Title: Determination of Actinides by Extraction Chromatography

Approvals (Signature/Date):

Kenneth Miller Technical Manager	1/7/2013 Date	Dave Harbinson Health & Safety Coordinator	1/7/2013 Date
Sarah Nagel Quality Assurance Manager	1/07/2013 Date	Jodie Carnes Laboratory Director	1/7/13 Date

1. SCOPE AND APPLICATION

1.1. This procedure describes a method for separation and measurement of isotopic plutonium, americium, curium, thorium, uranium, strontium, lead and Radium from water and a variety of environmental and bioassay samples. The sample preparation consists of either direct reduction of the sample or leachate to dryness, or the performance of a calcium phosphate precipitation (RL-PRP-010).

Refer to Policy P-R-01 for method detection limit information.

2. SUMMARY OF METHOD

After the initial sample preparation, the various isotopes are separated from other actinides by chromatographic resins prior to measurement by alpha spectrometry or gas flow proportional counter. Tracers are used to monitor chemical recoveries and yield correct analyte results.

3. DEFINITIONS

Chromatography – A method of separating and analyzing mixtures of chemical substances by chromatographic adsorption.

4. INTERFERENCES

4.1. Actinides with unresolvable alpha energies such as Np-237 and Th-230 must be chemically separated to enable measurement. This method separates these isotopes effectively. It has been demonstrated that high levels of phosphate can cause problems with these analyses, especially thorium. This is to be considered when selecting the sample preparation method.

4.2. Samples with large amounts of iron require oxidation state readjustment prior to running the americium portion of the method. This adjustment is accomplished with sulfamic and ascorbic acid. Iron (III) competes for sites on Eichrom TRU® resin and thus can diminish americium yields.

4.3. Np-237 can interfere with the Pu-242 peak if not removed from the column before eluting plutonium.

4.4. Thorium and uranium will not be analyzed sequentially by this procedure.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Minimum Personal Protective Equipment (PPE) for this SOP consists of safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes.

5.1. Specific Safety Concerns or Requirements

Do not put Hydrofluoric acid (HF) into glass containers. Always dispense HF from its original, drip-free shipping bottle.

WARNING: HF is one of the most corrosive acids known. Do not assume that dilute solutions do not require special precautions! Always double check your PPE before each use of HF. A pinhole in a glove or leaky container can cause an accident.

HF burns penetrate deeply into skin and muscle tissue and cannot be treated by simply flushing the area with water. HF causes delayed burns over several hours so immediate care is essential to prevent further harm.

FIRST AID MEASURES FOR HF ACID EXPOSURE

1. Flush area with cold water for at least 15 minutes.
2. Remove contaminated clothing immediately.
3. After washing exposed skin, use gloves to rub a generous amount of calcium gluconate gel into burn area.
4. For areas too large to apply gel, use an Epsom salts solution in a concentration of ½ to 1 cup of Epsom salts in one quart of iced water. Immerse the limb into a bucket of solution or soak the solution into gauze and apply to the wound. This dressing should be replaced or re-soaked every two minutes.
5. If area affected is greater than 2 inches by 2 inches, give 6 tablets of calcium gluconate orally.
6. If area affected is greater than 4 inches by 4 inches, assume significant inhalation injury and treat accordingly.

5.2. Primary Materials Used

The following is a list of the materials used in this method that have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Material Safety Data Sheet (MSDS) for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-TWA	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. **THE FLUORIDE ION READILY PENETRATES THE SKIN, CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous. It is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sodium Nitrite	Oxidizer Poison Reactive	None	**Danger! Strong oxidizer and toxic material. Heat, shock, or contact with other material may cause fire or explosive decomposition.** Causes irritation to the respiratory tract and systemic poisoning. Can irritate the mouth, esophagus, stomach, etc. Excessive amounts affect the blood and blood vessels. Signs and symptoms of nitrite poisoning include intense cyanosis, nausea, dizziness, vomiting, collapse, spasms of abdominal pain, rapid heart beat, irregular breathing, coma, convulsions, and death due to circulatory collapse. Causes irritation, redness and pain to the skin and eyes. May be absorbed through the skin causing systemic poisoning.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Balance, capable of reading 0.01 g.

