

Idaho National Laboratory

RADIOSTRONTIUM DETERMINATION BY SOLID PHASE EXTRACTION AND GAS-FLOW PROPORTIONAL COUNTING	Identifier: AL-3500-LI-005	Page: 1 of 36
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Materials and Fuels Complex	Laboratory Instruction	USE TYPE 4	eCR Number: 607921
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Manual: MFC Analytical Laboratory Manual

PROCEDURE REVIEW REQUIREMENTS PER SP-20.1.4			
DISCIPLINE	REVISION	DISCIPLINE	REVISION
PIE FACILITIES (HFEF, NRAD, TREAT)	N/A	INTER-FACILITY TRANSFERS	N/A
RESEARCH LABS (AL, EFF, EML, FASB, IMCL, RCL)	X	MAINTENANCE	N/A
SECURE FACILITIES (FMF, SSFSF, ZPPR)	N/A	NUCLEAR SAFETY REVIEW	N/A
SPENT FUEL FACILITIES (FCF, MSCC, ORSA, RSWF, SCMS, SSB, TREAT-WH)	N/A	OUTSIDE REVIEW	N/A
BALANCE OF PLANT	N/A	PACKAGING AND TRANSPORTATION	N/A
CUI REVIEW	N/A	QUALITY	N/A
ENGINEERING	N/A	RADIOLOGICAL CONTROLS	*
ENVIRONMENTAL	*	SAFEGUARDS AND SECURITY	N/A
FIRE PROTECTION	N/A	S&T	N/A
HOISTING AND RIGGING	N/A	TRAINING	*
INDUSTRIAL HYGIENE	*	WASTE GENERATOR SERVICES	*
INDUSTRIAL SAFETY	*		

*DOCUMENT OWNER OR QUALIFIED REVIEWER SHALL DETERMINE THE NEED FOR THESE REVIEWS
BASED UPON THE SCOPE OF THE CHANGE AND THE HAZARDS IDENTIFIED

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1. PURPOSE/SCOPE/APPLICABILITY

This procedure provides instructions to use a solid phase extraction material to separate strontium from other fission products. The sample is loaded on the extraction cartridge in 8 M nitric acid and eluted with American Society for Testing and Materials (ASTM) Type I water. The total radiostrontium activity is determined by gas flow proportional counting (GFPC). Elemental strontium yield is determined gravimetrically.

Radiostrontium is rapidly separated from fission products, making this procedure particularly applicable to aqueous samples, health physics smears, or filter samples requiring results a few hours after collection. Solid samples are acid-leached (see Subsection 5.1) or prepared by the appropriate dissolution technique prior to radiostrontium separation. Radiostrontium may also be separated from the cartridge load waste from AL-3500-LI-006, "Selective Actinide Separation by Solid Phase Extraction."

This method quantifies total radiostrontium. Strontium values on material that has not been exposed to a neutron flux for 500 days can be considered 90Sr.

Extraction chromatography is used in this method to separate strontium. The three major components of an extraction chromatography system include an inert support, a stationary phase, and a mobile phase. Sr Resin, available from Eichrom Industries, uses an inert organic polymer resin as the support and 1 M 4,4'(5')-di-*t*-butylcyclohexano 18-Crown-6 (crown ether) in 1-octanol as the stationary phase. Because the uptake of Sr on the resin increases with increasing nitric acid concentration, an 8 M nitric acid solution is used to load Sr onto the extraction cartridge and water is used to elute it from the cartridge.

Greater than 99% of the fission products and contaminants will elute from the cartridge in the load step and subsequent acid rinse step. The major known interference is barium, which is also retained on the cartridge. Since barium's uptake peak occurs in approximately 3 M nitric acid, loading and rinsing the cartridge with 8 M nitric acid washes barium from the cartridge.

The elution curve for strontium is very sharp with >98% eluting in the first 5-mL fraction of water. A further separation by precipitation of strontium carbonate is performed and the precipitate is filtered and the chemical yield is determined gravimetrically via stable strontium carrier.

This procedure is performed by qualified personnel within the Analytical Laboratory (AL).

The activities directed by this procedure have been designated Quality Level 3 per Quality Level Determination MFC-000642.

