AMERICIUM, NEPTUNIUM, PLUTONIUM, THORIUM, CURIUM, URANIUM, AND STRONTIUM IN WATER

(WITH VACUUM BOX SYSTEM)

1. SCOPE

1.1. This is a method for the separation and measurement of americium, neptunium, plutonium, thorium, curium, uranium and strontium in water. The method is designed to separate actinides for measurement using alpha spectrometry and strontium for measurement by low background proportional counting. After separating actinides using this method, source preparation for measurement of actinides by alpha spectrometry is performed by electrodeposition (Eichrom SPA02) or rare earth fluoride micro precipitation (Eichrom SPA01).

1.2. This method does not address all aspects of safety, quality control, calibration or instrument set-up. However, enough detail is given for a trained radiochemist to achieve accurate and precise results for the analysis of the analyte(s) from the appropriate matrix, when incorporating the appropriate agency or laboratory safety, quality and laboratory control standards.

2. SUMMARY OF METHOD

2.1. Up to 1L of water sample is acidified to pH 2 and actinides are co-precipitated by calcium phosphate.

2.2. Tetravalent actinides (Pu, Th, Np) are retained on TEVA Resin and other actinides (U, Am) are retained on TRU Resin and Sr is retained on Sr Resin.

2.3. Alpha spectrometry sources are prepared by rare earth fluoride co-precipitation or electrodeposition. Sr sources are prepared by evaporating the column eluent on a stainless steel planchet.

2.4. Actinides are quantified by alpha spectrometry and strontium is quantified by low background proportional counting.

2.5. If ^{89}Sr and ^{90}Sr are both to be quantified, the user is referred to Eichrom method SRW01.

3. SIGNIFICANCE OF USE

3.1. This method allows for the isotopic analysis of six actinides and radioactive strontium from a single sample. The use of vacuum assisted flow and stacked cartridges allows sample preparation for batches of 12-24 samples to be performed in as little as 10 hours.

4. INTERFERENCES

4.1. Nuclides with unresolvable alpha energies such as ^{241}Am and ^{238}Pu, ^{237}Np and ^{234}U, or ^{232}U and ^{210}Po must be chemically separated to enable measurement. This method separates these isotopes effectively.

4.2. The ^{232}U tracer should be cleaned prior to use in this method (^{228}Th free) to avoid false positive measurement of ^{228}Th in the thorium fraction if thorium is analyzed (Eichrom Method TP01).

4.3. When neptunium is analyzed, a ^{236}Pu tracer must be used, instead of ^{242}Pu, to achieve a cleaner separation of ^{237}Np and plutonium peaks.

4.4. Very high levels of phosphate in the sample may lead to reduced recovery of actinides in calcium phosphate precipitation and column separations. Adjusting the amount of phosphate added to co-precipitate the actinides may be necessary in these cases.

4.5. When Cm is analyzed together with Th and its ^{229}Th tracer, DGA resin should be used to remove the ^{225}Ac and ^{221}Fr (daughters of ^{229}Th) that interfere with measurement of ^{244}Cm and ^{242}Cm isotopes by alpha spectrometry.

4.6. The sample preparation procedure outlined in this method will adequately recover actinides from freshly collected, well preserved, homogenous water samples. Older, poorly preserved samples or samples with significant organic or solid matter may require more aggressive treatment to recover actinides which have precipitated or adsorbed to the walls of the storage container or solid matter. Rinsing the empty storage container with warm HNO_3, adjusting the HNO_3 concentration of the sample to 1M HNO_3 and

boiling, and/or wet-ashing the calcium phosphate precipitate may be required for older, poorly preserved samples.

5. APPARATUS

- Alpha spectroscopy system, low background
- Analytical balance, 0.0001 g sensitivity
- Cartridge reservoirs, 10 mL (Eichrom Part: AR-25-RV10) or 20 mL (Eichrom Part: AR-25-RV20)
- Centrifuge tubes, 50mL and 250mL
- Centrifuge, with rotor and carriers for 50mL and 250mL tubes
- Fume hood
- Gas flow proportional counter, low background
- Hotplate
- Inner support tubes, Eichrom Part: AR-1000-TUBE-PE
- Planchets, stainless steel, 2 inch diameter
- Stir rods, glass
- Vacuum box liner, Eichrom Part: AR-24-LINER or AR-12-LINER
- Vacuum box system, Eichrom Part: AR-24-BOX or AR-12-BOX
- Vacuum box yellow outer tips, Eichrom Part: AR-1000-OT
- Vacuum pump, dry pump 115 V, 60 Hz Fisher Part: 01-092-25 or house vacuum
- Vortex mixer

6. REAGENTS

Note: Analytical grade or ACS grade reagents and trace metal grade (or equivalent) acids are recommended. Evaluation of key reagents, such as aluminum nitrate and ammonium hydrogen phosphate, for contribution to method background levels from naturally occurring radioactive materials is recommended.