6.2. Beakers, various sizes.

6.3. Centrifuge tubes 50 mL conical (plastic)

6.4. Extension funnel for cartridge

6.5. Hot plate.

6.6. Pipette, adjustable, capable of measuring 0.1 through 5 mL, non-critical, with disposable plastic tips.

6.7. Syringe filters

6.8. Vacuum box with disposable pipet tips

6.9. Vacuum pump and tubing

6.10. Watch glasses, various sizes.

7. REAGENTS AND STANDARDS

7.1. Reagents are prepared from analytical reagent grade chemicals unless otherwise specified below. Reagent water, which must have an electrical resistivity of 1 mega-ohm-cm or greater when obtained, is used throughout. Reagent water is obtained from the Nano-pure system. Label all reagents as outlined in procedure RL-RPL-001.

NOTE: Consult the MSDS for the properties of these reagents and how to work with them.

7.2. Ammonium iodide (NH_4I) – granular – NH_4I, FW 144.96 g/mol

7.3. Ammonium Bioxalate (0.1M $NH_4HC_2O_4$) – Dissolve 6.31 g of $H_2C_2O_4 \cdot 2H_2O$ (oxalic acid) and 7.11 g of $(NH_4)_2C_2O_4 \cdot H_2O$ (ammonium oxalate) in 900 mL of reagent water, and dilute to 1 L with reagent water.

7.4. Ascorbic acid - powdered form is preferred.

7.5. Ascorbic acid solution - 2.5 g. dissolved in 10 ml of reagent water.
Prepare solution each day of use (i.e. within a 24 hour period).

7.6. Hydrochloric acid (12M HCl) – concentrated hydrochloric acid (sp gr 1.19)

7.7. Hydrofluoric acid (28M HF) - concentrated HF (sp gr 1.2).

7.8. Sodium Nitrite (3M) – Dissolve 207.0 g of $NaNO_2$ in 500 mL water and dilute to 1L with reagent water. Or analyst may prepare according to sample load, i.e. 2.07g of $NaNO_2$ in 5 mL reagent water and dilute to 10 mL.
Prepare solution each day of use (i.e. within a 24 hour period).

7.9. Sodium Nitrite – granular – ($NaNO_2$), FW 69.00 g/mol

7.10. Hydrochloric acid (4M HCl) - Add 333 mL of concentrated HCl to approximately 500 mL of reagent water. Dilute to 1 liter with reagent water and mix well. **CAUTION**: Corrosive.

7.11. Hydrochloric acid (4M HCl) – 0.1M Hydrofluoric acid (0.1M HF) - Add 333 mL of concentrated HCl to approximately 500 mL of reagent water. Add 3.6 mL of HF and dilute to 1L with reagent water and mix well.
Do not prepare or store solution in glass. CAUTION: Corrosive.

7.12. Hydrochloric acid (8M HCl) - Add 667 mL of concentrated HCl to approximately 100 mL of reagent water. Dilute to 1L with reagent water and mix well. **CAUTION**: Corrosive.

7.13. Hydrochloric acid (9M HCl) - Add 750 mL of concentrated HCl to approximately 100 mL of reagent water. Dilute to 1L with reagent water and mix well. **CAUTION**: Corrosive.

7.14. Hydrochloric acid (0.1M HCl) - hydrofluoric acid (0.05M HF) – ammonium iodide (0.1M NH_4I) solution -- Add 8.33 mL concentrated HCl and 1.78 mL of concentrated HF and 14.49g NH_4I to 400 mL of water and dilute to 1L with water. **Do not prepare or store solution in glass. Prepare solution each day of use (i.e. within a 24 hour period).**

7.15. Hydrogen peroxide (30% H_2O_2). **WARNING**: Corrosive and Oxidizer.

7.16. Nitric acid (HNO_3) Concentrated. **CAUTION**: Corrosive and Oxidizer.

7.17. Nitric acid (3M HNO_3) - Add 191 mL of concentrated HNO_3 to approximately 700 mL of reagent water. Dilute to 1L with reagent water and mix well. **CAUTION**: Corrosive and Oxidizer.

