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Rapid Radiochemical Method for Curium-244 in Water Samples for Environmental Remediation Following Radiological Incidents

U.S. Environmental Protection Agency

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Rapid Radiochemical Method for Curium-244 in Air Particulate Filters, Swipes and Soils

Revision History

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RAPID RADIOCHEMICAL METHOD FOR CM-244 IN WATER SAMPLES FOR ENVIRONMENTAL REMEDIATION FOLLOWING RADIOLOGICAL INCIDENTS

1. Scope and Application

- 1.1. This method provides for the rapid determination of ^{244}Cm in water samples.
- 1.2. The method uses radiochemical separation techniques to rapidly isolate curium from a water matrix using ^{243}Am tracer as a yield monitor.
- 1.3. A sample test source is prepared by microprecipitation. The test source is counted by alpha spectrometry for ^{244}Cm .
 - 1.3.1. Cm-243 emits alpha particles that are isoenergetic with ^{244}Cm . Alpha spectrometry measurements that show activity in the region of interest for ^{244}Cm should be reported as $^{244/243}\text{Cm}$.
- 1.4. This method is capable of achieving a required method uncertainty for ^{244}Cm of 2.0 pCi/L at an analytical action level of 15 pCi/L. To attain the stated measurement quality objectives (MQOs), a sample volume of approximately 0.2 L and count time of at least 4 hours are recommended. Sample count times may vary based on differences in instrument parameters such as detection efficiency and background.
- 1.5. The ^{244}Cm method was single-laboratory 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* (Reference 16.1) and Chapter 6 of the *Multi-Agency Radiological Laboratory Analytical Protocols Manual* (MARLAP, Reference 16.2).
 - 1.5.1. Since californium and americium track closely with curium through the chemical separation, it may be possible to determine isotopes of californium, as well as isotopes of americium (e.g., ^{241}Am) that may be present in the sample test source. The specific method performance (yield, required method uncertainty [u_{MR}], minimum detectable activity, and critical level) for other isotopes of californium, americium, or curium (e.g., ^{249}Cf , ^{241}Am , or $^{244/243}\text{Cm}$) must be validated by the laboratory prior to performing determinations for these radionuclides).
 - 1.5.2. The sample turnaround time and throughput may vary based on additional project MQOs, the time for analysis of the sample test source, and initial sample weight / volume.
 - 1.5.3. The method must be validated prior to use following the protocols provided in *Method Validation Guide for Qualifying Methods Used by Radiological Laboratories Participating in Incident Response Activities* (Reference 16.1).

2. Summary of Method

- 2.1. This method is based on the use of extraction chromatography resins (TEVA[®] + DGA Resins) to isolate and purify curium by removing interfering radionuclides and other matrix components and prepare the curium fraction for counting by alpha spectrometry. The method utilizes vacuum-assisted flow to improve the speed of the

separations. An ^{243}Am tracer is equilibrated with the water sample and used as a yield monitor. Following chemical separation of Cm and Am, the sample test source (STS) is prepared by microprecipitation with cerium fluoride (CeF_3). The alpha emissions from the source are measured using an alpha spectrometer and used to calculate the activity of ^{244}Cm in the sample.

3. Definitions, Abbreviations, and Acronyms

- 3.1. Analytical Protocol Specifications (APS). The output of a directed planning process that contains the project's analytical data needs and requirements in an organized, concise form.
- 3.2. Analytical Action Level (AAL). The term "analytical action level" is used to denote the value of a quantity that will cause the decision-maker to choose one of the alternative actions.
- 3.3. Discrete Radioactive Particles (DRPs or "hot particles"). Particulate matter in a sample of any matrix where a high concentration of radioactive material is contained in a tiny particle (μm range).
- 3.4. *Multi-Agency Radiological Analytical Laboratory Protocols Manual (MARLAP)* provides guidance for the planning, implementation, and assessment phases of those projects that require the laboratory analysis of radionuclides (Reference 16.2).
- 3.5. Measurement Quality Objective (MQO). MQOs are the analytical data requirements of the data quality objectives and are project- or program-specific. They can be quantitative or qualitative. MQOs serve as measurement performance criteria or objectives of the analytical process.
- 3.6. Required Method Uncertainty (u_{MR}). The required method uncertainty is a target value for the individual measurement uncertainties and an estimate of uncertainty (of measurement) before the sample is actually measured. The required method uncertainty is applicable below an AAL.
- 3.7. Required Relative Method Uncertainty (ϕ_{MR}). The required relative method uncertainty is the u_{MR} divided by the AAL and is typically expressed as a percentage. It is applicable above the AAL.
- 3.8. Sample Test Source (STS). This is the final form of the sample that is used for nuclear counting. This form is usually specific for the nuclear counting technique used in the method such as a solid deposited on a filter for alpha spectrometry analysis.

4. Interferences

4.1. Radiological

- 4.1.1. The alpha emissions from ^{243}Cm fall in the same region as ^{244}Cm and cannot be differentiated from those of ^{244}Cm using alpha spectrometric determinations.
 - 4.1.1.1. Although the ^{244}Cm and ^{243}Cm alpha emissions overlap, monitoring the region of the spectrum between 5.8 and 6.0 MeV for less intense emissions of ^{243}Cm may qualitatively indicate the presence of ^{243}Cm in a sample.

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- 4.1.1.2. Alpha spectrometry measurements that show activity in the region of interest for ^{244}Cm should be reported as $^{244/243}\text{Cm}$.
 - 4.1.2. Americium and californium are chemical analogs of curium in the separations scheme used for this analysis. Several isotopes of californium or americium emit alpha particles within the region of interest for ^{244}Cm . These include ^{249}Cf and ^{251}Cf . If high levels of californium could be present in samples, alpha spectrometry results should be monitored for other isotopes of californium.
 - 4.1.3. Americium ^{243}Am may be present in certain sources that contain ^{244}Cm . In cases where ^{243}Am is observed or suspected to be present in samples, ^{241}Am may be used in place of ^{243}Am as the yield tracer. Although there is no reason to expect different performance, the approach should be validated by the laboratory prior to implementing.
 - 4.1.4. Radionuclides of other elements (or their short-lived progeny) that emit alpha particles that are isoenergetic with ^{244}Cm (e.g., ^{227}Th or ^{225}Ac at 5.8 MeV) must be chemically separated to prevent positive interference with the measurement. This method separates these radionuclides effectively. For example, a thorium removal rinse is performed on DGA Resin in the event that thorium passes through TEVA[®] Resin onto DGA Resin.
 - 4.1.5. Curium present as a solid (e.g., in DRPs) will not be chemically available and will not be determined unless it is dissolved prior to chemical separation.
 - 4.1.6. Vacuum box lid and holes should be cleaned frequently to prevent cross-contamination of samples.
 - 4.2. Non-Radiological:
 - 4.2.1. Anions that can complex curium and americium, including fluoride and phosphate, may lead to depressed yields. Aluminum in the load solution will complex both fluoride and residual phosphate.
 - 4.2.2. High levels of calcium can have an adverse impact on curium and americium retention on DGA Resin. Calcium retention is minimized and curium and americium affinity enhanced by increasing nitrate concentrations in the load and initial rinse solutions. A dilute nitric acid rinse is performed on DGA Resin to remove calcium that could otherwise end up in the sample test source as the fluoride. For samples containing elevated concentrations of calcium, it may be advisable to increase the volume of this rinse step slightly to better remove calcium ions and possibly improve alpha peak resolution. This modification must be validated by the laboratory prior to use with samples.
5. Safety
 - 5.1. General
 - 5.1.1. Refer to your safety manual for concerns of contamination control, personal exposure monitoring, and radiation dose monitoring.