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2. RISK AND CONTROLS

Sequence of Basic Activities	Potential Hazard	Hazard Control
1. Working with non-routine samples.	1. Radiation contamination/exposure.	1. Perform all work under an applicable RWP.
2. Working inside of and exiting an RBA.	2. Radiation exposure and/or release of radioactive material.	2. 1) Verification surveys of miscellaneous paperwork to perform tasks (data sheets, logs, rounds sheets, work orders, procedures); personal property (jewelry, watches, glasses, pens/pencils, keys); flashlights, dosimetry, portable communication equipment, and security equipment may be made by any trained radiological worker. If there is any indication of radioactive contamination, contact a HPT. 2) Except for the items listed above, HPT survey is required on all materials, items, tools, and equipment prior to exit from a RBA. 3) Obey all radiological postings.
3. Working in a CA.	3. Exposure to contamination.	3. 1) Limit the potential for contamination spread whenever possible (that is, use of a glove bag in a hood). 2) Follow the requirements of the applicable RWP.

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Sequence of Basic Activities	Potential Hazard	Hazard Control
4. Working in a known or suspected RA, HRA or HRA-ACR.	4. Radiological exposure.	4. 1) Based on survey results consider the use of temporary shielding to maintain personnel dose ALARA. 2) Follow the requirements of the applicable RWP. 3) Stage all tools, parts, materials, etc. prior to entering a RA, HRA, or LHRA to reduce time personnel are in the area.
5. Handling of a carcinogen (if known to be from an area where carcinogens were present).	5. Carcinogen/lead exposure.	5. 1) Contact IH to determine the need for exposure assessment, monitoring activities, work practices, and control measures (PPE, HEPA filters, post regulated areas, entry and exit logs, etc.). 2) Based on exposure assessment place employee on medical surveillance.
6. Working with P-10 gas (Ar and CH4).	6. Gas explosion and/or asphyxiation.	6. 1) The gas cylinder valves shall remain shut when not in use and when empty. 2) Store gas cylinders in a properly ventilated area. 3) If a concentrated P-10 gas leak occurs, evacuate the area and immediately notify Facility Management.

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Sequence of Basic Activities	Potential Hazard	Hazard Control
7. Collecting and storing samples.	7. Improper storage.	4) In the event of a concentrated gas inhalation, move the affected person to fresh air and administer CPR if necessary. Seek medical attention by dialing 777.
7. Collecting and storing samples.	7. Improper storage.	7. 1) Storage of samples is limited to 1 year per company procedures; however, some samples may require storage for greater than one year for performance and demonstration testing. 2) Samples may be temporarily stored by a laboratory following testing provided they are being held for a specific purpose. Through negotiations with DEQ, as documented in previous Notices of Violation, agreement has been reached that storage of samples for greater than one year must be accompanied by specific, documented authorization. If samples will be retained in the laboratory or other location after analysis and not sent back to the generator or dispositioned as waste within 30 days, the responsible Manager needs to maintain documentation listing the samples proposed for extended storage and stating the reason for storage, the length of expected storage, and the storage location.

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Sequence of Basic Activities	Potential Hazard	Hazard Control
		3) This documentation must be updated annually to justify continued storage until the sample is used, returned to the generator or dispositioned as waste. Samples that undergo a hazardous waste determination and are found to be non-hazardous are exempt from this requirement. 4) Samples no longer required by the project will be returned to the originator. If unable to return samples to the generator, contact WGS for proper disposal.
8. Operating sample changing gas flow proportional counter.	8. Pinching or crushing fingers.	8. 1) Maintain safe body position. 2) Verify objects and materials are clear of sample changer.
9. Use of chemicals.	9. Chemical exposures.	9. 1) Read and understand the MSDS information for each chemical in use. 2) Minimum required PPE unless otherwise noted: Nitrile or neoprene gloves changed out upon chemical contact, safety glasses with side shields and lab coat. 3) Emergency eyewash and shower is operational and readily accessible. 4) Chemicals are stored properly in designated locations.

