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

(WITH VACUUM BOX SYSTEM)

1. SCOPE

1.1. This is a method for the separation of americium, neptunium, plutonium, thorium, curium and uranium in water. After completing this method, source preparation for measurement of actinides by alpha spectrometry is performed by electrolytic deposition onto stainless steel planchets (Eichrom Method SPA02) or by rare earth fluoride microprecipitation onto polypropylene filters (Eichrom Method 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 1 liter 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.

3. SIGNIFICANCE OF USE

3.1. This method allows for the isotopic analysis of six actinides from a single sample. The use of vacuum assisted flow and stacked cartridges enable completion of sample preparation for batches of 12-24 samples in as little as ten 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 TP01).

4.3. When neptunium is analyzed, a ^{236}Pu tracer must be used, instead of ^{242}Pu, to achieve a cleaner separation of the ^{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 coprecipitate 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

- 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
- Hotplate

- Inner support tube, Eichrom Part: AR-1000-TUBE-PE
- 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 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, $NaNO_2$
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, 2mL 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 (0.1M)* - 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 (3.2M)* - Dissolve 106g of $(NH_4)_2HPO_4$ in 200mL of water. Heat gently to dissolve. Dilute to 250mL with water.

6.3. *Ascorbic acid (1M)* - Dissolve 17.6 grams ascorbic acid in 80mL of water. Dilute to 100mL with water. **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 (0.1M) - hydrofluoric acid (0.05M) - titanium chloride (0.03M)*- Add 30mL of 10% $TiCl_3$, 4.2mL of concentrated HCl, and 0.9mL concentrated HF to 400mL of water. Dilute to 500mL with water. **Prepare immediately before 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 333 mL 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/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 (3M)* - Add 188mL of concentrated HNO_3 to 700mL of water. Dilute to 1L with water.

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

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

6.14. *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 of 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 1mL of 1.25 M $Ca(NO_3)_2$ to each beaker.

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

7.1.6.3. Turn down hot plate.

7.1.6.4. Add 0.75 mL of phenolphthalein indicator and 3mL of 3.2 M $(NH_4)_2HPO_4$ 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 calcium phosphate precipitate. Heat sample for 20-30 minutes.

7.1.6.6. Remove samples from hot plate, cool to room temperature, and allow the 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 using deionized water. Centrifuge the precipitate for approximately 10 minutes at 2000 rpm.

7.1.6.9. Decant the 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.

7.1.6.11. Dissolve precipitate in 5mL conc. HNO_3 and transfer to a 100mL beaker. Rinse centrifuge tube with 2-3mL of conc. HNO_3 and transfer to the same 100mL beaker. Evaporate to dryness.

7.2. Actinide Separations using Eichrom Resins:

7.2.1. Dissolve each precipitate with 12mL of 3M HNO_3 - 1M $Al(NO_3)_3$.

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, 7.2.2 through 7.2.5 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, add 0.1mL of 5 mg/mL iron solution. If Np is not required then this step in 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 3.5 M $NaNO_2$. Swirl to mix well.

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

7.2.6. Setup of TEVA and TRU cartridges on the vacuum box system

7.2.6.1. Place the inner tube rack into the vacuum box with a 50mL centrifuge tube for each sample to be analyzed. Alternatively, the vacuum box liner may be used here. Fit the lid to the vacuum box system.

7.2.6.2. Place the 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.

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, unused vacuum box holes can be sealed using scotch tape affixed to the vacuum box lid.

7.2.6.3. For each sample solution, fit the TRU cartridge onto the inner support tube. Attach 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 box. Turn the vacuum pump on and ensure proper fitting of the lid.
Add 5mL of 3M HNO_3 to each reservoir to precondition the TEVA and TRU cartridges. Adjust the vacuum pressure to achieve a flow rate of 1-2 mL/minute.

7.2.6.6. Transfer each load solution from step 7.2.5. into the appropriate reservoir. Allow the solution to pass the cartridges at a flow rate of 1-2 mL/minute.

7.2.6.7. Add 3mL of 3 M HNO_3 to rinse to each sample tube. Transfer the rinse into the appropriate reservoir. Allow the solution to pass through the cartridges at 1-2mL/min.

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

7.2.6.9. Turn vacuum off.

7.2.6.10. Separate TEVA cartridge from TRU cartridge. Set TRU cartridges aside while Np, Pu and Th are separated on TEVA.

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

7.2.7.1. Place each TEVA cartridge along with a clean reservoir on the appropriate opening on the vacuum box lid. Empty the solution from the centrifuge tubes below each cartridge and discard as waste.

7.2.7.2. Add 20mL of 3M HNO_3 to each reservoir. Turn on vacuum. Allow solution to pass through TEVA cartridges at 1-2mL/min.

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

7.2.7.4. Place tube rack with clean, labeled tubes under each cartridge. Placing clean inner support tubes and yellow outer tips below each TEVA cartridges at this point can help ensure a clean Th fraction in the following step.

7.2.7.5. Add 15mL of 9M HCl to each cartridge reservoir. Strip Th at 1mL/min. Turn off vacuum

7.2.7.6. Set Th samples aside for alpha source preparation.

7.2.7.7. Place clean, labeled tubes in the tube rack under each cartridge. Placing clean inner support tubes and yellow outer tips below each TEVA cartridges at this point can help ensure a clean Pu/Np fraction in the following step.