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- 5.1.2. Refer to your laboratory's chemical hygiene plan (or equivalent) for general safety rules regarding chemicals in the workplace.
 - 5.2. Radiological
 - 5.2.1. Hot particles (DRPs)
 - 5.2.1.1. Hot particles, also termed "discrete radioactive particles" (DRPs), are small, usually much smaller than 1 mm. Typically, DRPs are not evenly distributed in the media and their radiation emissions are anisotropic (i.e., not uniform in all directions).
 - 5.2.1.2. Samples containing measureable activity of ^{244}Cm may have DRPs. If suspended solids are removed by filtration, they may be checked for potential radioactivity.
 - 5.2.2. For samples with detectable activity concentrations of these radionuclides, labware should be used only once due to potential for cross contamination.
 - 5.3. Procedure-Specific Non-Radiological Hazards: Particular attention should be paid to the use of hydrofluoric acid (HF). HF is an extremely dangerous chemical used in the preparation of some of the reagents and in the microprecipitation procedure. Appropriate personal protective equipment (PPE) must be used in strict accordance with the laboratory safety program specification.
6. Equipment and Supplies
 - 6.1. Alpha spectrometer calibrated for use over a range that at minimum includes 4.5 and 7.0 MeV.
 - 6.2. Analytical balance with minimum 10^{-2} g readability.
 - 6.3. Centrifuge tubes, 225-milliliter (mL), 50-mL capacity, or equivalent.
 - 6.4. Centrifuge, to accommodate centrifuge tubes.
 - 6.5. Heat lamp.
 - 6.6. Hot Plate.
 - 6.7. Laboratory ware of plastic, glass, or Teflon; 150-, 250-, 500- and 1,000-mL capacities, as needed.
 - 6.8. Pipettor, electronic, and appropriate plastic tips, 1-10 mL as needed.
 - 6.9. Pipettors and appropriate plastic tips, 100-microliter (μL), 200- μL , 500- μL and 1-mL, or equivalent, as needed.
 - 6.10. Sample test source mounts:
 - 6.10.1. Polypropylene filter, 0.1 micrometer (μm) pore size, 25-mm diameter, or equivalent.
 - 6.10.2. Stainless steel planchets, adhesive backed disks (e.g., Environmental Express, Inc., Charleston, SC, part number R2200) or equivalent, calibrated for 25-mm filter geometry.
 - 6.11. Tweezers.
 - 6.12. Vacuum box system
 - 6.12.1. Vacuum box/rack (e.g., Eichrom Technologies, Inc., Lisle, IL, part number AC-24-BOX), or equivalent.

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- 6.12.2. Cartridge reservoirs, 10 or 20 mL syringe style with locking device, or columns (e.g., empty Luer-lock tip, Image Molding, Denver, CO part number CC-10-M) plus 12 mL reservoirs (e.g., Image Molding, Denver, CO, part number CC-06-M), or equivalent.
 - 6.12.3. Vacuum box tips, white inner tubes, Eichrom Technologies, Inc., Lisle, IL, part number AC-1000-TUBE-PE, or perfluoroalkoxy (PFA) 5/32" × 1/4" heavy-wall tubing connectors, natural, Cole Parmer Instrument Company, LLC, Vernon Hills, IL, part number 00070EE, cut to 1 inch, or equivalent.
 - 6.12.4. Vacuum box tips, yellow outer, Eichrom Technologies, Inc., Lisle, IL, part number AC-1000-OT, or equivalent.
 - 6.12.5. Laboratory vacuum source.
- 6.13. Vortex mixer.

7. Reagents and Standards

NOTE: All reagents are American Chemical Society (ACS) reagent grade or equivalent unless otherwise specified.

NOTE: Unless otherwise indicated, all references to water should be understood to mean Type I Reagent water (ASTM D1193, Reference 16.4). All solutions used in microprecipitation should be prepared with water filtered through a 0.45 µm (or better) filter.

NOTE: Low-levels of uranium are typically present in $\text{Al}(\text{NO}_3)_3$.

- 7.1. Aluminum nitrate solution, 2 M: Add 750 g of aluminum nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$) to ~500 mL of water and dilute to 1 liter with water.
- 7.2. Americium-243 tracer solution: 10-40 disintegrations per minute (dpm) of ^{243}Am per aliquant.
- 7.3. Ammonium hydrogen phosphate, 3.2 M: Dissolve 106 g of $(\text{NH}_4)_2\text{HPO}_4$ in 200 mL of water, heat gently to dissolve and dilute to 250 mL with water.
- 7.4. Ammonium hydroxide, 15 M: Concentrated NH_4OH .
- 7.5. Ascorbic acid, 1.5 M: Dissolve 66 g $\text{C}_6\text{H}_8\text{O}_6$ in 200 mL of water, warming gently to dissolve, and dilute to 250 mL with water. Shelf life is 30 days or less.
- 7.6. Calcium nitrate, 1.25 M: Dissolve 73.8 g of $\text{Ca}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$ in 100 mL of water and dilute to 250 mL with water.
- 7.7. Cerium carrier, 0.5 mg Ce/mL: dissolve 0.16 g $\text{Ce}(\text{NO}_3)_3 \cdot 6 \text{H}_2\text{O}$ in 50 mL water and dilute to 100 mL with water.
- 7.8. Curium-244 standard solution: 10-40 dpm of ^{244}Cm per aliquant.
- 7.9. DGA Resin, normal, 2-mL cartridge, 50- to 100-µm mesh size, Eichrom Technologies, Inc., Lisle, IL, part number DN-R50-S, or equivalent.
- 7.10. Ethanol, 95%: Reagent $\text{C}_2\text{H}_5\text{OH}$, or mix 95 mL 100% ethanol and 5 mL water.
- 7.11. Hydrochloric acid, 12 M: Concentrated HCl.
 - 7.11.1. Hydrochloric acid, 0.25 M: Add 21 mL of concentrated HCl to 500 mL of water and dilute with water to 1 L.
 - 7.11.2. Hydrochloric acid, 4 M: Add 333 mL of concentrated HCl to 500 mL of water and dilute with water to 1 L.