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Sequence of Basic Activities	Potential Hazard	Hazard Control
	Chemicals used: <ul style="list-style-type: none"> • 18-Crown-6 • Ion exchange and eichrom resins • Phenolphthalein Indicator 	5) Special requirements: <ul style="list-style-type: none"> • Neoprene or nitrile gloves, lab coat, safety shoes, and safety glasses. • Neoprene or nitrile gloves, lab coat, safety shoes, and safety glasses. • Neoprene or Nitrile gloves, lab coat, safety shoes, and safety glasses.
10. Use of flammable/combustible liquids.	10. Fire and/or exposure to irritation Flammable/Combustible liquids used: <ul style="list-style-type: none"> • Ethanol/Methanol/Octanol • Ethanol 	10. 1) Use away from ignition sources. 2) Provide suitable fire extinguishing media. 3) Special requirements: <ul style="list-style-type: none"> • Butyl rubber gloves, Nitrile, or Neoprene gloves changed out <1-hour use, safety glasses with side shields and lab coat. • Neoprene or nitrile gloves changed every <4 hours, lab coat, safety shoes, and safety glasses.
11. Use of oxidizers.	11. Fires, irritation, burns	11. 1) Use away from combustible materials. 2) Emergency eyewash and shower is operational and readily accessible. 3) Segregate oxidizers away from organics and other incompatible chemicals and materials.

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	Oxidizers used: <ul style="list-style-type: none"> • Strontium Nitrate 	4) Special requirements: <ul style="list-style-type: none"> • Neoprene or nitrile gloves, lab coat, safety shoes, safety glasses.
12. Use of corrosives, toxics, and irritants.	12. Exposure to chemical burns, toxic effects and/or irritation Corrosives, toxics, and irritants used: <ul style="list-style-type: none"> • Ammonium Carbonate • Ammonium Hydroxide, Concentrated. • Nitric Acid • Sodium Carbonate • Strontium Carbonate (SrCO₃) 	12. 1) Emergency eyewash and shower is operational and readily accessible. 2) Minimum Required PPE: Butyl rubber or Neoprene gloves, safety glasses with side shield and lab coat. <ul style="list-style-type: none"> • Neoprene or nitrile gloves, lab coat, safety shoes, and safety glasses. • Neoprene or nitrile gloves, lab coat, safety shoes, and safety glasses. • Neoprene gloves or PVC gloves changed every <4 hours, lab coat, safety shoes, and safety glasses. • Neoprene or nitrile gloves, lab coat, safety shoes, and safety glasses. • Neoprene gloves, lab coat, safety shoes, and safety glasses.

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Sequence of Basic Activities	Potential Hazard	Hazard Control
	<ul style="list-style-type: none"> • Strontium Carrier (85Sr solution in 1% HNO₃) • Sulfuric Acid. 	<ul style="list-style-type: none"> • Neoprene or nitrile gloves, lab coat, safety shoes, and safety glasses. • Neoprene, nitrile, or PVC gloves changed every <4 hours, lab coat, safety shoes, and safety glasses.
<p>13. Using Hydrofluoric acid >1% in quantities >60 mL.</p>	<p>13. Hydrofluoric acid.</p>	<p>13. 1) Neoprene or butyl rubber gloves changed upon chemical contact, goggles and face shield or goggles and secondary barrier (for example, hood sash), lab coat, safety shoes, and protective apron.</p> <p>2) Calcium Gluconate gel will be available in working area when working with HF.</p> <p>3) In a hot cell, no PPE is required.</p> <p>4) In a glovebox, PPE is one pair of neoprene gloves for work less than 4 hours or changed upon chemical contact.</p>

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Sequence of Basic Activities	Potential Hazard	Hazard Control
<p>14. Using Hydrofluoric Acid >1% in quantities <60 mL.</p>	<p>14. Corrosive, HF exposure to the eyes may result in blindness or permanent eye damage. HF readily penetrates human skin, allowing it to destroy soft tissues and decalcify bone. If the exposure covers a large amount of skin surface area, excessive amounts of calcium can be inactivated leading to systemic fluoride poisoning, hypocalcaemia, and hypomagnesaemia. Heart function is diminished in hypocalcaemia (reduced calcium levels in the blood), whereby the heartbeat becomes abnormal, and cardiac arrhythmia (ventricular fibrillation) can occur.</p>	<p>14. 1) Neoprene or Butyl Rubber gloves changed upon chemical contact; safety glasses with secondary barrier (for example, hood sash) or goggles, lab coat; safety shoes; and protective apron. 2) Calcium Gluconate gel will be available in working area when working with HF. 3) In a hot cell, no PPE is required. 4) In a glovebox, PPE is one pair of neoprene gloves for work less than 4 hours or changed upon chemical contact.</p>

