plutonium, Steps 12.2.4.1 and 12.2.4.2 may be omitted.

12.2.4.1 Add 5 mL of 2*M* HNO_3 into the reservoir from 12.2.3.7 and allow to drain, reducing the flow rate to ~1 mL/min by the end of this rinse.

12.2.4.2 Add 5 mL of 2*M* HNO_3-0.1*M* $NaNO_2$ into the reservoir, rinsing the reservoir while adding the 2*M* HNO_3-0.1*M* $NaNO_2$, and allow to drain.

NOTE 11—Sodium nitrite is used to oxidize any Pu^{+3} to Pu^{+4} and enhance the Pu/Am separation.

12.2.4.3 Add 5 mL of 0.5*M* HNO_3 to the reservoir and allow to drain.

NOTE 12—Before conversion to the chloride system, 0.5*M* HNO_3 is used to lower the nitrate concentration.

12.2.4.4 Discard the load and effluent solutions to waste.

12.2.4.5 Place a clean, labeled tube, at least 25-mL capacity, into the rack.

NOTE 13—The effluent from the remaining steps is collected in a single tube and retained.

12.2.4.6 Add 3 mL of 9*M* HCl to the reservoir to convert to the chloride system.

12.2.4.7 Add 20 mL of 4*M* HCl to elute americium.

12.2.4.8 Discard the CMPO cartridge.

12.2.4.9 *Preparation for neodymium fluoride microprecipitation:*

NOTE 14—An alternate neodymium fluoride microprecipitation mounting method can be found in C1163.

(1) Transfer the combined eluates from 12.2.4.6 and 12.2.4.7 to a 50-mL beaker.

(2) Rinse the tube with a few millilitres of water and add to the beaker.

(3) Evaporate the solution to near dryness.

NOTE 15—*Important*—Do not bake the residue.

(4) Allow the beaker to cool slightly and then add a few drops of concentrated HCl followed by 1 mL of water.

(5) Transfer the solution from 12.2.4.9*(4)* to a 10-mL plastic culture tube or 15-mL centrifuge tube. Wash the beaker twice with 1-mL washes of 1*M* HCl. Transfer the washings to the culture tube. Mix by gently swirling the solution in the tube.

(6) Proceed to neodymium fluoride microprecipitation in 12.3.

12.3 *Preparation of the Sample Test Source:*

NOTE 16—The instructions in 12.3.1 – 12.3.17 describe preparation of a single STS. Several STSs can be prepared simultaneously if a multichannel vacuum box (whale apparatus) is available.

12.3.1 Add 100 µL of the neodymium carrier solution to the tube from 12.2.4.9*(5)* with a micropipette. Gently swirl the tube to mix the solution.

12.3.2 Add ten drops (0.5 mL) of concentrated HF to the tube and mix well by gentle swirling.

12.3.3 Cap the tube and place it in a cold-water bath for at least 30 min.

12.3.4 Set up the 25-mm filtration apparatus.

12.3.5 Place a 25-mm polymeric membrane filter face up on the stainless steel screen. Center the filter on the stainless steel screen support and apply the vacuum. Wet the filter with 100 % ethanol, followed by filtered Type I water. (**Warning—**There is no visible difference between the two sides of the filter. If the filter is turned over accidentally, it is recommended that the filter be discarded and a fresh one removed from the container.)

12.3.6 Lock the filter chimney firmly in place on the filter screen and wash the filter with additional filtered Type I water.

12.3.7 Pour 5.0 mL of neodymium substrate solution down the side of the filter chimney avoiding directing the stream at the filter. When the solution passes through the filter, wait at least 15 seconds before the next step. (**Warning—**Directing a stream of liquid onto the filter will disturb the distribution of the precipitate on the filter and render the sample unsuitable for optimum α-spectrometry resolution.)

12.3.8 Repeat 12.3.7 with an additional 5.0 mL of the substrate solution.

12.3.9 Pour the sample from 12.3.3 down the side of the filter chimney and allow the vacuum to draw the solution through.

12.3.10 Rinse the tube from Step 12.3.9 twice with 2 mL of 0.58*M* HF, stirring each wash briefly using a vortex mixer, and pouring each wash down the side of the filter chimney.