Aluminum nitrate nonahydrate, $Al(NO_3)_3 \cdot 9H_2O$
Ammonium hydrogen phosphate, $(NH_4)_2HPO_4$
Ammonium hydroxide(57%), concentrated NH_4OH
Ammonium oxalate monohydrate, $(NH_4)_2C_2O_4 \cdot H_2O$
Ammonium thiocyanate, NH_4SCN
Appropriate tracers or standards (Th-229, U-232, Am-243, Pu-242 or Pu-236)
Ascorbic acid powder, $C_6H_8O_6$

Calcium nitrate, $CaNO_3$
Deionized water, All reagents are prepared with deionized water
Ferric nitrate nonahydrate, $Fe(NO_3)_3 \cdot 9H_2O$
Hydrochloric acid (37%), concentrated HCl
Hydrofluoric acid (49%), concentrated HF
Hydrogen peroxide (30%), concentrated H_2O_2
Isopropyl alcohol, C_3H_7OH
Nitric acid (70%), concentrated HNO_3
Oxalic acid dihydrate, $H_2C_2O_4 \cdot 2H_2O$
Phenolphthalein pH Indicator
Sodium nitrite, $NaNO_2$
Sr® resin, 2mL prepacked cartridge, 50-100μm, Eichrom Part SR-R50-S
Strontium nitrate, $Sr(NO_3)_2$
Sulfamic Acid, H_3NSO_3
TEVA® resin, 2mL prepacked cartridge, 50-100μm, Eichrom Part TE-R50-S
Titanium (III) chloride, 10wt% $TiCl_3$ in 20-30wt% HCl
TRU® resin, 2 mL prepacked cartridge, 50-100μm, Eichrom Part TR-R50-S
*If using ^{229}Th tracer **and** measuring Cm isotopes:* *DGA® resin, normal,* 2mL prepacked cartridge, 50-100μm, Eichrom Part DN-R50-S

6.1. *Ammonium bioxalate (0.1 M)* - Dissolve 6.31g of oxalic acid and 7.11g of ammonium oxalate in 900mL of water. Dilute to 1L with water.

6.2. *Ammonium hydrogen phosphate (3.2 M)* - Dissolve 106g of $(NH_4)_2HPO_4$ in 200mL of water. Heat gently to dissolve. Dilute to 250mL with water.

6.3. *Ascorbic acid (1.0M)* - Dissolve 17.6g of ascorbic acid in 80mL of water. Heat to dissolve. Dilute to 100mL. **Prepare fresh weekly.**

6.4. *Calcium nitrate (1.25M)* - Dissolve 51g of $Ca(NO_3)_2$ in 100mL of water. Dilute to 250mL with water.

6.5. *Hydrochloric acid (0.1 M) - hydrofluoric acid (0.05M) - titanium chloride (0.03)*- Add 30mL of 10% $TiCl_3$, 4.2mL of concentrated HCl

and 0.9mL of concentrated HF to 400mL water. Dilute to 500mL with water. **Prepare fresh immediately prior to use.**

Note: This solution is used to strip Np and Pu from TEVA resin. $TiCl_3$ will interfere with electrodeposition of Np and Pu. If preparing alpha sources by electrodeposition, replace $TiCl_3$ with 0.04M rongalite (sodium formaldehyde sulfoxylate).

6.6. *Hydrochloric acid (4M)* - Add 333mL of concentrated HCl to 100mL of water. Dilute to 1L with water.

6.7. *Hydrochloric acid (4M)- hydrofluoric acid (0.2M)* - Add 333mL of concentrated HCl and 7.1mL of concentrated HF to 500mL of water. Dilute to 1L with water.

6.8. *Hydrochloric acid (9M*) - Add 750mL of concentrated HCl to 100mL of water. Dilute to 1L with water.

6.9. *Iron Carrier* - (5mg Fe/mL) - Dissolve 3.6g of $Fe(NO_3)_3 \cdot 9H_2O$ in 80mL of water. Dilute to 100mL with water.