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- 7.12. Hydrofluoric acid, 28M: Concentrated HF
 - 7.13. Hydrogen peroxide, 30 weight percent (wt. %) (H_2O_2).
 - 7.14. Iron carrier, 4 mg/mL: Dissolve 14 g of ferric nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$) in 300 mL water and dilute to 500 mL with water.
 - 7.15. Nitric acid, 16 M: Concentrated HNO_3 .
 - 7.15.1. Nitric acid, 0.1 M: Add 6.3 mL of concentrated HNO_3 to 700 mL of water and dilute to 1 L with water.
 - 7.15.2. Nitric acid, 3 M: Add 190 mL of concentrated HNO_3 to 700 mL of water and dilute to 1 L with water.
 - 7.15.3. Nitric acid, 6 M: Add 380 mL of concentrated HNO_3 to 500 mL of water and dilute to 1 L with water.
 - 7.16. Nitric acid 3 M – Aluminum nitrate solution 1 M: Add equal volumes of 6 M HNO_3 and 2 M $\text{Al}(\text{NO}_3)_3$.
 - 7.17. Nitric acid, 3 M – hydrofluoric acid, 0.25 M: Add 8.9 mL of concentrated HF and 190 mL of concentrated HNO_3 to 700 mL of water. Dilute to 1 liter with water and mix well.
 - 7.18. Phenolphthalein indicator solution, 0.5 wt. % ($\text{C}_{20}\text{H}_{14}\text{O}_4$): Dissolve 0.5 g in 100 mL ethanol (95%).
 - 7.19. Sodium nitrite solution, 3.5 M: Dissolve 6.1 g of NaNO_2 in 25 mL of water. Prepare fresh daily.
 - 7.20. Sulfamic acid solution, 1.5 M: Dissolve 72.8 g of H_3NSO_3 in 400 mL of water and dilute to 500 mL with water.
 - 7.21. TEVA[®] Resin, 2-mL cartridge, 50- to 100- μm mesh size, Eichrom Technologies, Inc., Lisle, IL, part number TE-R50-S and TE-R200-S, or equivalent.
8. Sample Collection, Preservation, and Storage
 - 8.1. Water samples:
 - 8.1.1. No sample preservation is needed if sample analysis is initiated within three days of sample collection.
 - 8.1.2. If sample analysis is not started within three days of sample collection, add concentrated HNO_3 to achieve a $\text{pH} < 2$ and then store for at least 16 hours prior to analysis.
 - 8.1.3. If the concentration of americium in the dissolved fraction is sought, the insoluble fraction must be removed by filtration before preserving with acid.
9. Quality Control
 - 9.1. Batch quality control results shall be evaluated and meet applicable Analytical Protocol Specifications (APS) prior to release of unqualified data. In the absence of project-defined APS or a project specific quality assurance project plan (QAPP), the quality control sample acceptance criteria defined in the laboratory quality manual and procedures shall be used to determine acceptable performance for this method.

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- 9.1.1. A Laboratory Control Sample (LCS) shall be run with each batch of samples. The concentration of the LCS should be at or near the action level or level of interest for the project.
- 9.1.2. One method blank shall be run with each batch of samples. The laboratory blank should consist of demineralized water.
- 9.1.3. One laboratory duplicate shall be run with each batch of samples. The laboratory duplicate is prepared by removing an aliquant from the original sample container.
- 9.1.4. A matrix spike sample is not required as a chemical yield tracer is used in each sample.
- 9.2. The source preparation method should produce a sample test source in which the full width-at-half-maximum (FWHM) for the tracer peak is less than 100 keV.¹
 - 9.2.1. Review each spectrum for evidence of peaks that overlap or evidence of non-analyte activity that interferes with tracer or analyte peaks.
 - 9.2.2. The sample test source may require reprocessing to remove interfering mass if the FWHM limit cannot be achieved and peak overlap or non-analyte peaks impact the quantification of ²⁴⁴Cm.

10. Calibration and Standardization

- 10.1. Set up the alpha spectrometry system according to the manufacturer's recommendations consistent with ASTM Standard Practice D7282, Section 9.3, "Alpha Spectrometry Initial Instrument Set-up" (Reference 16.3). The energy range of the spectrometry system should, at minimum, include the range that encompasses 4.5 and 7.0 MeV.
- 10.2. Establish initial instrument quality controls as described in ASTM Standard Practice D7282, Sections 10-15, "Initial Instrument Quality Control Testing" (Reference 16.3)
- 10.3. Calibrate each detector used to count samples according to ASTM Standard Practice D7282, Section 18, "Alpha Spectrometry Instrument Calibrations" (Reference 16.3).
- 10.4. Perform Continuing Instrument Quality Control Testing according to ASTM Standard Practice D7282, Sections 20, 21, and 24, "Continuing Instrument Quality Control Testing" and "Quality Control for Alpha Spectrometry Systems" (Reference 16.3).

11. Procedure

11.1. Rapid Curium Separation using TEVA[®] and DGA Resins

NOTE: This method addresses the analysis of soluble curium only. Solid material, if present, must be removed from the sample prior to aliquanting by filtering the unpreserved sample aliquant through a 0.45- μ m filter. The solid material may be screened for radioactivity or saved for potential future analysis.

11.1.1. Aliquanting and Preparation

- 11.1.1.1. Aliquant 200 mL of sample into a 225-mL centrifuge tube.

¹ This helps minimize interference from alpha-emitting isotopes with potentially overlapping energies.

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- 11.1.1.2. Aliquant a second 200 mL portion of one sample into a 225-mL centrifuge tube as a sample duplicate.
- 11.1.1.3. Add 200 mL reagent water to an empty 225-mL centrifuge tube as a reagent blank
- 11.1.1.4. Add 200 mL reagent water to an empty 225-mL centrifuge tube for the LCS.
- 11.1.1.5. Acidify each sample with concentrated HNO₃ to a pH of less than 2.0 by adding HNO₃. This usually requires about 0.5 mL of HNO₃.
- 11.1.1.6. Add 10-40 dpm ²⁴⁴Cm standard solution to the LCS centrifuge tube, following laboratory protocol.
- 11.1.1.7. Add 10-40 dpm ²⁴³Am tracer to the blank, LCS, and sample and sample duplicates, following laboratory protocol.
- 11.1.1.8. Add 1 mL of 1.5 M Ca(NO₃)₂, 3 mL of 3.2 M (NH₄)₂HPO₄ solution and 2–3 drops of phenolphthalein indicator to each centrifuge tube.
- 11.1.1.9. Slowly add concentrated NH₄OH to each centrifuge tube with a squeeze bottle. Add enough NH₄OH to reach a dark pink phenolphthalein end point and form Ca₃(PO₄)₂ precipitate. Cap and mix tubes and centrifuge at 2000 revolutions per minute (rpm) or more for ~5 minutes.

Note: If a sample aliquant larger than 200 mL is needed, the aliquant may be added to a large beaker, heated on a hot plate to near boiling with reagents added, and allowed to cool and settle. After pouring off enough of the supernate, the precipitate may be transferred to a 225 mL tube, rinsing the beaker well with water, and centrifuged.

- 11.1.1.10. Decant supernatant solution and discard to waste.

11.1.2. Preparation of the Load Solution

- 11.1.2.1. Dissolve the calcium phosphate precipitate with 15 mL of 3 M HNO₃ - 1.0 M Al(NO₃)₃. If the residue volume is large, or if residual solids remain, an additional 5 mL may be needed to obtain complete dissolution.
- 11.1.2.2. Add 0.5 mL of 1.5 M sulfamic acid to each sample. Swirl to mix.
NOTE: If elevated levels of ²³⁷Np are potentially present in the sample, also add 0.5 mL of 4 mg/mL iron carrier to enhance neptunium (Np) reduction to Np⁴⁺. The addition of ascorbic acid in the next step will convert Fe³⁺ to Fe²⁺ and ensure removal of Np on TEVA[®] Resin.
- 11.1.2.3. Add 1.25 mL of 1.5 M ascorbic acid to each sample. Swirl to mix. Wait 3 minutes.