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Sequence of Basic Activities	Potential Hazard	Hazard Control
15. Using Hydrofluoric Acid <1% in a hood.	15. Corrosive, HF exposure to the eyes may result in blindness or permanent eye damage. HF readily penetrates human skin, allowing it to destroy soft tissues and decalcify bone. If the exposure covers a large amount of skin surface area, excessive amounts of calcium can be inactivated leading to systemic fluoride poisoning , hypocalcaemia, and hypomagnesaemia. Heart function is diminished in hypocalcaemia (reduced calcium levels in the blood), whereby the heartbeat becomes abnormal, and cardiac arrhythmia (ventricular fibrillation) can occur	15. 1) Neoprene or butyl rubber gloves changed upon chemical contact; safety glasses with secondary barrier (for example, hood sash) or goggles, lab coat; safety shoes; and protective apron. 2) Calcium Gluconate gel will be available in working area when working with HF.
16. Using hot plates.	16. Thermal burns and /or fire	16. 1) A sign warning of the hazard shall be placed in the area of any unattended thermal equipment. 2) Use thermally non-conducting tools (e.g. tongs). 3) Wear thermal protective gloves and safety glasses when handling or working near materials >50 degrees C. 4) Combustible material is to be removed from the area around any heated material or equipment.

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2.1 Training

- RDLYMFNP01, Analytical Laboratory Worker Job Code
- AFAL0001, Analytical Lab General Core Checklist, OJT
- AFAL0083, Analytical Lab Gas Proportional Counters, OJT Checklist.

2.2 Precautions and Limitations

2.2.1 Handle all chemicals per MCP-3635, "Chemical Hygiene Plan."

2.3 Waste Disposal Requirements

NOTE: *Accountable nuclear materials do not become waste until such time as the final package has been prepared, Safeguards and Security has completed its review of the attractiveness and program value and terminated Safeguards controls, and custody of the material has been transferred to a waste management organization. Accountable nuclear materials as defined in DOE Order 470.4-6, Change 1 are depleted uranium, enriched uranium, americium-241 and 243, curium, berkelium, californium-252, plutonium 238-242, lithium-6, uranium-233, normal uranium, neptunium-237, deuterium, tritium, and thorium.*

- 2.3.1 Manage all waste generated per this section and MCP-3365, "Waste Management at the Analytical Laboratory."
- 2.3.2 Project personnel shall ensure waste stream profiles have been established and satellite accumulation areas established as necessary with WGS for the type of waste being generated.
- 2.3.3 Waste that is generated and will result in a mixed or hazardous waste must be stored in an appropriate container in an established Satellite Accumulation Area (SAA) until the waste is ready for disposition by WGS. SAA training is required for all personnel generating hazardous waste.
- 2.3.4 If the waste generating process will result in a new radioactive, transuranic (TRU) or mixed waste, contact WGS before generating the waste to verify that the waste has an approved path forward and for proper disposal requirements. Generators of TRU or mixed TRU waste must follow the requirements of AL-5000-LI-003.

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- 2.3.5 Bag low-level radioactively contaminated waste and place in the radioactive waste laydown area and complete Form 435.42, "Radioactive Waste Inventory Sheet."
- 2.3.6 Contact WGS for proper disposal of any waste generated that has not been identified.