6.10. *Nitric acid (3M) - Aluminum nitrate (1M) solution* - Dissolve 375g of $Al(NO_3)_3 \cdot 9H_2O$ in 500mL of water, add 188mL of concentrated HNO_3. Dilute to 1L with water.

Note: The nitric acid- aluminum nitrate may be scrubbed using UTEVA Resin to lower natural uranium levels in the aluminum nitrate if needed.

6.11. *Nitric acid solution (0.05M)* - Add 3.2mL of concentrated HNO_3 to 900mL of water. Dilute to 1L with water.

6.12. *Nitric acid solution (3M)* - Add 188mL of concentrated HNO_3 to 700mL of water. Dilute to 1L with water.

6.13. *Nitric acid solution (8M)* - Add 500mL of concentrated HNO_3 to 400mL of water. Dilute to 1L with water.

6.14. *Phenolphthalein solution* - Dissolve 1g phenolphthalein in 100mL 95% isopropyl alcohol. Dilute with 100mL of water.

6.15. *Sodium nitrite (3.5M) solution* - Dissolve 6.1g of sodium nitrite in 20mL of water. Dilute to 25mL with water. **Prepare fresh daily.**

6.16. *Strontium carrier* - 20mg/mL solution (standardized.) Dissolve 4.83g of anhydrous $Sr(NO_3)_2$ in 80mL of water. Dilute to 100mL with water.

6.17. *Sulfamic acid (1.*5M) - Dissolve 72.7g of sulfamic acid in 400mL of water. Dilute to 500mL with water.

7. PROCEDURE

7.1. Water Sample Preparation:

7.1.1. If required, filter the sample through a 0.45 micron filter.

7.1.2. If samples larger than 1L are analyzed, evaporate the sample to approximately 1L.

7.1.3. Aliquot 500 to 1000mL of the sample (or enough to meet required detection limit) into an appropriate size beaker.

7.1.4. Add 5mL concentrated HNO_3.

7.1.5. Add appropriate tracers per lab protocol.

Note: If using self-cleaning ^{232}U tracer (Eichrom Method TP01), vortex mix and centrifuge standard to ensure that ^{228}Th and its daughters are effectively removed from ^{232}U by the $BaSO_4$ precipitate.

7.1.6. Calcium phosphate precipitation:

7.1.6.1. Add 2mL of 1.25 M $Ca(NO_3)_2$, and 0.2mL of strontium carrier to each beaker.

7.1.6.2. Warm samples on a hotplate at medium setting for 30-60 minutes.

7.1.6.3. Turn down heat setting.

7.1.6.4. Add 0.75mL of phenolphthalein indicator and 5mL of 3.2M $(NH_4)_2HPO_4$ solution per liter of sample.

7.1.6.5. While stirring, slowly add enough concentrated NH_4OH to reach the phenolphthalein end point and form a calcium phosphate precipitate. Heat an additional 20-30 minutes.

7.1.6.6. Remove samples from hot plate, cool to room temperature, and allow precipitate to settle until solution can be decanted (30 minutes to 2 hours) or centrifuge.

7.1.6.7. Decant supernate and discard to waste.

7.1.6.8. Transfer the precipitate to a centrifuge tube with deionized water and centrifuge the precipitate for approximately 10 minutes at 2000 rpm.

7.1.6.9. Decant supernate and discard to waste.

7.1.6.10. Wash the precipitate with an amount of water approximately twice the volume of the precipitate. Mix well on a vortex mixer. Centrifuge for 5-10 minutes. Discard the supernate.

7.2. Actinide Separations using Eichrom Resins:

7.2.1. Dissolve each precipitate with 16mL of 3M HNO_3 -1.0M $Al(NO_3)_3$.

Note: Make sure that all reagents and the load solution have cooled to room temperature. Warm solutions can cause reactions that will affect oxidation adjustments performed in the following steps.

Note: Pu and Np must be present in (IV) state to be retained on TEVA Resin. The following steps, will ensure that Pu (III) and Pu (VI) are converted to Pu (IV) and Np (V) to Np (IV)

Note: Ascorbic acid is used to reduce plutonium to Pu (III). Np is reduced more effectively if iron (II) is also present. Ascorbic acid will reduce the iron (III) added to iron (II). The amount of iron is minimized to prevent iron interference on TRU Resin and is only added if Np is analyzed.