7.2.7.8. Add 20mL of 0.1M HCl/0.05M HF/0.03M $TiCl_3$ to each reservoir. Turn on Vacuum. Elute Np and Pu at 1mL/min.

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

7.2.7.9. Set Pu/Np samples aside for alpha source preparation.

7.2.8. 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 7.2.8.1. ^{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 7.2.8.2.

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

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

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

7.2.8.1.3. Remove TRU cartridge and set aside for uranium elution, step 7.2.8.3.

7.2.8.1.4. Attach a clean reservoir to each DGA cartridge.

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

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

7.2.8.1.7. 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.

7.2.8.1.8. Add 15mL of 0.25M HCl to each reservoir. Turn on vacuum. Elute Am and Cm at 1mL/min.

7.2.8.1.9. Set Am/Cm samples aside for alpha source preparation.

7.2.8.1.10. Place TRU cartridges from step 7.2.8.1.3 on vacuum box and GO TO step 7.2.8.3.

7.2.8.2. Am and Cm elution using TRU Resin

7.2.8.2.1. 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.

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

7.2.8.2.3. Set Am/Cm samples aside for alpha source preparation.

7.2.8.3. Uranium elution from TRU Resin

7.2.8.3.1. 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.

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

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

7.2.8.3.4. Set U samples aside for alpha source preparation.

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

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

9. 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., et al., "Rapid analysis of Emergency Urine and Water Samples", J. Radioanal. Nucl. Chem., 275(3), 497-502 (2008).

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 $Ca(NO_3)_2$.

4) Heat samples at medium setting for 30-60 minutes.

5) Remove samples from heat.

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) Cool to room temperature. 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.

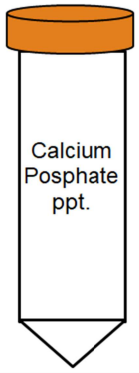
12) Add 10mL DI water to ppt. Mix well. Centrifuge. Decant supernate. Dissolve ppt with 5mL conc. HNO_3. Transfer to 100mL beaker.

13) Rinse centrifuge tube with 2-3mL conc. HNO_3. Transfer to 100mL beaker. Evaporate to dryness.

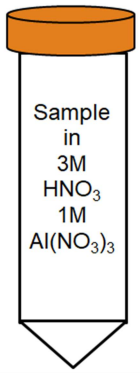
14) Dissolve reside in 16mL 3M HNO_3-1M $Al(NO_3)_3$. Add 1mL 1.5M Sulfamic Acid, 0.5 mL Fe, and 1mL 1M Ascorbic Acid. Swirl to mix. Wait 3-5 minutes.

15) Add 1mL 3.5M $NaNO_2$. Swirl to mix.

Water Sample in glass beaker. Acidify pH 2.



Centrifuge.
Decant Supernate.
Wash ppt with H_2O.
Centrifuge. Decant.



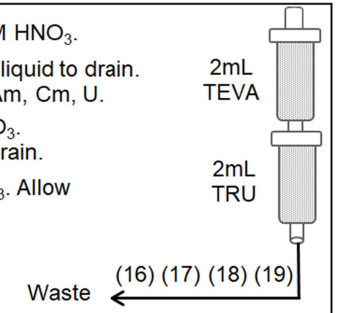
16) Precondition TEVA-TRU with 5mL 3M HNO_3.

17) Load sample onto TEVA-TRU. 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. Allow liquid to drain.

19) Rinse TEVA-TRU with 5mL 3M HNO_3. Allow liquid to drain.

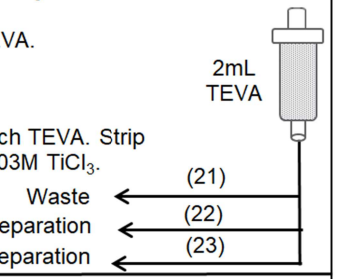
20) Separate TEVA and TRU cartridges.



21) Rinse TEVA column with 20mL 3M HNO_3.

22) Place clean centrifuge tube below TEVA. Strip Th with 15mL 9M HCl.

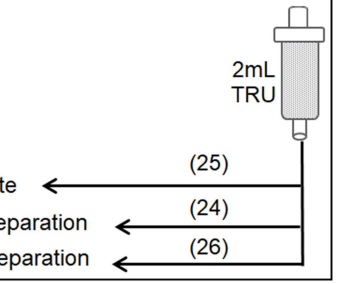
23) Place clean centrifuge tube below each TEVA. Strip Pu-Np with 20mL 0.1M HCl-0.05M HF-0.03M $TiCl_3$.



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.



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 $Ca(NO_3)_2$.
4) Warm samples at medium setting for 30-60 minutes.
5) Remove samples from heat.
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) Cool to room temperature. 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.
Water Sample in glass beaker. Acidify pH 2.
Calcium Posphate ppt.
Centrifuge. Decant Supernate. Wash ppt with H_2O. Centrifuge. Decant.
Sample in 3M HNO_3 1M $Al(NO_3)_3$
16) Precondition TEVA-TRU with 5mL 3M HNO_3.
17) Load sample onto TEVA-TRU. Allow liquid to drain. TEVA retaines Th, Pu, Np. TRU retains U, Am, Cm.
18) Rinse sample tube with 5mL 3M HNO_3. Add rinse to TEVA-TRU. Allow liquid to drain.
19) Rinse TEVA-TRU with 5mL 3M HNO_3. Allow liquid to drain.
20) Separate TEVA and TRU cartridges.
2mL TEVA
2mL TRU
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
(25) (26) (27) (28)
Waste
(29)
Am/Cm sample to source preparation
(31)
U sample to source preparation