7.18. Nitric acid (3M HNO_3) - Aluminum Nitrate solution (1M $Al(NO_3)_3$) - Dissolve 375 g of aluminum nitrate non-hydrate or 212 g anhydrous aluminum nitrate in approximately 500 mL of reagent water and add 191 mL of concentrated HNO_3. Dilute to 1L with reagent water and mix well. Pass solution through a UTEVA column prior to using, to extract any uranium present.

7.19. Nitric acid (0.5M HNO_3) - Add 32 mL of concentrated HNO_3 to approximately 900 mL of reagent water. Dilute to 1L with reagent water and mix well. **CAUTION:** Corrosive and oxidizer.

7.20. Sulfamic acid (NH_2SO_3H) Caution – causes severe irritation and burns upon contact.

7.21. Sulfamic acid (NH_2SO_3H) solution Dissolve 2.0 grams sulfamic acid in 10 ml of reagent water.

7.22. Titanium trichloride, $TiCl_3$ (Titanous Chloride) 20% solution, commercially available.

7.23. PB resin - prepackaged cartridges

7.24. SR resin – prepackaged cartridges

7.25. TEVA resin - prepackaged cartridges.

7.26. TRU resin - prepackaged cartridges.

8. SAMPLE COLLECTION, PRESERVATIVES, SHIPMENT AND STORAGE

8.1. The sample may be collected in glass or plastic containers. Storage of the sample prior to analysis should not exceed six months.

8.2. It is recommended that water samples be preserved at the time of collection by adding enough 1M HNO_3 to the sample to bring it to pH 2.

9. QUALITY CONTROL

9.1. All quality control (QC) data shall be maintained and available for easy reference.

9.2. Yield monitors (carriers and tracers) and QC spikes are prepared with a pre-set mass and/or activity and distributed appropriately in coded vials for use during sample analysis. Consult the latest version of the client specific Quality Assurance Summary (QAS) for the appropriate yield monitors, spikes, carriers, and/or tracers to use.

9.3. Consult the QAS for client-specific information regarding QC frequency.

9.4. Refer to SOP RL-DR-001 for sample and QC data acceptance criteria and corrective action.

10. CALIBRATION

10.1. Refer to appropriate detector SOP.

11. PROCEDURE

NOTE: If any parameter is found to be out of limits, consult supervision. Also, issue a Nonconformance Memo to the Quality Assurance Group.

NOTE: One time procedural variations are allowed if deemed necessary by the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall require approval by supervision and immediate notification of the Quality Assurance Group. If contractually required, the client shall be notified prior to any procedure changes. Complete a Nonconformance Memo and forward to the Quality Assurance Group within one day of the supervisor's approval. The Nonconformance Memo will be filed in the project file.

NOTE: To resolve problems with instrumentation or support equipment when the solution is not contained in this SOP, refer to SOP RL-QA-005 Troubleshooting Guide.

11.1. Sample Preparation:

11.1.1. Determine if sample can be reduced to dryness by direct evaporation. If so, proceed to step 11.1.2.

NOTE: For urine samples for Am, Pu and isotopic U, and environmental samples for isotopic U, it may be necessary to prepare the sample by calcium phosphate precipitation using SOP RL-PRP-010. When preparation is complete, proceed with step 11.2.

NOTE: In some water samples, calcium sulfate precipitate may form during evaporation. If this occurs, prepare the sample by calcium phosphate precipitation using SOP RL-PRP-010. When preparation is complete, proceed to step 11.2.

NOTE. If Th is requested, RL-PRP-010 should NOT be used.

11.1.2. Gently evaporate the sample to dryness and wet ash by dissolving the residue with 16M HNO_3 and adding small amounts of H_2O_2 while heating the samples. Evaporate to dryness

11.2. Actinide Separations using Eichrom resins:

11.2.1. Dissolve the sample residue with approximately 20 mL of 3M HNO_3-1M $Al(NO_3)_3$ (load solution) and swirl to dissolve the precipitate or residue. If necessary, the beakers may be lightly warmed, if covered with a non-ribbed watch glass. Cool to room temperature.

NOTE: An additional 5-10 mL may be necessary if the precipitate volume is large. Consult supervision if more than 30 mL is required

NOTE: When thorium is requested, and significant amounts of phosphate are suspected, increased amounts of load solution may be necessary. 40 mL of load solution is recommended for urine.