7.2.2. Add 0.5mL of 1.5M sulfamic acid to each solution. Swirl to mix.

7.2.3. If Np is analyzed, then add 0.2mL of 5mg/mL Fe solution. If Np is not required, then this step is not necessary.

7.2.4. Add 1.5mL of 1M ascorbic acid. Swirl to mix. Wait for 3 minutes.

7.2.5. Add 1mL of 3.5M $NaNO_2$. Swirl to mix.

Note: This will oxidize Pu(III) to Pu(IV).

7.2.6. Setup of TEVA, TRU and Sr cartridges in tandem on the vacuum box system.

7.2.6.1. Place the inner tube rack into the vacuum box with the 50mL centrifuge tubes in the rack. Alternatively, the vacuum box liner may be used here. Fit the lid to the vacuum system box.

7.2.6.2. Place yellow outer tips into all 12 or 24 openings of the lid of the vacuum box. Fit an inner support tube into each yellow tip.

7.2.6.3. For each sample solution, fit a Sr Resin cartridge into the inner support tube. Attach a TRU Resin cartridge to the

top end of the Sr Resin cartridge. Attach a TEVA cartridge to the top end of the TRU cartridge.

7.2.6.4. Attach syringe barrels (funnels/reservoirs) to the top end of the TEVA cartridge.

7.2.6.5. Connect the vacuum pump to the vacuum box. Turn the vacuum pump on and ensure proper fitting of the lid.

Note: Any unused openings on the vacuum box should be sealed. The yellow manifold plugs supplied with the vacuum box system can be used to plug unused inner support tubes to achieve good seal during the separation. Alternatively, the unused vacuum box holes can be sealed using scotch tape affixed to the vacuum box lid.

7.2.6.6. Add 5mL of 3M HNO_3 to the funnel to precondition the TEVA, TRU and Sr cartridges. Adjust the vacuum pressure to achieve a flow rate of 1mL/minute.

7.2.7. Loading Np, Th and Pu on TEVA; Am, Cm and U on TRU; and Sr on Sr Resin.

7.2.7.1. Transfer each sample solution from step 7.2.5. into the appropriate reservoir. Allow solution to pass through the cartridges at a flow rate of 1mL/minute.

7.2.7.2. Add 3mL of 3M HNO_3 to rinse to each sample tube. Transfer each solution into the appropriate reservoir. Allow the solution to pass through the cartridges at 1mL/min.

7.2.7.3. Add 5mL of 3M HNO_3 into each reservoir. Allow the solution to pass through the cartridges at 1-2mL/min.

7.2.7.4. Turn vacuum off.

7.2.7.5. Separate TEVA, TRU, and Sr Resin cartridge from each other. Set TRU and Sr Resin cartridges aside while Np, Pu and Th are eluted from TEVA.

7.2.8. Elution of Np, Pu and Th from TEVA cartridge

7.2.8.1. Place each TEVA cartridge along with a clean reservoir on a new opening on the vacuum box lid.

7.2.8.2. Add 20mL of 3M HNO_3 to each reservoir. Turn on vacuum. Allow solution to pass through TEVA cartridge at 1mL/min.

7.2.8.3. Stop vacuum and remove inner waste liner or centrifuge tubes.

7.2.8.4. Place tube rack with clean, labeled tubes under each cartridge. Placing clean vacuum box inner support tubes and yellow outer tips below each TEVA cartridge at this point will help to ensure clean Th fractions in the next step.

7.2.8.5. Add 20mL of 9M HCl to each reservoir. Turn on vacuum. Strip Th at 1mL/min.

7.2.8.6. Turn off vacuum. Set Th samples aside for alpha spectrometry source preparation.

7.2.8.7. Ensure that clean, labeled tubes are placed in the tube rack under each TEVA cartridge. Placing clean vacuum box inner support tubes and yellow outer tips below each TEVA cartridge at this point will help to ensure clean Np/Pu fractions in the next step.

7.2.8.8. Add 20mL of 0.1M HCl/0.05M HF/0.03M $TiCl_3$ to each reservoir. Turn on vacuum. Strip Np/Pu at 1mL/min.

Note: $TiCl_3$ will interfere with electrodeposition. If electrodeposition will be used, use 0.04M rongalite (sodium formaldehyde sulfoxylate) instead of 0.03M $TiCl_3$.

7.2.8.9. Set Np/Pu samples aside for alpha spectrometry source preparation.

7.2.9. Elution of Am, Cm and U from TRU cartridge.

Note: If using ^{229}Th as a tracer for Th <u>AND</u> measuring Cm isotopes proceed to 7.2.9.1. ^{225}Ac and ^{221}Fr, daughters of ^{229}Th, will interfere with ^{242}Cm and ^{244}Cm in the alpha spectrum. Additional steps using DGA resin to separate Ac and Fr from the Cm fraction will be required following the separation of Am/Cm on TRU Resin.