12.3.11 Repeat the rinse using 2 mL of filtered Type I water once.

12.3.12 Repeat the rinse using 2 mL of 80 % ethyl alcohol once.

NOTE 17—Using 12.3.10 and 12.3.12 were shown to improve the full-width at half-maximum (FWHM) in the alpha spectrum, providing more consistent peak resolution.

12.3.13 Wash any drops remaining on the sides of the chimney down toward the filter with a few millilitres of 80 % ethyl alcohol.

12.3.14 Without turning off the vacuum, remove the filter chimney.

12.3.15 Turn off the vacuum to remove the filter. Discard the filtrate to waste for future disposal. If the filtrate is to be retained, it should be placed in a plastic container to avoid dissolution of the glass vessel by dilute HF.

12.3.16 Place the filter on a properly labeled mounting disk. Secure with a mounting ring or other device that will render the filter flat for counting.

12.3.17 Let the sample air dry for a few minutes and, when dry, place in a container suitable for transfer and submit for counting.

NOTE 18—Other methods for STS preparation, such as electroplating (see, for example, C1284) or microprecipitation with cerium fluoride, may be used in lieu of the neodymium fluoride microprecipitation, but any such substitution shall be validated.

13. Calculation or Interpretation of Results

13.1 *Equations for Determination of Final Result, Combined Standard Uncertainty, and Radiochemical Yield (if Requested):*

13.1.1 The activity concentration of an analyte and its combined standard uncertainty are calculated using:

$$AC_a = \frac{A_t \times R_a \times D_t \times I_t}{V_a \times R_t \times D_a \times I_a} \quad (1)$$

and

$$u_c(AC_a) = \sqrt{u^2(R_a) \times \frac{A_t^2 \times D_t^2 \times I_t^2}{V_a^2 \times R_t^2 \times D_a^2 \times I_a^2} + AC_a^2 \times \left(\frac{u^2(A_t)}{A_t^2} + \frac{u^2(V_a)}{V_a^2} + \frac{u^2(R_t)}{R_t^2} \right)} \quad (2)$$

where:

AC_a = activity concentration of the analyte at time of count (Bq/L);

A_t = activity of the tracer added to the sample aliquant at its reference date/time (Bq);

R_a = net count rate of the analyte in the defined region of interest (ROI), in counts per second;

R_t = net count rate of the tracer in the defined ROI in counts per second;

V_a = volume of the sample aliquant (L);

D_t = correction factor for decay of the tracer from its reference date and time to the midpoint of the counting period;

D_a = correction factor for decay of the analyte from the time of sample collection (or other reference time) to the midpoint of the counting period, if required;

I_t = probability of α emission in the defined ROI per decay of the tracer (Table 1);

I_a = probability of α emission in the defined ROI per decay of the analyte (Table 1);

$u_c(AC_a)$ = combined standard uncertainty of the activity concentration of the analyte (Bq/L);

$u(A_t)$ = standard uncertainty of the activity of the tracer added to the sample (Bq);

$u(V_a)$ = standard uncertainty of the volume of sample aliquant (L);

$u(R_a)$ = standard uncertainty of the net count rate of the analyte in counts per second; and

$u(R_t)$ = standard uncertainty of the net count rate of the tracer in counts per second.

NOTE 19—The uncertainties of the decay-correction factors and the probability of decay factors are assumed to be negligible.

NOTE 20—The equation for the combined standard uncertainty $[u_c(AC_a)]$ calculation is arranged to eliminate the possibility of dividing by zero if $R_a = 0$.

NOTE 21—The standard uncertainty of the activity of the tracer added to the sample shall reflect that associated with the activity of the standard reference material and any other significant sources of uncertainty such as those introduced during the preparation of the tracer solution (for example, weighing or dilution factors) and during the process of adding the tracer to the sample.

NOTE 22—The alpha spectrum of americium isotopes should be examined carefully and the ROI reset manually, if necessary, to minimize the spillover of ^{241}Am peak into the ^{243}Am peak.