If only thorium is required, proceed to step 11.3.

11.2.2. Add 1 mL ascorbic acid solution to the sample cover with a watch glass and warm for approximately 5 minutes.

11.2.3. Add 1.5 mL 3M Sodium Nitrite solution to each beaker, swirling to mix and warm for approximately 5 minutes.

NOTE: If additional load solution was used, add 0.5 mL of 3M sodium nitrite solution for every 5 mL addition.

11.2.4. Remove samples from heat and let cool until they are near room temperature

11.3. Multi resin separation.

11.3.1. Set up vacuum box per SOP RL-ALP-017.

11.3.2. For each sample solution, place a TEVA cartridge on the vacuum box. If americium and/or uranium is also required, attach a TRU cartridge beneath the TEVA cartridge (see NOTE below). If Sr or Pb is

requested attach a SR or PB cartridge beneath the TRU Label each cartridge with the appropriate sample ID. Place a waste collecting container in box.

NOTE: If Am is requested and the samples may contain Iron, the samples will require an additional oxidation state adjustment after passing through the TEVA cartridge but prior to loading on the TRU cartridge. For these samples, only attach the TEVA cartridges to the vacuum box and collect the load, beaker rinse and 15 ml 3M HNO_3 Rinse in labeled centrifuge tubes.

NOTE: Some samples may need to be centrifuged or filtered at this point. If there are visible solids present, silicates are suspected, or there is the possibility of other interferences, the solutions must be filtered or centrifuged. Otherwise the very fine mesh of the TEVA cartridges will become clogged and flow will be diminished.

NOTE: The sample must be clear of any undissolved material at this time. If this material persists contact supervision.

11.3.3. Just prior to loading the sample, add 5 mL of 3M HNO_3 into each reservoir (to condition the resin) and pass through the cartridge with vacuum at approximately 3 mL/minute.

NOTE: if the additional oxidation state adjustment is not necessary and Radium analysis is requested place clean labeled tubes in the vacuum box and collect the waste from steps 11.3.4 through 11.3.6for Radium analysis.

11.3.4. Transfer each sample solution into the appropriate cartridge reservoir using a syringe filter. Allow the solution to pass through the cartridge at a flow rate of 1 mL/min.

11.3.5. Rinse each beaker with 5 to 10 mL of 3M HNO_3. Transfer each rinse through filter into the appropriate cartridge.

11.3.6. Add 15 mL of 3M HNO_3 to each reservoir and allow to pass through the cartridge at a flow rate of 3 mL per minute.

NOTE: if the load and rinses are being collect for oxidation state adjustment, remove the tubes and set aside for step 11.4.3

11.3.7. If multiple cartridges were required, separate them. The TEVA cartridge can be set up on a vacuum box and continue as follows. The TRU cartridges can be set aside for step 11.4 If thorium was requested place a clean, labeled centrifuge tube under each cartridge.

NOTE: For Sr set up the SR cartridges from step 11.3.2 and continue from step 11.2.4 of RL-GPC-010. For Pb set up the PB cartridges from step 11.3.2 and continue from step 11.19 of RL-GPC-011.

11.3.8. Add 20 mL of 9M HCl into each reservoir and allow it to pass through and collect eluate for Th analysis.

NOTE: This rinse converts the resin to the chloride form. Some Np may be removed.

11.3.9. Add 5 mL of 6M HCl solution into each reservoir and allow it to pass through the cartridge and collect this in the same tubes that were used in the previous step.

NOTE: This rinse removes neptunium and thorium from the column. The 9M HCl and 6M HCl rinses also remove any residual ferrous ions that might interfere with electrodeposition.

11.3.10. Remove the tubes containing the Th. fraction and place a clean, labeled centrifuge tube under each cartridge.

11.3.11. Transfer the thorium HCl solution, with approximately 5 ml of concentrated HNO_3. to a clean labeled beaker and evaporate to near dryness. Proceed to either the electrodeposition procedure (RL-ALP-015) or the micro-coprecipitation procedure (RL-ALP-016).

11.3.12. **For routine plutonium samples**: Pipet 20 mL of 0.1M HCl-0.05M HF-0.1M NH_4I solution into each reservoir to elute the plutonium. Allow the liquid to pass through cartridge at a flow rate of 1 mL per minute.