NOTE: Plutonium (Pu), if present, will be adjusted to Pu⁴⁺ to ensure retention and removal on TEVA[®] Resin. A small amount of brown fumes results from nitrite reaction with sulfamic acid. The solution should clear with swirling. If the solution does not clear (is still dark) an additional small volume of sodium nitrite may be added to clear the solution.

11.1.2.4. Add 1 mL of 3.5 M NaNO₂ to each sample. Swirl to mix.

NOTE: The load solution nitrate concentration is increased after valence adjustment to provide greater retention of Am and more effective elution of calcium ions on DGA Resin.

11.1.2.5. Add 1.5 mL concentrated HNO₃ to each sample and swirl to mix.

NOTE: The steps in this section were optimized for a commercially available filtration system. Other vacuum systems may be substituted here. The cartridges may be set up and conditioned with nitric acid so that they are ready for column loading just prior to completion of the valence adjustment steps. More than one vacuum box may be used to increase throughput.

11.1.3. Set up TEVA[®] and DGA cartridges on the vacuum box system.

11.1.3.1. Place the inner centrifuge tube rack (supplied with vacuum box) into the vacuum box with the centrifuge tubes in the rack. Place the lid on the vacuum box system.

11.1.3.2. Place the yellow outer tips into all 24 openings of the lid of the vacuum box. Fit an inner white tip into each yellow tip.

11.1.3.3. For each sample, assemble a TEVA[®] and a DGA cartridge and lock these onto the inner white tip (DGA cartridge below TEVA[®]).

11.1.3.4. Place reservoirs on the top end of the TEVA[®] cartridge.

11.1.3.5. Seal unused openings on the vacuum box by inserting yellow caps included with the vacuum box into unused white tips to achieve a good seal during the separation. Alternately, plastic tape can be used to seal the unused lid holes.

11.1.3.6. Turn the vacuum on and ensure proper fitting of the lid.

11.1.3.7. Add 5 mL of 3 M HNO₃ to the column reservoir to precondition the TEVA[®] cartridges.

11.1.3.8. Adjust the vacuum to achieve a flow-rate of ~1 mL/min.

IMPORTANT: Unless the method specifies otherwise, use a flow rate of ~ 1 mL/min for load and strip solutions and ~ 2 -3 mL/min for rinse solutions.

11.1.4. TEVA[®] and DGA Resin Separation

11.1.4.1. Transfer each solution from Step 11.1.2.5 into the appropriate reservoir. Allow solution to pass through the stacked TEVA[®] + DGA cartridge at a flow rate of ~1 mL/min.

11.1.4.2. Add 5 mL of 6 M HNO₃ to each tube/beaker as a rinse and transfer each solution into the appropriate reservoir (the flow rate can be adjusted to ~2 mL/min).

11.1.4.3. Add a 5 mL rinse of 6 M HNO₃ to each column (the flow rate can be adjusted to ~2 mL/min).

11.1.4.4. Turn off vacuum, discard rinse solutions and remove reservoirs and TEVA[®] cartridges and discard. Place new reservoirs on the DGA cartridges.

11.1.4.5. Add a 20 mL rinse of 0.1 M HNO₃ to each reservoir (flow rate ~1-2 mL/min).

NOTE: The rinses with dilute nitric acid remove uranium while curium and americium are retained. Precipitation of uranium during

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microprecipitation is inhibited by adding hydrogen peroxide to ensure uranium is present as UO_2^{2+} .

- 11.1.4.6. Add 15 mL of 3 M HNO_3 –0.25 M HF to each reservoir at ~1-2 mL/min to complex and remove Th from the DGA Resin.
- 11.1.4.7. Add 3 mL of 4 M HCl to each reservoir at ~1–2 mL/min to rinse column of residual fluoride. Once the HCl has passed through the column, quickly pulse the vacuum two or three times to minimize the amount of residual HCl in the column prior to proceeding.
- 11.1.4.8. Ensure that clean, labeled plastic tubes are placed in the tube rack under each cartridge. For maximum removal of interferences during elution, also change connector tips prior to Cm/Am elution.
- 11.1.4.9. Add 10 mL of 0.25 M HCl solution to elute curium and americium from each cartridge, reducing the flow rate to ~1 mL/min (or slightly slower).
- 11.1.4.10. Set the curium fraction in the plastic tube aside for cerium fluoride coprecipitation, Step 11.2.
- 11.1.4.11. Discard the DGA cartridge.

11.2. Preparation of the Sample Test Source

NOTE: Instructions below describe preparation of a single sample test source (STS). Several STSs can be prepared simultaneously if a multi-channel vacuum manifold system is available.

- 11.2.1. Pipet 100 μL of the cerium carrier solution into each tube.
- 11.2.2. Pipet 0.5 mL 30 wt. % H_2O_2 into each tube to prevent residual uranium from precipitating.
- 11.2.3. Pipet 1 mL of concentrated HF into each tube.
- 11.2.4. Cap the tube and mix. Allow samples sit for ~ 15 minutes before filtering.
- 11.2.5. Set up a filter apparatus to accommodate a 0.1-micron, 25-mm membrane filter on a microprecipitation filtering apparatus.
Caution: Following deposition of the microprecipitate, there is no visible difference between the two sides of the filter.
- 11.2.6. If a hydrophobic filter is used, add a few drops of 95% ethanol to wet each filter and apply vacuum. Ensure that there are no leaks along the sides before proceeding.
- 11.2.7. While vacuum is applied, add 2-3 mL of filtered Type I water to each filter and allow the liquid to drain.
- 11.2.8. Add the sample to the reservoir, rinsing the sample tubes with ~3 mL of water and transfer this rinse to filter apparatus. Allow to drain.
- 11.2.9. Wash each filter with ~2-3 mL of water and allow to drain.
- 11.2.10. Wash each filter with ~1-2 mL of 95% ethanol to displace water.
- 11.2.11. Allow to drain completely before turning the vacuum off.
- 11.2.12. Mount the filter on a labeled adhesive mounting disk (or equivalent) ensuring that the filter is not wrinkled and is centered on the mounting disk.

- 11.2.13. Place the filter under a heat lamp for approximately 5 minutes or longer until it is completely dry.
- 11.2.14. Count filters for an appropriate period of time by alpha spectrometry.
- 11.2.15. Discard the filtrate to waste for future disposal. If the filtrate is to be retained, it should be stored in a plastic container since glass will be attacked by HF.

NOTE: Other methods for STS preparation, such as electrodeposition or microprecipitation with neodymium fluoride, may be used in lieu of the cerium fluoride microprecipitation, but any such substitution must be validated as described in Step 1.5.