13.1.2 The net count rate of an analyte or tracer and its standard uncertainty can be calculated using:

$$R_x = \frac{C_x}{t_s} - \frac{C_{bx}}{t_b} \quad (3)$$

and

TABLE 1 Alpha Particle Energies and Abundances of Importance[A]

Nuclide	Half-Life (Years)	Λ (s^{-1})	Abundance	α Energy (MeV)
^{241}Am	432.6	5.077×10^{-11}	0.848	5.486
			0.131	5.443
			0.0166	5.388
^{243}Am	7.37×10^{3}	2.98×10^{-12}	0.871	5.275
			0.112	5.233
			0.0136	5.181

[A] Only the most abundant particle energies and abundances have been noted here. Source: www.nndc.bnl.gov/nudat2; December 2013

$$u(R_x) = \sqrt{\frac{C_x+1}{t_s^2}+\frac{C_{bx}+1}{t_b^2}} \quad (4)$$

where:

R_x = net count rate of analyte or tracer in counts per second,
C_x = sample counts in the analyte or the tracer ROI,
t_s = sample count time (s),
C_{bx} = background counts in the same ROI as for x,
t_b = background count time (s), and
$u(R_x)$ = standard uncertainty of the net count rate of tracer or analyte in counts per second. (See Note 23.)

NOTE 23—For methods with very low counts, MARLAP Section 19.5.2.2,[3] recommends adding one count each to the gross counts and the background counts when estimating the uncertainty of the respective net counts. This minimizes negative bias in the estimate of uncertainty and protects against calculating zero uncertainty when a total of zero counts are observed for the sample and background.

13.1.3 If the radiochemical yield of the tracer is requested, the yield and its combined standard uncertainty can be calculated using:

$$RY = \frac{R_t}{A_t \times D_t \times I_t \times \varepsilon} \quad (5)$$

and:

$$u_c(R\ Y) = RY \times \sqrt{\frac{u^2(R_t)}{R_t^2}+\frac{u^2(A_t)}{A_t^2}+\frac{u^2(\varepsilon)}{\varepsilon^2}} \quad (6)$$

where:

RY = radiochemical yield of the tracer expressed as a fraction,
R_t = net count rate of the tracer in counts per second,
A_t = activity of the tracer added to the sample (Bq),
D_t = correction factor for decay of the tracer from its reference date and time to the midpoint of the counting period,
I_t = probability of α emission in the defined ROI per decay of the tracer (Table 1),
ε = detector efficiency expressed as a fraction,
$u_c(RY)$ = combined standard uncertainty of the radiochemical yield,
$u(R_t)$ = standard uncertainty of the net count rate of the tracer in counts per second,
$u(A_t)$ = standard uncertainty of the activity of the tracer added to the sample (Bq), and
$u(\varepsilon)$ = standard uncertainty of the detector efficiency.

13.1.4 If the critical level concentration (S_c – Eq 7) or the minimum detectable concentration (MDC – Eq 9) in terms of Bq / L are requested (at an error rate of 5 %), they can be calculated using Eq 7 and Eq 9. (See Note 24.)

NOTE 24—The formulations for the critical level and minimum detectable concentrations are as recommended in MARLAP Section 20A.2.2, Equation 20.54 and Section 20A.3.2, Equation 20.74, respectively.[3] For methods with very low numbers of counts, these expressions provide better estimates than do the traditional formulas for the critical level and MDC assuming that the observed variance of the background conforms to Poisson statistics. Consult MARLAP when background variance may exceed that predicted by the Poisson model or other decision error rates may apply.

13.1.5 When the Type I decision error rate, α, equals 0.05, $z_{1-\alpha}$ = 1.645, and the constant, d, from the Stapleton approximation is set to 0.4, Eq 7 becomes Eq 8.

13.1.6 When the Type I decision error rate, α, equals 0.05, $z_{1-\alpha}$ = 1.645, and the Type II decision error rate, β, equals 0.05, $z_{1-\beta}$ = 1.645, Eq 9 becomes Eq 10.

where:

R_{ba} = background count rate for the analyte in the defined ROI in counts per second.

$$S_c = \frac{\left[d \times \left(\frac{t_s}{t_b} - 1\right)+\frac{z_{1-\alpha}^2}{4}\times\left(1+\frac{t_s}{t_b}\right)+z_{1-\alpha}\sqrt{(R_{ba}\ t_b + d)\times\frac{t_s}{t_b}\times\left(1+\frac{t_s}{t_b}\right)}\right]\times A_t \times D_t \times I_t}{t_s \times V_a \times R_t \times D_a \times I_a} \quad (7)$$

$$S_c = \frac{\left[0.4 \times \left(\frac{t_s}{t_b} - 1\right)+0.677\times\left(1+\frac{t_s}{t_b}\right)+1.645\sqrt{(R_{ba}\ t_b + 0.4)\times\frac{t_s}{t_b}\times\left(1+\frac{t_s}{t_b}\right)}\right]\times A_t \times D_t \times I_t}{t_s \times V_a \times R_t \times D_a \times I_a} \quad (8)$$