NOTE: For samples high in Np-237, elute the plutonium with 20 ml of 8M HCl and 0.4 ml of $TiCl_3$. Mix the HCl and the $TiCl_3$ by adding 10 ml of the 8 M HCl to the column, pipetting 0.4 ml of $TiCl_3$, and then adding the remaining 10 ml of 8 M HCl. Collect the eluant and proceed with micro-coprecipitation. Electrodeposition is not an option with this eluant since the Titanium would also electrodeposit.

11.3.13. Transfer each sample to a small, labeled, beaker with small amounts of concentrated HNO_3 and evaporate to dryness.

11.3.14. Wet ash each sample with concentrated HNO_3 and 30% H_2O_2 at least once. Proceed with SOP RL-ALP-015 (Electrodeposition) or RL-ALP-016 (Coprecipitation).

11.4. Separation of americium from uranium using TRU resin:

11.4.1. Attach the TRU cartridge from step 11.3.8 to the vacuum box and insert the waste container.

NOTE: For samples requiring additional oxidation state adjustment, set up the vacuum box with a new TRU cartridges for Am/Cm/U; if Pb or Sr are requested also attach a PB or SR cartridge beneath the TRU cartridge. Label each cartridge with the appropriate sample ID. If radium is also requested collect the waste from step 11.4.4 for Radium analysis

11.4.2. Rinse the funnel and/or reservoir with 5 ml of 3M HNO_3 and allow to drain to waste and then continue with step 11.4.3 if oxidation state adjustment is required. If oxidation state adjustment is NOT required proceed to step 11.4.5.

11.4.3. If iron is suspected slowly add to the combined load and rinse from the TEVA column from 11.3.2 2.5 ml of NH_2SO_3H solution with swirling. The solution may bubble as the nitrite in the sample reacts with the NH_2SO_3H.

11.4.3.1. After the solution stops bubbling, add 3 ml of the ascorbic acid solution and heat the solution for approximately 3 minutes.

11.4.3.2. Allow sample to cool to approximately room temperature.

11.4.4. Pass the solution from step 11.4.3.1 through the column at a flow rate of approximately 1ml per minute.

NOTE: if additional oxidation state adjustment was performed allow the solution to pass through the column then separate the labeled TRU and SR or PB cartridges. For Sr set up the SR cartridges from step 11.4.1 and continue from step 11.2.4 of RL-GPC-010. For Pb set up the PB cartridges from step 11.4.1 and continue from step 11.19 of RL-GPC-011.

11.4.5. **Continue with the TRU cartridge:** Add 5mL of 0.5M HNO_3 to the reservoir and allow to pass through.
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NOTE: 0.5M HNO_3 is used to lower the nitrate concentration prior to conversion to the chloride form and remove the nitrite ion.

11.4.6. Remove the waste container and place a clean labeled centrifuge tube under each column to elute the americium. If electrodeposition is requested, add a pre-filter to the column.

11.4.7. Add approximately 20 mL of 4M HCl to each reservoir to elute the americium. Allow the 4M HCl to pass through the cartridge at a flow rate of 1 mL per minute.

11.4.8. Transfer each sample to a small, clean, labeled beaker with small amounts of reagent water, and evaporate to dryness.

11.4.9. Wet ash each sample with concentrated HNO_3 and 30% H_2O_2 at least once. Proceed with SOP RL-ALP-015 (Electrodeposition) or RL-ALP-016 (Coprecipitation).

11.4.10. If isotopic U is required, continuing with the TRU cartridge, place a waste container into the vacuum box.

11.4.11. Rinse the cartridge reservoirs with 25 mL of 4M HCl-0.1M HF and allow the solution to pass through the cartridge at a flow rate of 3 mL per minute.

NOTE: 4M HCl-0.1M HF is used to selectively remove any residual thorium that may be present on the TRU cartridge and leave the uranium.

11.4.12. Place clean, labeled centrifuge tubes below each cartridge.

11.4.13. Add 10 mL of 0.1M $NH_4HC_2O_4$ to elute the uranium and allow to pass through the cartridge at a flow rate of 1 mL per minute.