Note: If <u>not</u> using ^{229}Th as a tracer for Th -or- <u>not</u> measuring Cm isotopes, proceed to step 7.2.9.2.

7.2.9.1. Am and Cm elution using TRU Resin and DGA Resin

7.2.9.1.1. Attach a DGA cartridge to bottom end of each TRU cartridge. Place TRU-DGA on the inner tips on vacuum box lid. Place vacuum box inner liner or inner rack with centrifuge tubes below each set of cartridges.

7.2.9.1.2. Add 15mL of 4M HCl to each reservoir. Turn on vacuum. Strip Am/Cm from TRU onto DGA at 1mL/min.

7.2.9.1.3. Remove TRU cartridge and set aside for uranium elution, step 7.2.9.3.

7.2.9.1.4. Attach a clean reservoir to each DGA cartridge.

7.2.9.1.5. Add 5mL of 1M HNO_3 to each reservoir. Allow solution to pass through DGA cartridges at 1-2mL/min.

7.2.9.1.6. Add 15mL of 0.1M HNO_3 to each reservoir. Allow solution to pass through DGA cartridges at 1-2mL/min.

7.2.9.1.7. Turn off vacuum. Remove inner liner or centrifuge tubes and place inner tube rack into the vacuum box. Place a clean, labeled tube below each cartridge to collect Am and Cm. Placing clean inner support tubes and outer yellow tips under each DGA cartridge will help ensure clean Am/Cm fractions in the following step.

7.2.9.1.8. Add 10mL of 0.25M HCl to each reservoir. Turn on vacuum. Elute Am and Cm at 1-2mL/min.

7.2.9.1.9. Set Am/Cm samples aside for alpha spectrometry source preparation.

7.2.9.1.10. Place TRU cartridges from step 7.2.9.1.3 on vacuum box and GO TO step 7.2.9.3.

7.2.9.2. Am and Cm elution using TRU Resin

7.2.9.2.1. Place TRU cartridges on the appropriate vacuum box hole inner white tip. Place clean, labeled tubes under each cartridge for americium elution. Placing clean inner support tubes and outer yellow tips under each TRU cartridge will help ensure clean Am/Cm fractions in the following step.

7.2.9.2.2. Add 15mL of 4M HCl into each cartridge. Turn on vacuum. Elute Am and Cm at 1mL/min.

7.2.9.2.3. Set Am/Cm samples aside for alpha spectrometry source preparation.

7.2.9.3. Uranium elution from TRU Resin

7.2.9.3.1. Place the vacuum box inner liner or inner rack with centrifuge tubes in the vacuum box. Add 12mL of 4M HCl-0.2 M HF to each reservoir. Turn on vacuum. Allow solution to pass through TRU cartridges at 2mL/min.

Note: This rinse will remove any residual Th from the TRU resin.

7.2.9.3.2. Turn off vacuum. Place a clean, labeled tube below each cartridge for uranium elution. Placing clean inner support tubes and outer yellow tips under each TRU cartridge will help ensure clean uranium fractions in the following step.

7.2.9.3.3. Add 15mL of 0.1 M ammonium bioxalate to each cartridge reservoir. Turn on vacuum. Strip the uranium at 1-2mL/min.

7.2.9.3.4. Set U samples aside for alpha spectrometry source preparation.

7.2.10. Elution of Sr from Sr Resin

7.2.10.1. Place each Sr cartridge from on the appropriate inner white tip on the vacuum box. Attach a clean reservoir each cartridge. Insert liner into the vacuum box and place lid on top.

7.2.10.2. Add 5mL of 3M HNO_3-0.05 M oxalic acid to each reservoir. Turn on vacuum. Allow rinse to pass through column at 1-2mL/min.

7.2.10.3. Add 5mL of 8M HNO_3 to each reservoir. Allow rinse to pass through column at 1mL/min. **Record time and date for the end time of the rinse for Sr-90/Y-90 separation.**

7.2.10.4. Turn vacuum off. Remove liner from vacuum box and replace it with rack. Place labeled tube below the cartridge. Replace lid. Placing clean inner white tips and outer yellow tips under each Sr cartridge will help ensure clean uranium fractions in the following step.