$$MDC = \frac{\left[\frac{(z_{1-\alpha}+z_{1-\beta})^2}{4}\times\left(1+\frac{t_s}{t_b}\right)+(z_{1-\alpha}+z_{1-\beta})\times\sqrt{R_{ba}t_s\times\left(1+\frac{t_s}{t_b}\right)}\right]\times A_t \times D_t \times I_t}{t_s \times V_a \times R_t \times D_a \times I_a} \quad (9)$$

$$MDC = \frac{\left[2.71\times\left(1+\frac{t_s}{t_b}\right)+3.29\times\sqrt{R_{ba}t_s\times\left(1+\frac{t_s}{t_b}\right)}\right]\times A_t \times D_t \times I_t}{t_s \times V_a \times R_t \times D_a \times I_a} \quad (10)$$

14. Report

14.1 The following items should be reported for each result:

14.1.1 Volume of sample used,

14.1.2 Yield of tracer and its uncertainty when required by the Analytical Protocol Specifications or Statement of Work, and

14.1.3 Full width at half maximum (FWHM) of each peak used in the analysis.

14.2 The following conventions should be used for each result:

14.2.1 Result in scientific notation ± combined standard uncertainty.

14.2.2 If solid material was filtered from the solution and analyzed separately, the results of that analysis should be reported separately as Bq/L of the original volume from which the solids were filtered if no other guidance is provided on reporting of results for the solids.

14.2.2.1 *Example*—^{241}Am for Sample 12-1-99.

(1) Filtrate result—$(4.74 \pm .55) \times 10^{-1}$ Bq/L.

(2) Filtered residue result—$(9.3 \pm 1.1) \times 10^{-2}$ Bq/L.

15. Quality Control

15.1 To be certain that analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the following QC procedures and tests shall be followed. The batch size should not exceed 20 samples, not including QC samples.

15.2 *Detector Efficiency*—With the use of a ^{243}Am radiotracer, the determination of the detector efficiency is not necessary. However, when it is necessary to report the chemical yield for a sample, the detector efficiency shall be known. Radiotracers and standards used in this test method shall be traceable to a national standards laboratory such as NIST or NPL.

15.2.1 Two options are recommended for the determination of the detector efficiency: the use of a ^{241}Am traceable plated source having the same active area as the STS and the preparation of three ^{241}Am (with radiotracer) STSs following Step 12.2.4.9. Determine the mean detector efficiency and associated standard error of the mean of three measurements of the plated source or the three STSs. The source-to-detector distance for the detector efficiency measurements shall be the same as for the routine samples.

15.3 *Initial Demonstration of Laboratory/Instrument Capability:*

15.3.1 If a laboratory or analyst has not performed this test before or if there has been a major change in the measurement system, for example, significant instrument change, new instrument, and so forth, a precision and bias study shall be performed to demonstrate laboratory/instrument capability.

15.3.2 Analyze seven replicates of a standard solution prepared from an independent reference material (IRM) containing accurately known concentrations of ^{241}Am at concentrations sufficient to minimize the counting uncertainty to less than 2 % at two sigma. Each replicate shall be taken through the complete analytical test method including any sample preservation and pretreatment steps. The matrix and chemistry of the solution should be equivalent to that of the samples.

15.3.3 Calculate the mean and standard deviation of the replicate values and compare to the acceptable ranges of precision and mean bias of 10 and ±10 %, respectively, based on a review of the collaborative study data. Test Method D5847 should be consulted on the manner by which precision and mean bias are determined from the initial demonstration study.

15.3.4 This test method shall not be used for official samples until precision and bias requirements are met.

15.4 *Laboratory Control Sample (LCS):*

15.4.1 To ensure that the test method is in control, analyze a LCS with each batch of no more than 20 samples. The LCS should contain ^{241}Am at a concentration exceeding approximately two to five times the client specified MDC or the laboratory's default LCS concentration. The LCS shall be taken through all the steps of the method. The result obtained for the LCS shall fall within the limit of ±25 % of the expected value.

15.4.2 If the result is not within these limits, reporting of the results is halted until the problem is resolved. An indication of the occurrence should accompany the reported results.