11.4.14. Transfer each sample to a small, clean, labeled beaker with small amounts of reagent water, and evaporate to dryness.

11.4.15. Wet ash each sample with concentrated HNO_3 and 30% H_2O_2. Proceed with SOP RL-ALP-015 (Electrodeposition) or RL-ALP-016 (Coprecipitation).

11.4.16. Rinse each cartridge with 5-10 mL of water prior to disposal.

NOTE: Consult technical supervision prior to attempting the electrodeposition of americium by this method. Special cleanup is need to remove organics that "poison" the electrodeposition procedure.

11.5. Plutonium 241 analysis

11.5.1. After the sample has been analysed by alpha spectrometry, strip the sample from the disc following RL-ALP-015 (electrodepostition) or RL-ALP-016 (coprecipitatio0n).

11.5.2. Evaporate the solution to dryness. Wet ash the sample with concentrated HNO_3 and 30% H_2O_2 until the solution is clear. Add 2 ml of concentrated HCl and evaporate to dryness.

11.5.3. Add 2 ml of 1M HCl to the beaker. Warm the sample and then transfer the solution to a labeled plastic liquid scintillation vial.

11.5.4. Repeat step 11.5.3 three times using 1 ml of reagent water instead of 2 ml of 1M HCl. Do not exceed 5 ml of solution in the vial. Add 15 ml of liquid scintillation cocktail to the vial, and shake vigorously. Prepare samples for the count room.

11.5.5. Instrument blanks are prepare by adding 2 ml of 1M HCl, 3 ml of reagent water and 15 ml of liquid scintillation cocktail to a plastic liquid scintillation vial, and mixing thouroughly.

12. DATA ANALYSIS AND CALCULATIONS

12.1. For computer calculation of the sample activity and its total propagated uncertainty (TPU), consult the RadCalc Users Guide. The complete computer calculation includes blank subtraction if requested by the customer and complete error propagation.

13. METHOD PERFORMANCE

13.1. The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use.

13.2. Method Demonstration of Capability documentation is maintained in the quality files.

14. POLLUTION PREVENTION

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on

anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. This method produces only 120 mL of aqueous waste from the column separation steps. This method produces less than 10% of the waste volume produced by traditional actinide separation procedures. In addition, this method does not produce mixed organic waste as is produced by more traditional actinide separation procedures.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Health and Safety Coordinator should be contacted if additional information is required.

16. REFERENCES

16.1. Chieco, N.A, ed. 1997 EML Procedures Manual, HASL-300, 28th Edition, Volume I., Method Se-03, Environmental Measurements Laboratory, US Department of Energy, New York, New York.

16.2. Horwitz, E.P., et al., 1992, "Separation and Preconcentration of Uranium from Acidic Media by Extraction Chromatography," **Analytica Chimica Acta**. 266, 25-37.

16.3. Maxwell, S.L., et al., October 1993, "High Speed Separations to Measure Impurities in Plutonium-238 Oxide and Trace Radionuclides in Waste," **34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,** Gatlinburg, TN.

16.4. Maxwell, S.L., et al., May 2002, "Rapid Separation Methods for Bioassay Samples," **Eichrom Technologies, Inc. North American Users' Group Workshop,** Westminster, CO.

16.5. Nelson, D., November 1992, "Improved Methods for the Analysis of Radioactive Elements in Bioassay and Environmental Samples," **38th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry**, Santa Fe, NM.

16.6. CW-E-M-001, TestAmerica Corporate Environmental Health and Safety Manual, latest revision

16.7. P-R-01, Minimum Detectable Concentration Determination, latest revision

16.8. RadCalc DB, Users Guide, Richland, latest revision

16.9. RL-QAM-001, TestAmerica Quality Assurance Manual, latest revision

16.10. Associated SOPs

16.10.1. RL-ALP-015 Electrodeposition of Actinides, latest revision

16.10.2. RL-ALP-016 Coprecipitation of Some Actinides on Neodymium Fluoride for Alpha-Particle Spectrometry, latest revision