7.2.10.5. Add 15mL of 0.05 M HNO_3 to each reservoir. Turn on vacuum. Elute Sr at 1mL/min.

7.2.10.6. Set tubes aside for strontium source mounting, step 7.4.

7.3. Prepare samples for the measurement of actinides by alpha spectrometry using electrodeposition (Eichrom SPA02) or rare earth fluoride micro precipitation (SPA01).

7.4. Sr Source Mounting and Measurement of Total Radioactive Sr

7.4.1.1. Anneal stainless steel planchets (~5-10mL volume) at 450°C for 1.5 hours. Annealed planchets will have a bronze-brown color. Heating to higher temperatures can make planchets more susceptible to acid degradation-oxide formation.

7.4.1.2. Allow planchets to cool. Label planchets by scratching bottom with glass cutting tool or other suitable tool.

7.4.1.3. Clean labeled planchets with paper towel moistened with ethanol. Allow planchets to completely dry.

7.4.1.4. Weigh each planchet on an analytical balance, recording the weight to the nearest 0.1mg.

7.4.1.5. Place hotplate(s) in hood. Place planchets on hotplate. Heat at medium setting.

7.4.1.6. Add each Sr fraction to the appropriate planchet, in successive 3mL portions. Allow each 3mL portion to evaporate to near dryness between additions. To prevent spattering, avoid going to complete dryness before adding the next 3mL portion.

7.4.1.7. Rinse the tube containing the Sr solution with 2mL of 0.05M HNO_3 and transfer to the planchet.

7.4.1.8. After all the solution has evaporated to dryness, heat for an additional 10-20 minutes. Cool the planchet.

7.4.1.9. Reweigh each planchet and record the weight.

7.4.1.10. If yield is >100%, moisture may still be present. Heat sample and weigh again.

7.4.1.11. Count samples on a low background proportional counter for a sufficient time to achieve the desired counting statistics and minimum detectable concentration.

7.4.1.12. If Sr-89 and Sr-90 are each to be quantified, refer to Eichrom method SRW01 beginning in section 7.4.

8. CALCULATIONS

Calculate the actinide activity as follows:

Calculate tracer yield:

$$\text{Yield} = \frac{(C_s - B_s)}{E_s \times A_s}$$

where:

C_s =measured actinide tracer, cpm
B_s =background, cpm
E_s =counting efficiency for tracer
A_s =tracer activity, dpm

Note: If any tracer may be present in the sample, a spiked and unspiked sample must be analyzed to determine chemical yield, where:

$$\text{Yield} = \frac{(\text{spiked sample tracer cpm} - \text{unspiked sample tracer cpm})}{E \times \text{actinide spike activity dpm}}$$

Percent yield = Yield x 100

Calculate actinide isotope activity:

$$\text{Sample dpm / g or dpm / L} = \frac{S - B}{E \times V \times Y}$$

where:

S = sample activity, cpm
B = background, cpm
E = counting efficiency = measured cpm/dpm of isotopic standard
V = sample weight, g or volume, L
Y = yield

Conversion of dpm/g to pCi/g: pCi/g = (dpm/g)/2.22

Strontium Activity Calculations: Refer to Eichrom method SRW01 for calculations for total radioactive strontium and for Sr-89/Sr-90.

9. PERFORMANCE DATA

9.1. This method has been tested with various water samples such as DI and tap waters using the calcium phosphate precipitation option in Eichrom's laboratory. The data listed below in Table 1 are the average tracer yields for the actinides and gravimetric yield for stable strontium. The Sr-90 spike recoveries were corrected by the gravimetric yield. The tap water used in this test had significant amounts of Ca (500 ppm), Mg (49 ppm) and Na (106 ppm).

Table 1

Sample Type	% Am-243 (N=5)	% Pu-242 (N=5)	% U-232 (N=5)	% Th-229 (N=5)	% Sr Carrier (N=5)	% Sr-90 Recoveries (N=5)
DI water	84 ± 4	79 ± 12	85 ± 6	82 ± 11	88.7 ± 5.4	105 ± 4
Tap water	80 ± 2	90 ± 5	80 ± 6	94 ± 8	74.1 ± 6	101 ± 2.4

9.2. This method is routinely used at the SRS Environmental laboratory, Washington Savannah River, Aiken, SC. The SRS data listed below in Table 2 is for a batch of 20 groundwater samples (200 mL each). (*Maxwell, S.L., 2006)*