3. PREREQUISITES

3.1 Special Tools and Equipment

- 3.1.1 Obtain the following items:
- Centrifuge tubes, glass and polypropylene, 50-mL
 - Beakers, glass and plastic, assorted sizes
 - Bunsen burner
 - Centrifuge, with heads for 50-mL tubes
 - Stirring rods, glass
 - Filtration flask, 2-L, side-arm
 - Eichrom vacuum box, or equivalent filter manifold
 - Filters, glass microfiber, 2- μ m pore size, 24-mm diameter
 - Vacuum filtration units, individual, 0.45- μ m nitrocellulose filter, Fisher or equivalent
 - Heat lamp
 - Hot plate, stirring
 - Stir bars, magnetic
 - Pipetors, Eppendorf or equivalent, assorted sizes with tips
 - Sr Resin extraction Cartridges, 100–150 μ m particle size.
 - Environmental Express filter holder/funnels
 - Cartridge connector, Eichrom part number AC-121

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- Cap plug, tapered, polyethylene, for 50-mL centrifuge tubes, Caplugs part number T-12X or equivalent
- Volumetric flasks, Class A, various sizes
- Planchet, flat, stainless steel, 2 in. diameter, pretaped, Coy Labs #6509-240 or equivalent
- Balance, 160-g minimum capacity and ± 0.1 g sensitivity
- Glass drying plate, 6 to 8 in. diameter
- Petri dish, polystyrene, 100 \times 15 mm
- Filters, quantitative, paper, coarse, fast flow rate, 11-cm diameter
- Latex tubing, or equivalent
- Vacuum pump or plant vacuum.

NOTE: *Analytical Reagent Grade chemicals and ASTM Type II conductivity water or better must be used for preparation of all reagents, standards, and samples, unless otherwise specified.*

3.2 Reagents

NOTE: *Volumes other than those specified may be prepared provided that constituent ratios are maintained.*

3.2.1 Obtain/mix the following reagents:

- Ammonium carbonate, saturated solution. Fill a 1-L container approximately 3/4 full with water. Add solid ammonium carbonate ($[\text{NH}_4]_2\text{CO}_3$) while mixing until heavy solids remain in the bottom (thus ensuring saturation).
- Ethanol, absolute.
- Iron carrier. Dissolve 48.2 g of ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) in 1 L of water.
- Nitric acid (HNO_3), concentrated, Trace Metal Grade
- Nitric acid, 8 M. Prepare by adding 500 mL concentrated Trace Metal Grade HNO_3 to water and diluting to 1 L.

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- Sodium carbonate, powder.
- Sulfuric acid (H₂SO₄), concentrated, Trace Metal Grade.
- Ammonium hydroxide (NH₄OH), concentrated.

3.3 Standards

NOTE: *Volumes other than those described may be prepared as long as amount and aliquot volume ratios are maintained.*

3.3.1 Obtain/mix the following standards:

- 3.3.1.1 Strontium carrier, 5 mg/mL. Prepare by weighing 12.0766 g of anhydrous strontium nitrate [Sr(NO₃)₂] into a 1-L volumetric flask and recording the weight to the nearest 0.01 mg.
- 3.3.1.1.1 Fill the flask approximately 3/4 full with water and mix to dissolve the salt.
- 3.3.1.1.2 Add 10 mL of concentrated Trace Metal Grade HNO₃, mix, and allow the solution to stabilize at room temperature.
- 3.3.1.1.3 Dilute to volume with water and store in a polyethylene bottle.
- 3.3.1.1.4 If necessary to verify the final concentration, titrate the solution against standardized ethylenediaminetetraacetic acid (EDTA) using metal phthalein (phthalein purple) indicator.
- 3.3.1.1.5 Use within 1 year of preparation.
- 3.3.1.2 ⁹⁰Sr Stock Standard Solutions. Purchase two commercial solutions containing ⁹⁰Sr at 1-5 × 10⁶ Bq/g (e.g., NIST SRM 4234A and an equivalent NIST-traceable standard solution). Use within the manufacturer-specified shelf life.
- 3.3.1.3 Laboratory Control Sample (LCS) Solution. Prepare a solution containing 1-100 dps/mL ⁹⁰Sr in 5% HNO₃ from a stock standard independent of that used for instrument calibration. Replace or reverify the activity every 2 years.

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3.3.1.4 Efficiency Calibration Standard. Prepare a ^{90}Sr standard solution at 1-100 dps/mL from a NIST-traceable source in a 5% HNO_3 matrix. Replace or reverify the activity every 2 years.