15.5 *Method Blank (Blank):*

15.5.1 Analyze a reagent water test blank with each batch of no more than 20 samples. The concentration of the analyte found in the blank should be less than one half of the customer's MDC specification or the laboratory's default MDC specification.

15.5.2 The method blank shall be taken through all the steps of the test method.

15.5.3 If the concentration of analyte is found above the one-half MDC limit, the results shall be flagged and discussed in the case narrative.

15.6 *Matrix Spike:*

15.6.1 The performance of a matrix spike analysis with every batch is not required given the use of a radiotracer with each sample. The tracer radiochemical yield would indicate any problems with interferences in a specific sample matrix. Section 13.1.3 addresses the calculation of the radiochemical yield.

15.7 *Duplicate:*

15.7.1 To check the precision of the test method, analyze a sample in duplicate with each batch of no more than 20 samples.

15.7.2 In those cases in which there is insufficient sample to allow performance of a duplicate sample analysis, a duplicate analysis of a laboratory control sample duplicate (LCS-D) shall be performed.

15.7.3 Determine the statistical agreement of the two-sample results by calculating the duplicate error ratio (DER) according to the following equation:

$$DER = \frac{|AC_{original} - AC_{dup}|}{\sqrt{u_c^2(AC_{original}) + u_c^2(AC_{dup})}} \quad (11)$$

where:

$AC_{original}$ = original sample activity concentration,

AC_{dup} = duplicate sample activity concentration,
$u_c(AC_{original})$ = combined standard uncertainty of the original sample result, and
$u_c(AC_{dup})$ = combined standard uncertainty of the duplicate sample result.

15.7.4 If the result exceeds the DER precision limit of three, all samples in the batch shall be reanalyzed or the results shall be flagged with an indication that they do not fall within the performance criteria of the method. In addition, an explanation as to why the precision criterion was not met shall be included in the case narrative for the batch of samples.

15.8 *IRM:*

15.8.1 To verify the quantitative value produced by the test method, analyze an IRM submitted on at least single-blind basis (if practical) to the laboratory at least once per quarter that samples are analyzed.

15.8.2 The concentration of analyte in the national standards laboratory traceable reference material should be appropriate to the typical purpose for which the method is used. The value obtained shall demonstrate acceptable performance as defined by the program or the outside source.

15.8.3 In the absence of other acceptance criteria for the IRM sample, compare the IRM sample result to the IRM known values as follows:

$$R = \frac{|IRM_{measured} - IRM_{known}|}{\sqrt{u_c^2(IRM_{measured}) + u_c^2(IRM_{known})}} \quad (12)$$

where:

R = relative difference,
$IRM_{measured}$ = measured IRM concentration,
IRM_{known} = known IRM concentration,
$u_c(IRM_{measured})$ = combined standard uncertainty of the measured IRM concentration, and
$u_c(IRM_{known})$ = combined standard uncertainty of the known IRM concentration.

15.8.4 The value of R should be less than or equal to 3.0. If the value of R is greater than 3.0, the method should be investigated to determine the cause of the discrepancy.

16. Precision and Bias

16.1 A single operator test was conducted by a laboratory not familiar with the test method. The following test levels in Atlanta drinking water (Table 2) were analyzed by the laboratory:

16.1.1 Three concentration test levels with seven replicates for each test level.

16.1.1.1 *^{241}Am concentrations of 0.279, 0.559, and 1.68 Bq/L*—The test samples included interfering radionuclides of ^{238}Pu and natural uranium at concentrations given in Table 3.

(1) Uncertainty stated with a coverage factor of $k = 2$.

16.1.2 Required minimum detectable concentration test level with ten samples.

16.1.2.1 ^{241}Am required MDC level of 0.056 Bq/L.

16.1.3 Blank test level of seven Atlanta municipal water blanks.

TABLE 2 Composition of Atlanta Drinking Water Used for the SOT

NOTE 1—Analyses conducted by independent laboratories.