Table 2

Tracer	**% Tracer Yields**	**LCS ***	**% LCS Recoveries**
Am-243 (N=20)	92.6 ± 6	Am-241	109
Pu-242 (N=20)	104 ± 4.9	Pu-238	98.5
U-232 (N=20)	85.6 ± 5.4	U-235	104
Sr Carrier (N=20)	98.1 ± 3.9	Sr-90	99.1

**LCS-% recoveries for laboratory control standard*

9.3. SRS Environmental Laboratory data is listed in Table 3 for 200 mL ground water samples for Am, Cm Pu and Th. *(Maxwell, S.L. 2006)*

Table 3

Tracer	% Tracer Yields	LCS *	% LCS Recoveries
Th-229 (N=4)	82.4 ± 4.4	Th-230	97.7
Am-243 (N=4)	100 ± 4.5	Cm-244	105.8
Pu-242 (N=4)	95.1 ± 4.9	Pu-238	98.4
U-232 (N=4)	82.2 ± 1.2	U-235	104

**LCS-% recoveries for laboratory control standard*

10. REFERENCES

1) Horwitz, E.P., et al. "Separation and Preconcentration of Actinides by Extraction Chromatography using a supported liquid anion exchanger: Application of the characterization of high-level nuclear waste solutions" Analytica Chimica Acta., Vol. 310, pp. 63-78 (1995)

2) Horwitz, E.P., et al. "Separation and Preconcentration of Actinides from Acidic Media by Extraction Chromatography", Analytica Chimica Acta, Vol. 281, pp. 361-372(1993)

3) Horwitz, E. P., et al., "Novel Extraction Chromatographic Resins Based on Tetraalkyldiglycolamides: Characterization and Potential Applications", Solvent Extraction and Ion Exchange., 23, 219 (2005)

4) Maxwell, S.L., "Rapid Column Extraction Method for Actinides and Sr-89/90 in Water Samples", J. Radioanal. Nucl. Chem., 267, 537-543 (2006)

5) ASTM Method D7282-06, "Standard Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements."

6) ASTM Method D3648-14, "Standard Practices for the Measurement of Radioactivity."

1) Aliquot up to 1000mL of water into glass beaker.
2) Add 5mL concentrated HNO_3 and add yield tracers.
3) Add 1mL of 1.25M $Ca(NO_3)_2$ and 0.2mL of Sr carrier.
4) Heat samples at medium setting for 30-60 minutes.
5) Turn down hot plate.
6) Add 0.75mL of phenolphthalein and 3mL of 3.2M $(NH_4)_2HPO_4$.
7) While stirring sample, slowly add conc. NH_4OH until reaching pH 9.
8) Remove samples from hot plate, cool to room temperature, and allow precipitate to settle or centrifuge.
9) Decant supernate and discard as waste.
10) Transfer precipitate to centrifuge tube with DI water.
11) Centrifuge ~10minutes at 2000rpm. Decant supernate.
12) Add 10mL DI water to ppt. Mix well. Centrifuge. Decant supernate. Dissolve ppt with 5mL conc. HNO_3. Transfer to 100mL beaker.
13) Rinse centrifuge tube with 2-3mL conc. HNO_3. Transfer to 100mL beaker. Evaporate to dryness.
14) Dissolve reside in 16mL 3M HNO_3-1M $Al(NO_3)_3$. Add 1mL 1.5M Sulfamic Acid, 0.5 mL Fe, and 1mL 1M Ascorbic Acid. Swirl to mix. Wait 3-5 minutes.
15) Add 1mL 3.5M $NaNO_2$. Swirl to mix.
16) Precondition TEVA-TRU-Sr with 5mL 3M HNO_3.
17) Load sample onto TEVA-TRU-Sr. Allow liquid to drain. TEVA retaines Th, Pu, Np. TRU retains Am, Cm, U.
18) Rinse sample tube with 5mL 3M HNO_3. Add rinse to TEVA-TRU-Sr. Allow liquid to drain.
19) Rinse TEVA-TRU-Sr with 5mL 3M HNO_3. Allow liquid to drain.
20) Separate TEVA, TRU, and Sr cartridges.
2mL TEVA
2mL TRU
2mL Sr
Waste
(16) (17) (18) (19)
21) Rinse TEVA column with 20mL 3M HNO_3.
22) Place clean centrifuge tube below TEVA. Strip Th with 15mL 9M HCl.
23) Place clean centrifuge tube below each TEVA. Strip Pu-Np with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.
2mL TEVA
(21)
(22)
(23)
Waste
Th sample to source preparation
Pu-Np sample to source preparation
24) Place clean centrifuge tubes below TRU. Strip Am with 15mL of 4M HCl.
25) Rinse TRU with 12mL 4M HCl-0.1M HF. Discard as waste.
26) Place a clean centrifgue tube below each cartridge. Strip U with 15mL 0.1M ammonium bioxalate.
2mL TRU
(25)
(24)
(26)
Waste
Am sample to source preparation
Pu sample to source preparation
27) Rinse Sr with 5mL 3M HNO_3-0.05M oxalic acid. Discard as waste.
28) Rinse Sr with 5mL 8M HNO_3. Discard as waste. Record Sr-90/Y-90 separation time.
29) Strip Sr with 15mL 0.05M HNO_3.
2mL Sr
(27) (28)
(29)
Waste
Sr sample to source preparation