16.10.3. RL-ALP-017 Chromatographic Column Preparation, latest revision

16.10.4. RL-DR-001 Review of Bioassay and Environmental Data, latest revision.

16.10.5. RL-GPC-010 Separation of Strontium-90, latest revision.

16.10.6. RL-GPC-011 Determination of Lead-210 by Extraction Chromatography, lates revision.

16.10.7. RL-PRP-010 Urine and Water Sample Preparation by Calcium Phosphate Precipitation, latest revision

16.10.8. RL-RPL-001 Reagent and Non-Radioactive Standard Labeling, latest revision

16.10.9. RL-QA-005 Troubleshooting Guide, latest revision.

17. MISCELLANEOUS

17.1. Responsibilities

17.2. Analyst: Implements SOP as written.

17.3. Counting Room: Performs review on raw instrument data.

17.4. Technical Data Reviewer: Performs final data review.

17.5. Project Manager: Confirms final review and prepares data for reporting to client.

17.6. QA Manager: Performs product quality assessments as defined in the Quality Assurance policies.

17.7. Records Management/Documentation

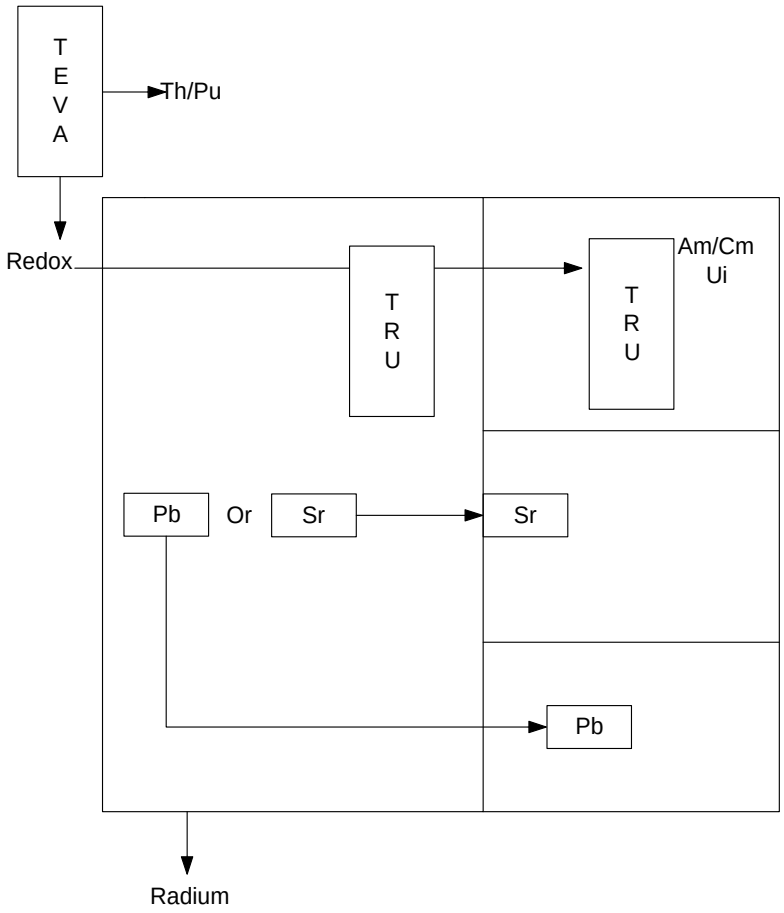
17.7.1. All records generated by this analysis will be filed and kept in accordance with the Richland facility of TestAmerica QA policies for records management and maintenance.

18. REVISION HISTORY

- Revision 1, 11/20/2008
  - Added sequential uranium analysis steps.
- Revision 2, 2/11/10
  - Updated HASL300 Reference
  - Reformatted outline and body of text
- Revision 3, 10/31/2010
  - Referenced RL-QA-005
  - Moved note regarding iron form after step 11.3.2 to after step 11.3.3
  - Clarified step 11.4.2
- Revision 4, 07/16/2012
  - Step 1 added ‘Sr, Pb & Ra’ to scope.
  - Section ‘11 Procedure’ was amended to describe the separation of Sr, Pb & Ra
  - Section ‘11 Procedure’ clarified instructions relating to oxidation state adjustment
  - Step 11.3.12 order changed to emphasize the routine sample information
  - Section 11.5 Plutonium 241 analysis was added
  - Updated formatting through procedure
  - Procedural Flow Charts updated.