12. Data Analysis and Calculations

12.1. Equations for activity concentration, combined standard uncertainty, and radiochemical yield (if required):

- 12.1.1. The activity concentration of the analyte and its combined standard uncertainty are calculated using the following equations:

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

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

$$D_t = e^{-\lambda_a t_t}$$

$$D_a = e^{-\lambda_a t_a}$$

Where

- AC_a = activity concentration of the analyte at time of collection (or other specified reference time), in picocuries per liter (pCi/L)
- A_t = activity of the tracer added to the sample aliquant on the tracer solution reference date/time (pCi)
- R_a = net count rate of the analyte in the defined region of interest (ROI), in counts per second (see 12.1.2)
- R_t = net count rate of the tracer in the defined ROI, in counts per second (see 12.1.2)
- 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

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| | |
|-------------|---|
| I_t | = probability of α emission in the defined ROI per decay of the tracer (Table 17.1) |
| I_a | = probability of α emission in the defined ROI per decay of the analyte (Table 17.1) |
| $u_c(AC_a)$ | = combined standard uncertainty of the activity concentration of the analyte (pCi/L) |
| $u(A_t)$ | = standard uncertainty of the activity of the tracer added to the sample (pCi) |
| $u(R_a)$ | = standard uncertainty of the net count rate of the analyte (s^{-1}) (see 12.1.2) |
| $u(R_t)$ | = standard uncertainty of the net count rate of the tracer (s^{-1}) (see 12.1.2) |
| $u(V_a)$ | = standard uncertainty of the size of the sample aliquant volume (L) |
| λ_t | = decay constant for the tracer radionuclide (s^{-1} , see Table 17.1), |
| λ_a | = decay constant for the analyte radionuclide (s^{-1} , see Table 17.1), |
| t_t | = time elapsed between the activity reference date for the tracer and the midpoint of the sample count (s). |
| t_a | = time elapsed between the activity reference date for the sample (e.g., collection date) and the midpoint of the sample count (s). |

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

NOTE: 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: The standard uncertainty of the activity of the tracer added to the sample must reflect the uncertainty 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 (e.g., weighing or dilution factors) and during the process of adding the tracer to the sample.

12.1.2. The net count rate of an analyte or tracer and its standard uncertainty are calculated using the following equations:

$$R_x = \frac{C_x}{t_s} - \frac{C_{bx}}{t_b}$$

and

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

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 |

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| | | |
|----------|---|--|
| t_s | = | sample count time (s) |
| C_{bx} | = | background counts in the same ROI as for x |
| t_b | = | background count time (s) |
| $u(R_x)$ | = | standard uncertainty of the net count rate of tracer or analyte, in counts per second ² |

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

$$RY = \frac{R_t}{0.037 \times A_t \times D_t \times I_t \times \varepsilon}$$

and

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

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 (pCi) |
| 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 17.1) |
| 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 (pCi) |
| $u(\varepsilon)$ | = | standard uncertainty of the detector efficiency |

12.1.3. If the critical level concentration (L_c) or the minimum detectable concentration (MDC) are requested (at an error rate of 5%), they can be calculated using the following equations:³

² For methods with very low counts, MARLAP Section 19.5.2.2 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.

³ The formulations for the critical level and minimum detectable concentration are based on the Stapleton Approximation as recommended in MARLAP Section 20A.2.2, Equations 20.54 and 20A.3.2, and Equation 20.74, respectively (EPA 2004). The formulations presented here assume an error rate of $\alpha = 0.05$, $\beta = 0.05$ (with $z_{1-\alpha} = z_{1-\beta} = 1.645$) and $d = 0.4$. For methods with very low numbers of counts, these expressions provide better estimates than do the traditional formulas for the critical level and MDC.

Rapid Radiochemical Method for Curium-244 in Water Samples

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

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

where:

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

12.2. Results Reporting

12.2.1. The following data should be reported for each result: volume of sample used; yield of tracer and its uncertainty; and FWHM of each peak used in the analysis.

12.2.2. The following conventions should be used for each result:

12.2.2.1. Result in scientific notation \pm combined standard uncertainty.

13. Method Performance

13.1. Method validation results performed prior to analyzing samples are to be documented and reported as required.

13.2. Expected processing time per batch of 10-20 samples plus QC:

13.2.1. For an analysis of a 0.2 L sample aliquant, precipitation and preparation of load solution takes ~1 h.

13.2.2. Purification and separation using cartridges and vacuum box system should take ~2 h.

13.2.3. The sample test source preparation step takes ~0.75 h.

13.2.4. A four-hour counting time should be sufficient to meet the MQOs listed in Step 1.4, assuming detector efficiency of 0.15–0.3, and radiochemical yield of at least 0.5. A different counting time may be necessary to meet these MQOs if any of the relevant parameters are significantly different.

13.2.5. Data should be ready for reduction ~8-9 h after beginning of analysis.

14. Pollution Prevention: The method utilizes small volume (2 mL) extraction chromatographic resin columns. This approach leads to a significant reduction in the volumes of load, rinse and strip solutions, as compared to classical methods using ion exchange resins to separate and purify the curium fraction.

15. Waste Management

- 15.1. Types of waste generated per sample analyzed
 - 15.1.1. Approximately 210 mL basic waste from the initial sample preconcentration.
 - 15.1.2. Approximately 65 mL of acidic waste from loading and rinsing the two extraction columns will be generated.
 - 15.1.3. Approximately 25 mL of acidic waste from the microprecipitation method for source preparation will be generated. The waste contains 1 mL of HF and ~ 5 mL of ethanol.
 - 15.1.4. TEVA[®] cartridge – ready for appropriate disposal.
 - 15.1.5. DGA cartridge – ready for appropriate disposal.
 - 15.1.6. These waste streams may contain low levels of ²⁴³Am (added as tracer), ²⁴⁴Cm (added to LCS) and other radionuclides as present in samples.
- 15.2. Evaluate all waste streams according to disposal requirements by applicable regulations.

16. References

Cited References

- 16.1. EPA 2009. *Method Validation Guide for Radiological Laboratories Participating in Incident Response Activities*. Revision 0. Office of Air and Radiation, Washington, DC. EPA 402-R-09-006, (June 2009). Available [here](#).
- 16.2. EPA 2004. *Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)*. 2004. EPA 402-B-1304 04-001A, July. Volume I, Chapters 6, 7, 20, Glossary; Volume II and Volume III, Appendix G. Available [here](#)
- 16.3. ASTM D7282 “Standard Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements,” ASTM Book of Standards 11.02, current version, ASTM International, West Conshohocken, PA.
- 16.4. ASTM D1193, “Standard Specification for Reagent Water,” ASTM Book of Standards 11.02, current version, ASTM International, West Conshohocken, PA.

Other References

- 16.5. EPA 2012. *Rapid Radiochemical Method for Americium-241 in Building Materials for Environmental Remediation Following Radiological Incidents*. Office of Air and Radiation, Washington, DC. Available [here](#).
- 16.6. Maxwell, S., Culligan, B. and Noyes, G. 2010. Rapid Separation Method for Actinides in Emergency Soil Samples, *Radiochimica Acta*. 98(12): 793-800.
- 16.7. Maxwell, S., Culligan, B., Kelsey-Wall, A. and Shaw, P. 2011. “Rapid Radiochemical Method for Determination of Actinides in Emergency Concrete and Brick Samples,” *Analytica Chimica Acta*. 701(1): 112-8.
- 16.8. VBS01, Rev.1.4, “Setup and Operation Instructions for Eichrom’s Vacuum Box System (VBS),” Eichrom Technologies, LLC., Lisle, Illinois (January 2014).