3.4 Sample Handling

3.4.1 If the pH of aqueous samples as received is >2 , contact the customer for instructions.

3.4.2 If instructed by the customer, acidify the sample with Trace Metal Grade nitric acid to $\text{pH}<2$ and wait at least 16 hours before removing aliquots for analysis.

4. FACILITY CONDITIONS

4.1 The applicable ventilation and eyewash/shower station must be operational in order for this procedure to be worked.

5. INSTRUCTIONS

5.1 Routing Table

5.1.1 Perform the applicable section per the following table, THEN return to this step.

Preparing Solid Samples by Acid Leaching	Perform Subsection 5.2
Preparing Solutions of Solid Samples Resulting from Appropriate Dissolution Technique	Perform Subsection 5.3
Preparing High Level Aqueous Samples	Perform Subsection 5.4
Preparing Low-Level Aqueous Samples	Perform Subsection 5.5
Preparing Aqueous Samples Containing Organics	Perform Subsection 5.6
Separating Sr by Extraction Chromatography	Perform Subsection 5.7
Gas-Flow Proportional Counting	Perform Subsection 5.8
Analytical Batch Quality Control	Perform Subsection 5.9

5.2 Preparing Solid Samples by Acid Leaching

5.2.1 Determine aliquot size (0.1–20 g), based on sample activity, required detection limits, and sample availability.

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- 5.2.2 IF samples are debris,
THEN perform size reduction as necessary.
- 5.2.3 Weigh representative aliquots into appropriately-sized beakers, and record the aliquot size (dependent on sample activity) in the applicable Laboratory Notebook.
- 5.2.4 Prepare an LCS by pipeting 1.0 mL of LCS Solution into an appropriately-sized beaker.
- 5.2.5 Label an appropriately-sized beaker for use as the laboratory blank (LB).
- 5.2.6 If necessary, prepare a duplicate sample by weighing a second aliquot of a sample into a separate beaker and record the aliquot size in the applicable Laboratory Notebook.
- 5.2.7 Add 1.0 mL of Sr Carrier solution to each sample.
- 5.2.8 Record all quality control (QC) sample preparation and carrier solution additions in the applicable Laboratory Notebook.
- 5.2.9 Add 50–100 mL of 8 M HNO₃ to each sample.
- 5.2.10 Place a magnetic stir bar in the beaker.
- 5.2.11 Heat the beaker on a hot plate and gently boil the solution for 1–2 hours while stirring to reduce the volume to less than the desired final volume of prepared sample.
- 5.2.11.1 Ensure that the volume is sufficiently reduced so that the combined total of solution and acid rinses necessary to transfer the solution to the filtration apparatus in Step 5.2.14 will not exceed the desired final volume of prepared sample.
- 5.2.12 Remove the beaker from the hot plate and allow it to cool.
- 5.2.13 Label and connect a vacuum filtration unit equipped with a 0.45- μ m analytical filter to the vacuum source and turn on the vacuum.
- 5.2.14 Quantitatively transfer the solution with 8 M HNO₃ to the vacuum filtration unit.
- 5.2.15 Rinse the solids with 8 M HNO₃, using a volume that, when combined with the sample solution, will not exceed the desired final volume of prepared sample.

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- 5.2.16 Quantitatively transfer the filtrate into an appropriately-sized volumetric flask using 8 M HNO₃.
- 5.2.17 Ensure that the solution has cooled to room temperature.
- 5.2.18 Dilute the flask contents to volume with 8 M HNO₃.

5.3 Preparing Solutions of Solid Samples Resulting from Appropriate Dissolution Technique

- 5.3.1 IF preparing a solution resulting from another dissolution technique, THEN pipet a suitable aliquot of the solution into an appropriately-sized beaker.
- 5.3.1.1 IF the sample was NOT spiked with carrier prior to dissolution, THEN consult supervision for appropriate carrier spiking instructions.
- 5.3.1.2 IF the acid concentration is NOT 8 M, THEN add enough concentrated HNO₃ to adjust the acid concentration to ≥8 M and document the acid addition in the applicable Laboratory Notebook.
- 5.3.2 IF the cartridge load waste from AL-3500-LI-006 is being used, THEN add enough concentrated HNO₃ to adjust the acid concentration of the solution to ≥8 M.