Metals by ICP	Concentration (mg/L)[A]
Silicon	3.18
Aluminum	<0.200
Barium	0.0133
Calcium	9.38
Iron	<0.100
Magnesium	<0.500
Potassium	<0.500
Sodium	<0.500
Inorganic Anions	
Chloride	12.7
Sulfate	15.6
Nitrogen, nitrate (as N)	1.19
Carbon Dioxide	
Bicarbonate alkalinity	23.8
Carbonate alkalinity	<3.00
Radionuclides	Concentration (Bq/L)[B]
Uranium 234, 235, 238	<0.0004, <0.0004, <0.0004
Plutonium 238, 239+240	<0.0007, <0.0007
Americium 241	<0.0007
Strontium 90	<0.011
Radium 226[C]	0.004 ± 0.010 –0.011 ± 0.017

[A] Values below the reporting level are presented as less than (<) values. No measurement uncertainty was reported with values greater than the "Reporting Level."
[B] Reported values represent the calculated minimum detectable concentration (MDC) for the radionuclide(s). Values rounded in conversion of pCi to Bq.
[C] Two samples analyzed. Expanded uncertainty (k = 2) as reported by the laboratory.

TABLE 3 Radionuclide Concentrations for the Three Test Levels

Test Level	Sample Size Prepared by Source Provider (L)	Number of Samples Tested	Nuclide(s)	Known Values Test Level Concentration(s) (Bq/L) ± $2u_c$ *
1	1	7	Am-241	0.2794 ± 0.0056
			Pu-238	0.2760 ± 0.0081
			U-238 (nat)	0.3693 ± 0.0015
			U-234	0.3682 ± 0.0015
2	1	7	Am-241	0.559 ± 0.011
			Pu-238	0.551 ± 0.015
			U-238 (nat)	0.736 ± 0.030
			U-234	0.736 ± 0.030
3	1	7	Am-241	1.680 ± 0.033
			Pu-238	1.661 ± 0.048
			U-238 (nat)	2.224 ± 0.093
			U-234	2.224 ± 0.093
Blanks	1	7	Blanks of Atlanta drinking water	See Table 2

16.2 The results for the three ^{241}Am concentration test levels are listed in Table 4. The sample volume was 200 mL and the counting time was 10 800 s (180 min).

16.3 The test results for the MDC test level of 0.056 Bq/L are listed in Table 5. The sample volume was 200 mL and the counting time was 10 800 s.

16.4 The test results for the method blanks are listed in Table 6. The sample volume was 200 mL and the counting time was 10 800 s.

16.5 The ^{241}Am results for the seven blank samples (Table 6), ten MDC samples (Table 5), and the PT samples on the

TABLE 4 Sample Results for the Three ^{241}Am Test Concentrations Levels

NOTE 1—Reported CSU is stated at a coverage factor of k = 1. Uncertainties of the known values are stated in Table 3.

Test Level 1 0.2794 Bq/L			Test Level 2 0.559 Bq/L			Test Level 3 1.680 Bq/L		
Sample #	Bq/L Measured	CSU	Sample #	Bq/L Measured	CSU	Sample #	Bq/L Measured	CSU
1	0.307	0.035	1	0.529	0.048	1	1.71	0.10
2	0.232	0.029	2	0.507	0.044	2	1.66	0.10
3	0.302	0.034	3	0.570	0.052	3	1.73	0.10
4	0.267	0.032	4	0.544	0.048	4	1.64	0.10
5	0.374	0.041	5	0.511	0.048	5	1.67	0.10
6	0.248	0.030	6	0.507	0.044	6	1.75	0.10
7	0.286	0.015	7	0.599	0.048	7	1.69	0.10

TABLE 5 Reported Results for Samples Containing ^{241}Am at the Required MDC of 0.0562 ± 0.0011 Bq/L (k = 2)

Sample ID	Concentration (Bq/L)	CSU[A] (Bq)
MA1	0.050	0.015
MA2	0.058	0.016
MA3	0.047	0.014
MA4	0.059	0.016
MA5	0.087	0.019
MA6	0.061	0.016
MA7	0.045	0.014
MA8	0.054	0.016
MA9	0.050	0.015
MA10	0.029	0.012
Mean[B]	0.054	
Standard deviation of results	0.015	

[A] k = 1 (coverage factor of 1).
[B] Mean and standard deviation were calculated before rounding.

TABLE 6 Reported ^{241}Am Concentrations for Blank Samples

Sample ID	Concentration (Bq/L)	CSU[A] (Bq/L)
03	0.0056	0.0056
07	0.0130	0.0070
10	-0.0004	0.0037
13	0.0015	0.0048
04	0.0074	0.0063
08	0.0141	0.0081
12	0.0170	0.0085
Mean[B]	0.0081	
Standard deviation of seven samples	0.0067	

[A] k = 1 or coverage factor of 1.
[B] Mean and standard deviation were calculated before rounding.

three test levels (Table 4) were evaluated for bias and precision. The results and interpretation of the evaluation are presented in Table 7.