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17. Tables, Diagrams, Flow Charts, and Validation Data

17.1. Tables

Table 17.1 Alpha Particle Energies and Abundances of Importance^[1]

| Nuclide | Half-Life (Years) | λ (s ⁻¹) | Abundance | α Emission Energy in kilo electron volts |
|----------------------------------|-----------------------|------------------------------|-----------|---|
| ²⁴⁴ Cm | 18.11 | 1.213×10 ⁻⁰⁹ | 0.7690 | 5805 |
| | | | 0.2310 | 5763 |
| ²⁴³ Cm | 29.1 | 7.55×10 ⁻¹⁰ | 0.0150 | 6066 |
| | | | 0.047 | 6058 |
| | | | 0.010968 | 6010 |
| | | | 0.0568 | 5992 |
| | | | 0.0069797 | 5876 |
| | | | 0.730 | 5785 |
| | | | 0.115 | 5742 |
| | | | 0.015954 | 5686 |
| | | | 0.0019942 | 5682 |
| | | | 0.0013959 | 5639 |
| ^{244/243} Cm (combined) | 18.11 | 1.213×10 ⁻⁰⁹ | 1.000 | 5805 |
| ²⁴² Cm | 0.4462 | 4.923×10 ⁻⁰⁸ | 0.2592 | 6113 |
| | | | 0.7408 | 6069 |
| ²⁴⁵ Cm | 8.50×10 ³ | 2.58×10 ⁻¹² | 0.0058 | 5529 |
| | | | 0.0083 | 5489 |
| | | | 0.0045 | 5370 |
| | | | 0.9320 | 5361 |
| | | | 0.5000 | 5304 |
| ²⁴⁶ Cm | 4.76×10 ³ | 4.61×10 ⁻¹² | 0.0032 | 5234 |
| | | | 0.822 | 5387 |
| ²⁴⁷ Cm | 1.560×10 ⁷ | 1.408×10 ⁻¹⁵ | 0.178 | 5344 |
| | | | 0.138 | 5267 |
| | | | 0.057 | 5212 |
| | | | 0.0120 | 5147 |
| | | | 0.0200 | 4985 |
| | | | 0.0160 | 4943 |
| | | | 0.710 | 4870 |
| | | | 0.047 | 4820 |
| ²⁴³ Am | 7.370×10 ³ | 2.980×10 ⁻¹² | 0.0016 | 5349 |
| | | | 0.0016 | 5321 |
| | | | 0.871 | 5275 |
| | | | 0.112 | 5233 |
| ²⁴³ Am (combined) | 7.370×10 ³ | 2.980×10 ⁻¹² | 0.0136 | 5181 |
| ²⁴¹ Am | 432.6 | 5.078×10 ⁻¹¹ | 0.9998 | 5275 |
| | | | 0.0037 | 5545 |
| | | | 0.00225 | 5512 |
| | | | 0.848 | 5486 |
| | | | 0.131 | 5443 |
| | | | 0.01660 | 5388 |

^[1] Particle energies with abundances less than 0.1% have been omitted unless they are contiguous with the radionuclide region of interest.

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Data were queried from the NUDAT 2 Decay Radiation database at the Brookhaven National Laboratory National Nuclear Data Center, (http://www.nndc.bnl.gov/nudat2/indx_dec.jsp) on 9/19/2014.

Table 17.2 Alpha Emissions Sorted by Decreasing Energy

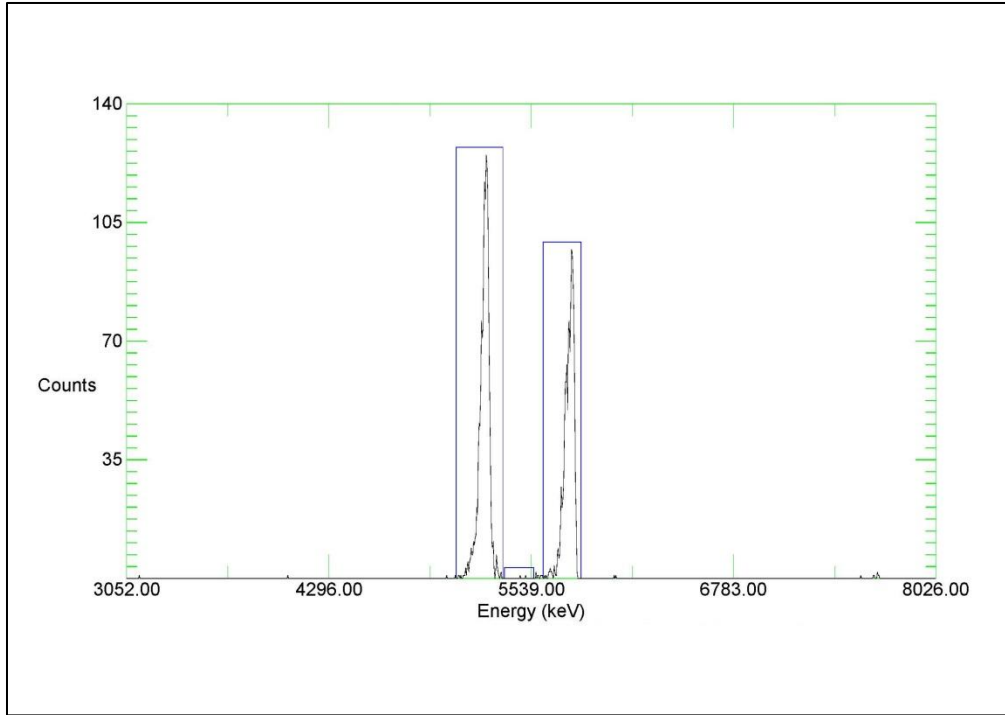
| Nuclide | Half-Life (years) | λ (s^{-1}) | α Emission Energy (keV) ^[1] | Abundance |
|-------------------|---------------------|-------------------------|---|-----------|
| ²⁴² Cm | 0.4462 | 4.923×10^{-08} | 6113 | 0.7408 |
| ²⁴² Cm | 0.4462 | 4.923×10^{-08} | 6069 | 0.2592 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 6066 | 0.0150 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 6058 | 0.047 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 6010 | 0.010968 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5992 | 0.0568 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5876 | 0.0069797 |
| ²⁴⁴ Cm | 18.11 | 1.213×10^{-09} | 5805 | 0.7690 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5785 | 0.730 |
| ²⁴⁴ Cm | 18.11 | 1.213×10^{-09} | 5763 | 0.2310 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5742 | 0.115 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5686 | 0.015954 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5682 | 0.0019942 |
| ²⁴³ Cm | 29.1 | 7.55×10^{-10} | 5639 | 0.0013959 |
| ²⁴¹ Am | 4.326×10^2 | 5.078×10^{-11} | 5545 | 0.0037 |
| ²⁴⁵ Cm | 8.50×10^3 | 2.58×10^{-12} | 5529 | 0.0058 |
| ²⁴¹ Am | 4.326×10^2 | 5.078×10^{-11} | 5512 | 0.00225 |
| ²⁴⁵ Cm | 8.50×10^3 | 2.58×10^{-12} | 5489 | 0.0083 |
| ²⁴¹ Am | 4.326×10^2 | 5.078×10^{-11} | 5486 | 0.848 |
| ²⁴¹ Am | 4.326×10^2 | 5.078×10^{-11} | 5443 | 0.131 |
| ²⁴¹ Am | 4.326×10^2 | 5.078×10^{-11} | 5388 | 0.01660 |
| ²⁴⁶ Cm | 4.76×10^3 | 4.61×10^{-12} | 5387 | 0.822 |
| ²⁴⁵ Cm | 8.50×10^3 | 2.58×10^{-12} | 5370 | 0.0045 |
| ²⁴⁵ Cm | 8.50×10^3 | 2.58×10^{-12} | 5361 | 0.9320 |
| ²⁴³ Am | 7.370×10^3 | 2.980×10^{-12} | 5349 | 0.0016 |
| ²⁴⁶ Cm | 4.76×10^3 | 4.61×10^{-12} | 5344 | 0.178 |
| ²⁴³ Am | 7.370×10^3 | 2.980×10^{-12} | 5321 | 0.0016 |
| ²⁴⁵ Cm | 8.50×10^3 | 2.58×10^{-12} | 5304 | 0.5000 |
| ²⁴³ Am | 7.370×10^3 | 2.980×10^{-12} | 5275 | 0.871 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 5267 | 0.138 |
| ²⁴⁵ Cm | 8.50×10^3 | 2.58×10^{-12} | 5234 | 0.0032 |
| ²⁴³ Am | 7.370×10^3 | 2.980×10^{-12} | 5233 | 0.112 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 5212 | 0.057 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 5147 | 0.0120 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 4985 | 0.0200 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 4943 | 0.0160 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 4870 | 0.710 |
| ²⁴⁷ Cm | 1.560×10^7 | 1.408×10^{-15} | 4820 | 0.047 |