5.4 Preparing High Level Aqueous Samples

NOTE: *This section is used to prepare samples containing >5 mR/hr beta. Subsection 5.6 is used if samples are expected to contain significant quantities of organic contaminants.*

- 5.4.1 Measure 1–5 mL aliquots of samples into appropriately-sized beakers.
- 5.4.2 Prepare a laboratory blank (LB) by adding 1–5 mL of water to an appropriately-sized beaker.
- 5.4.3 Prepare an LCS by adding 1.0 mL of LCS Solution and 1–5 mL of water to an appropriately-sized beaker.
- 5.4.4 Prepare a laboratory duplicate by measuring a second 1–5 mL aliquot of one of the field samples into an appropriately-sized beaker.

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- 5.4.5 Add concentrated HNO₃ in a 1:1 ratio to sample volume (e.g., 1 mL acid to 1 mL sample) to each beaker.
- 5.4.6 Add 1.0 mL of Sr carrier to each beaker.
- 5.4.7 Record all sample amounts and carrier volumes used in the applicable Laboratory Notebook.

5.5 Preparing Low-Level Aqueous Samples

NOTE: *This section is used to prepare samples containing <5 mR/hr beta. Subsection 5.6 is used if samples are expected to contain significant quantities of organic contaminants.*

- 5.5.1 Measure up to 1000 mL volumes of samples into appropriately-sized beakers.
- 5.5.2 Prepare a laboratory blank (LB) by adding up to 1000 mL of water to an appropriately-sized beaker.
- 5.5.3 Prepare an LCS by adding 1.0 mL of LCS Solution and up to 1000 mL of water to an appropriately-sized beaker.
- 5.5.4 Prepare a laboratory duplicate by measuring a second identical aliquot of one of the field samples into an appropriately-sized beaker.
- 5.5.5 Add 1 mL of Sr carrier to each beaker.
- 5.5.6 Record all sample amounts and carrier volumes used in the applicable Laboratory Notebook.
- 5.5.7 Add 1 mL of Fe carrier to each beaker.
- NOTE:** *Addition of Fe carrier aids in the collection of SrCO₃.*
- 5.5.8 Place the beakers on a hot plate and heat to boiling.

CAUTION

Adding Na₂CO₃ too rapidly will cause the sample to foam out of the beaker and cause analyte loss.

- 5.5.9 SLOWLY add small amounts of powdered Na₂CO₃ to each sample until the reactions cease.

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NOTE: *A heavy precipitate will form.*

- 5.5.10 Continue heating the samples for approximately 5 minutes to ensure completion of the reaction.
- 5.5.11 Remove the beakers from the hot plate, and allow the beaker to cool and the precipitate to settle.
- 5.5.12 Vacuum off and discard the supernate using a pipet tip attached to a side-arm filtration flask.
- 5.5.13 Transfer the precipitate using water washes to a 50-mL glass centrifuge tube.
- 5.5.14 Cap the tube and centrifuge the solution for approximately 3 minutes.
- 5.5.15 Discard the supernate.
- 5.5.16 Dissolve the precipitate in approximately 5 mL of concentrated HNO₃, heating with a Bunsen burner as necessary to aid dissolution.
- 5.5.17 Allow the sample to cool.
- 5.5.18 Add approximately 2 mL of water to give a final acid concentration of ≥ 8 M.

5.6 Preparing Aqueous Samples Containing Organics

NOTE: *This section is used when aqueous samples are expected to have significant concentrations of organic contaminants.*

- 5.6.1 Measure a suitable sample aliquot into an appropriately-sized beaker.
- 5.6.2 Prepare an LB by adding a similar amount of water to an appropriately-sized beaker.
- 5.6.3 Prepare an LCS by adding 1.0 mL of LCS Solution and a similar amount of water to an appropriately-sized beaker.
- 5.6.4 If necessary, prepare a laboratory duplicate by measuring a second identical aliquot of one of the field samples into an appropriately-sized beaker.
- 5.6.5 Add 1.0 mL of Sr carrier.