19. PROCEDURAL FLOW CHART

A. With Additional Oxidation State Adjustment.



Start with only the TEVA cartridge
Condition w/ 3M HNO3
Load w/ HNO3/Al
Rinse w/ 3M HNO3
Collect load and rinse for Redox
Elute Th w/ 9M and 6M HCl
Elute Pu w/ HCl/HF/NH4I

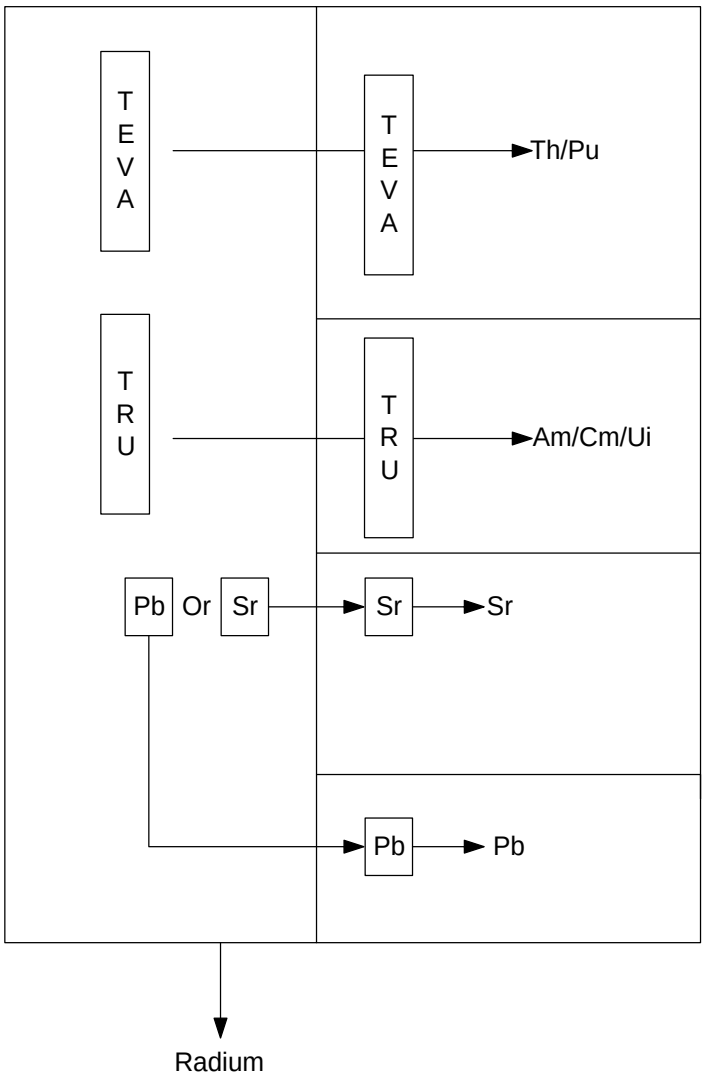
attach TRU on top with a SR or PB beneath
Condition w/ 3M HNO3
Load adjusted solution
Rinse w/ 3M HNO3
Collect load and rinse for Ra
Seperate cartridges

TRU
Rinse w/ 3M HNO3
Rinse w/ 0.5M HNO3
Elute AM/Cm w/ 4M HCl
Rinse w/ 4M HCl/HF
Elute Ui w/ bioxalate

SR
follow GPC-010

PB
Follow GPC-011

B. Without Additional Oxidation State Adjustment.



attach cartridges With TEVA on top. TRU beneath TEVA and the PB or SR beneath TRU.

While cartridges are together
Condition w/ 3M HNO3
Load w/ HNO3/Al
Rinse w/ 3M HNO3
Collect load and rinse for Ra
Seperate cartridges

TEVA
Elute Th w/ 9M and 6M HCl
Elute Pu w/ HCl/HF/NH4I

TRU
Rinse w 3M HNO3
Rinse w/ 0.5M HNO3
Elute Am/Cm / 4M HCl
Rinse w/ 4M HCl/HF
Elute Ui w/ Bioxalate

SR
Follow GPC-010

PB
Follow GPC-011