^[1]Particle energies with abundances less than 0.1% have been omitted unless they would be contiguous with the radionuclide region of interest.

Data were queried from the NUDAT 2 Decay Radiation database at Brookhaven National Laboratory National Nuclear Data Center, (http://www.nndc.bnl.gov/nudat2/indx_dec.jsp) on 9/19/2014.

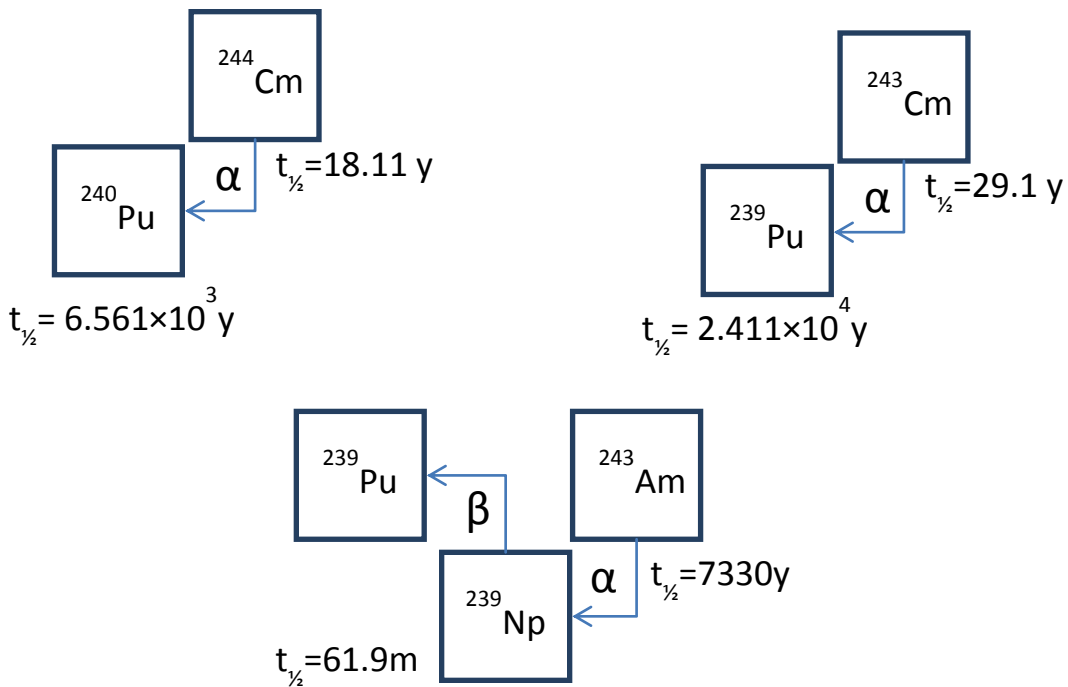
Ingrowth Curves and Ingrowth Factors
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17.2. Spectrum from a Processed Sample