16.6 An evaluation of the data indicates that there was no statistical bias for test levels 1, 2, and 3 and the MDC test level. Although a positive bias was indicated for the blank Atlanta water samples, the magnitude of the bias was insignificant compared to the concentrations of test levels 1, 2, and 3.

16.7 The mean and standard deviation of the radiochemical yield for the 64 test and QC samples analyzed in the single operator test were 94.7 ± 6.4 % (k = 1).

17. Keywords

17.1 alpha spectrometry; americium-241; curium; extraction chromatography; radioactivity; radiochemistry

TABLE 7 Bias and Precision Evaluation of the ^{241}Am Method[A]

Test Level	Number of Samples	Known Value ± CSU (k = 2) (Bq/L)	Mean of Measurements (Bq/L)	Standard Deviation[B] Bq/L	Bias % Difference from Known	Precision % Coefficient of Variation
Blanks	7	0.0	0.0081	0.0067	–	–
MDC	10	0.0562 ± 0.0011	0.054	0.015	-3.9	27
1	7	0.274 ± 0.0056	0.289	0.048	+3.3	17
2	7	0.559 ± 0.011	0.538	0.036	-3.7	6.6
3	7	1.680 ± 0.033	1.691	0.037	+0.7	2.2

[A] k = 1 (coverage factor of 1).
[B] Mean and standard deviation were calculated before rounding.

APPENDIX

(Nonmandatory Information)

X1. DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

X1.1 *Spectrum from a Processed Sample*—See Fig. X1.1.

X1.2 *Decay Scheme*—See Fig. X1.2.

X1.3 *Separation Scheme and Timeline for Determination of Am in Water Samples*—See Figs. X1.3 and X1.4.