1) Aliquot up to 1000mL of water into glass beaker.
2) Add 5mL concentrated HNO_3 and add yield tracers.
3) Add 1mL of 1.25M $Ca(NO_3)_2$ and 0.2mL Sr carrier.
4) Heat samples on medium setting for 30-60 minutes.
5) Turn down hot plate.
6) Add 0.75mL of phenolphthalein and 3mL of 3.2M $(NH_4)_2HPO_4$.
7) While stirring sample, slowly add conc. NH_4OH until reaching pH 9.
8) Remove samples from hot plate, cool to room temperature, and allow precipitate to settle or centrifuge.
9) Decant supernate and discard as waste.
10) Transfer precipitate to centrifuge tube with DI water.
11) Centrifuge ~10minutes at 2000rpm. Decant supernate.
12) Add 10mL DI water to ppt. Mix well. Centrifuge. Decant supernate. Dissolve ppt with 5mL conc. HNO_3. Transfer to 100mL beaker.
13) Rinse centrifuge tube with 2-3mL conc. HNO_3. Transfer to 100mL beaker. Evaporate to dryness.
14) Dissolve reside in 16mL 3M HNO_3-1M $Al(NO_3)_3$. Add 1mL 1.5M Sulfamic Acid, 0.5 mL Fe, and 1mL 1M Ascorbic Acid. Swirl to mix. Wait 3-5 minutes.
15) Add 1mL 3.5M $NaNO_2$. Swirl to mix.
16) Precondition TEVA-TRU-Sr with 5mL 3M HNO_3.
17) Load sample onto TEVA-TRU-Sr. Allow liquid to drain. TEVA retaines Th, Pu, Np. TRU retains Am, Cm, U.
18) Rinse sample tube with 5mL 3M HNO_3. Add rinse to TEVA-TRU-Sr. Allow liquid to drain.
19) Rinse TEVA-TRU-Sr with 5mL 3M HNO_3. Allow liquid to drain.
20) Separate TEVA, TRU, and Sr cartridges.
2mL TEVA
2mL TRU
2mL Sr
Waste
(16) (17) (18) (19)
21) Rinse TEVA column with 20mL 3M HNO_3.
22) Place clean centrifuge tube below TEVA. Strip Th with 15mL 9M HCl.
23) Place clean centrifuge tube below each TEVA. Strip Pu-Np with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.
2mL TEVA
(21)
Waste
(22)
Th sample to source preparation
(23)
Pu-Np sample to source preparation
24) Place clean centrifuge tubes below TRU. Add DGA cartridge below TRU.
25) Strip Am-Cm from TRU onto DGA with 15mL 4M HCl.
26) Separate TRU and DGA. Place clean centrifuge tube below DGA.
27) Rinse DGA with 5mL 1M HNO_3.
28) Rinse DGA with 15mL of 0.1M HNO_3.
29) Strip Am-Cm from DGA with 15mL 0.25M HCl.
30) Rinse TRU with 12mL 4M HCl-0.1M HF. Discard as waste.
31) Place a clean centrifgue tube below each cartridge. Strip U with 15mL 0.1M ammonium bioxalate.
2mL TRU
2mL DGA
Waste
(25) (26) (27) (28)
Am sample to source preparation
(29)
Pu sample to source preparation
(31)
32) Rinse Sr with 5mL 3M HNO_3-0.05M oxalic acid. Discard as waste.
33) Rinse Sr with 5mL 8M HNO_3. Discard as waste. Record Sr-90/Y-90 separation time.
34) Strip Sr with 15mL 0.05M HNO_3.
2mL Sr
Waste
(32) (33)
Sr sample to source preparation
(34)