Curium-244 Spectrum

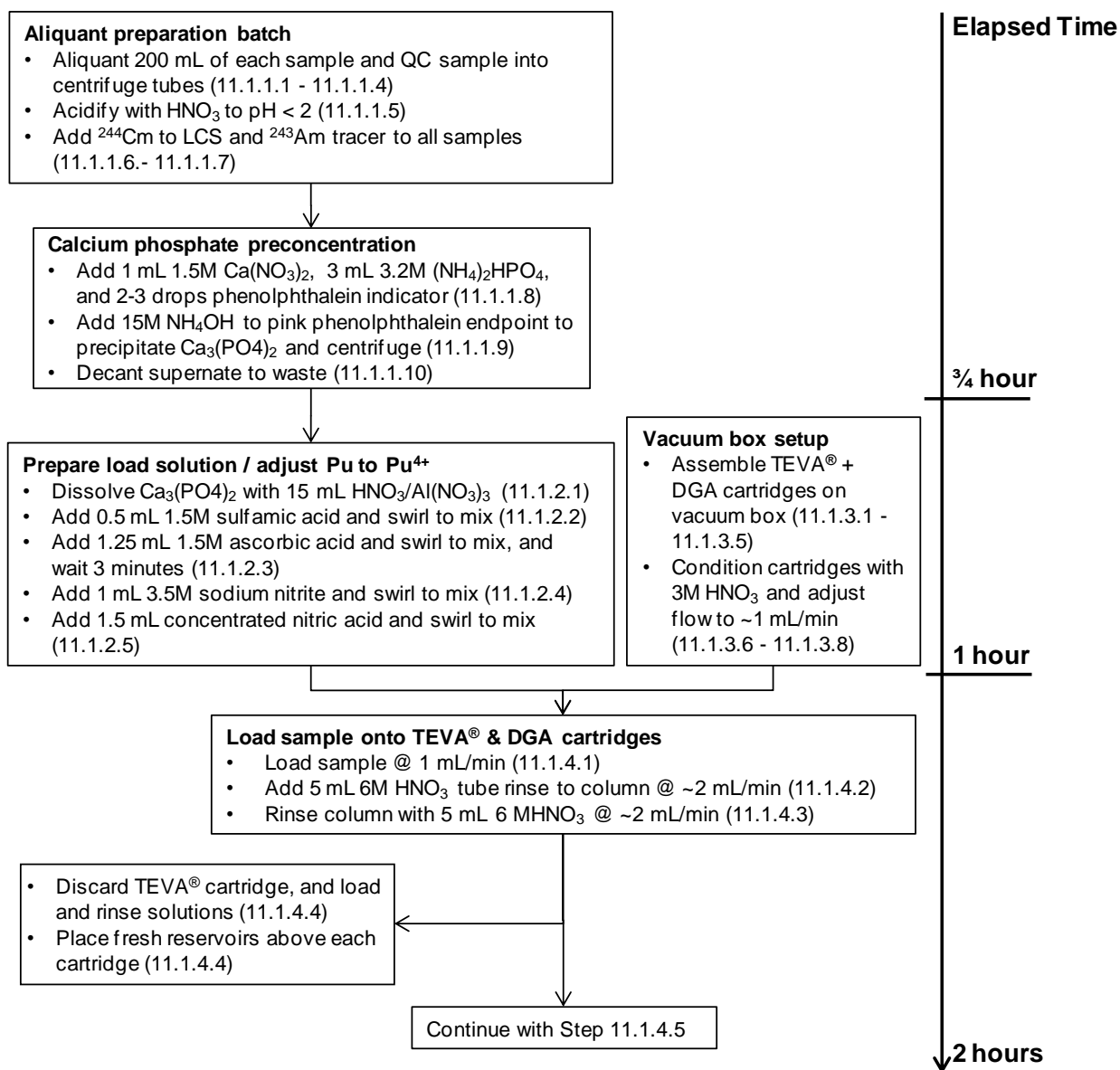
17.3. Decay Scheme



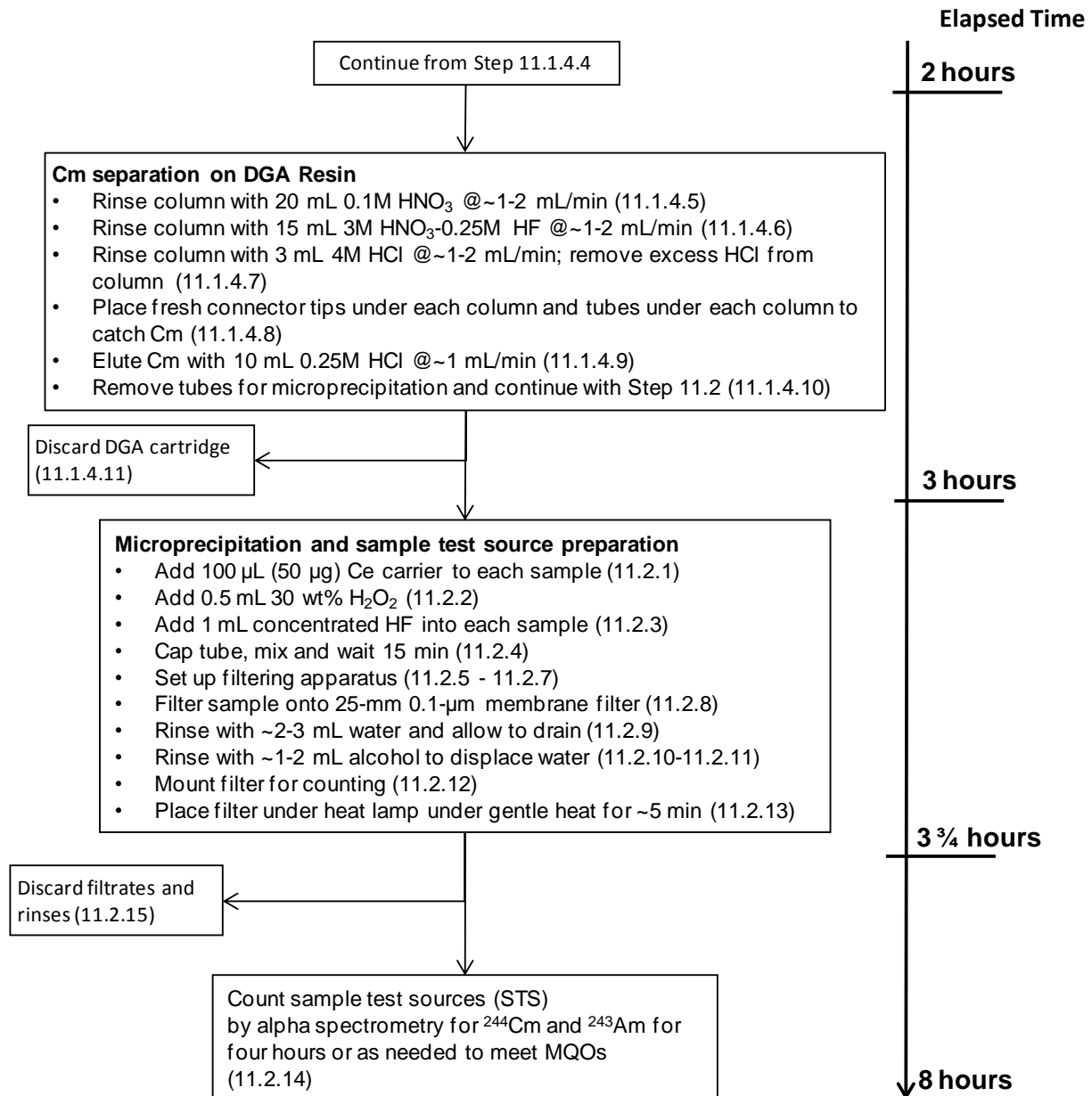
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17.4. Flow Chart

Separation Scheme and Timeline for the Determination of ²⁴⁴Cm in Water Samples



Separation Scheme and Timeline for the Determination of ²⁴⁴Cm in Water Samples (cont.)



**Appendix A:
Composition of Test Samples Used for Validation**

| Metals by ICP-MS | Concentration (µg/L) & |
|-------------------------|---------------------------------------|
| Be | 0.015 (J) |
| Na | 3,400 |
| Mg | 1,500 |
| Al | 15 (J) |
| K | 1,500 |
| Ca | 17,000 |
| V | 0.30 (J) |
| Cr | 0.075 (J) |
| Mn | 6.3 |
| Fe | 26 (J) |
| Co | 0.047 (J) |
| Ni | 0.69 (J) |
| Cu | 59 |
| Zn | 8.0 |
| As | 0.27 (J) |
| Se | 0.11 (J) |
| Mo | <0.41 (U) |
| Ag | <0.0082 (U) |
| Cd | 0.010 (J) |
| Sb | 0.060 (J) |
| Ba | 20 |
| Tl | 0.054 (J) |
| Pb | 0.23 (J) |
| U | 0.010 (J) |
| Radionuclide | Activity Concentration (pCi/L) |
| ²⁴⁴ Cm | 0.012 ± 0.050 # |
| ^{252/250} Cf | 0.001 ± 0.044 # |
| ²⁴¹ Am | 0.20 ± 0.29 # |
| ²³² Th | 0.003 ± 0.034 & |
| ²³⁰ Th | 0.052 ± 0.081 & |
| ²²⁸ Th | 0.31 ± 0.12 & |
| ²³⁸ U | 0.18 ± 0.12 & |
| ²³⁵ U | 0.219 ± 0.095 & |
| ²³⁴ U | 0.232 ± 0.096 & |
| ²²⁶ Ra | 0.049 ± 0.024 & |

Qualifiers:

(U) – Result is less than the Instrument Detection Limit (IDL) per SW846 Method 6020A

(J) – Result falls between the IDL and the reporting limit

& Mean ± 2 standard deviations of triplicate analyses of each of two Montgomery, Alabama, tap water

Mean ± 2 standard deviations of replicate analysis of seven samples of Montgomery, Alabama, tap water