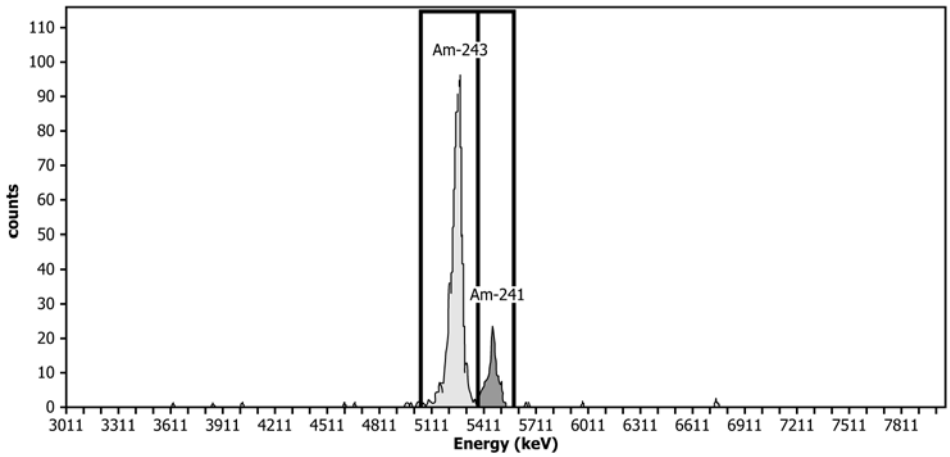

FIG. X1.1 Spectrum from a Processed Sample

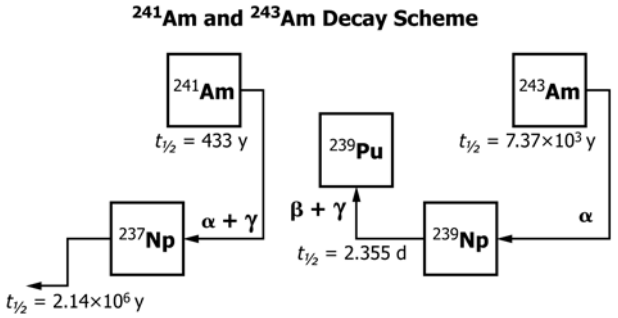

FIG. X1.2 Decay Scheme

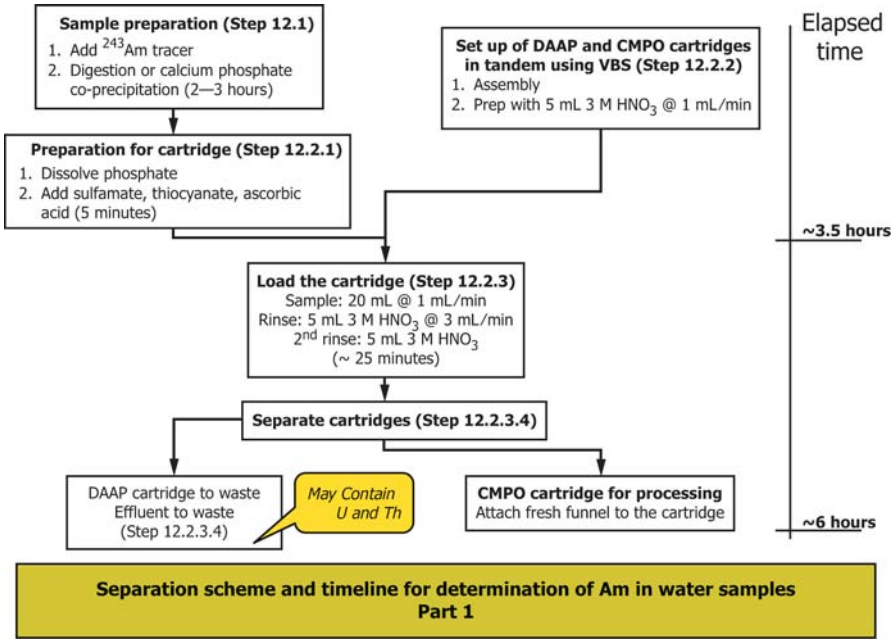

FIG. X1.3 Separation Scheme and Timeline for Determination of Am in Water Samples Part 1

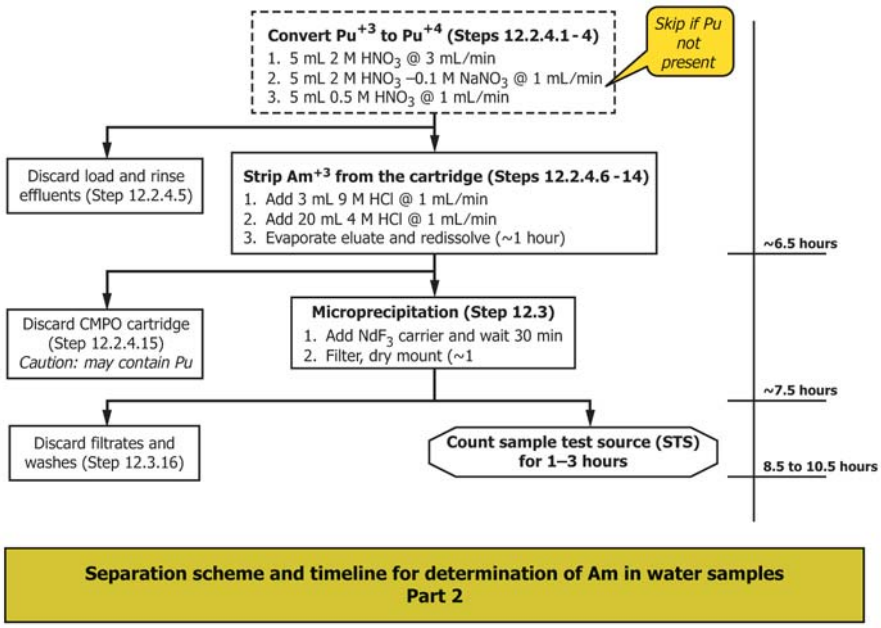

FIG. X1.4 Separation Scheme and Timeline for Determination of Am in Water Samples Part 2

BIBLIOGRAPHY

(1) G-03, V.1, "Microprecipitation Source Preparation for Alpha Spectrometry," HASL-300, 28th ed., February 1997.

(2) Reference ACW03 VBS, Rev. 1.6, "Americium, Plutonium, and Uranium in Water (with Vacuum Box System)," Eichrom Technologies, Inc., Lisle, IL, February 2005.

(3) VBS01, Rev.1.4, "Setup and Operation Instructions for Eichrom's Vacuum Box System," Eichrom Technologies, Inc., Lisle, IL, January 2014.