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5.6.6 Record all sample amounts and carrier volumes used in the applicable Laboratory Notebook.

5.6.7 Add 5–20 mL of concentrated H₂SO₄.

NOTE: *This forms the highly insoluble SrSO₄.*

5.6.8 Place the sample on a hot plate and evaporate to near dryness and heavy SO₃ fumes.

5.6.9 Add sufficient water to suspend the SrSO₄ precipitate, and quantitatively transfer it to a 50-mL glass centrifuge tube with water.

5.6.10 Centrifuge the sample for approximately 3 minutes.

5.6.11 Decant and discard the supernate.

5.6.12 Wash the precipitate with a small amount of water.

5.6.13 Centrifuge the sample for approximately 3 minutes.

5.6.14 Decant and discard the supernate.

5.6.15 Add 20 mL of 1:1 water/saturated Na₂CO₃, OR add a small amount (approximately 500 mg) of powdered Na₂CO₃ and approximately 20 mL of water to the precipitate.

NOTE: *SrSO₄ is converted to SrCO₃.*

5.6.16 Heat the centrifuge tube in a boiling water bath for approximately 30 minutes.

5.6.17 Centrifuge the sample for approximately 3 minutes.

5.6.18 Decant and discard the supernate.

5.6.19 Dissolve the precipitate in approximately 5 mL of concentrated HNO₃.

5.6.20 Add approximately 2 mL of water and swirl to mix.

5.7 Separating Sr by Extraction Chromatography

5.7.1 Equilibrate a Sr Resin extraction cartridge by passing approximately 10 mL of 8 M HNO₃ through the cartridge and collecting the effluent in a beaker.

5.7.2 Load the prepared sample on the cartridge.

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NOTE: *The Sr is retained on the cartridge. ^{90}Y and fission products pass through the cartridge and are collected in the effluent.*

5.7.3 Rinse the cartridge with approximately 15 mL of 8 M HNO_3 .

NOTE: *This rinse washes excess barium and any remaining fission products from the cartridge.*

5.7.4 Discard the effluent.

5.7.5 If performance of extra cleanup step is not needed, skip to Step 5.7.11.

5.7.6 Elute the Sr from the cartridge into a clean centrifuge tube with approximately 10 mL of water.

5.7.7 Add 5–8 drops of Fe carrier to centrifuge tube.

5.7.8 Add 5–10 mL of concentrated NH_4OH to precipitate $\text{Fe}(\text{OH})_3$.

NOTE: *All ^{90}Y , the decay product of ^{90}Sr , coprecipitates.*

5.7.9 Allow the precipitate to cool.

5.7.10 Filter the solution through a paper filter cone into a clean 50-ml plastic centrifuge tube containing 5-10 ml of saturated $(\text{NH}_4)_2\text{CO}_3$ solution.

5.7.10.1 Use 5–10 mL of water to facilitate quantitative transfer.

5.7.10.2 Wash the filter cone with 1–2 mL of water.

5.7.10.3 Proceed with Step 5.7.12.

5.7.11 Elute the Sr from the cartridge with approximately 10 ml of water into a clean centrifuge tube containing 5–10 mL of saturated $(\text{NH}_4)_2\text{CO}_3$ solution.

5.7.12 Allow approximately 5 minutes for complete precipitation.

5.7.13 Weigh a glass-fiber filter and record the tare weight to ± 0.1 mg in the applicable Laboratory Notebook.

5.7.14 Place the tared filter in a filter holder/funnel, mount on vacuum box, and apply vacuum.

5.7.15 Transfer the sample to the filtration apparatus and allow the sample solution to pass through the filter.

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- 5.7.16 Wash the centrifuge tube, filter holder/funnel, and filter paper with approximately 10-mL water to ensure quantitative transfer.
- 5.7.17 Wash the filter chimney and filter paper with a small amount (approximately 5 mL) of absolute ethanol.
- 5.7.18 Carefully remove the filter from the filtration apparatus and place it in a labeled glass drying plate.
- 5.7.19 Place the glass drying plate containing the filter under a heat lamp for 5-10 minutes to dry the filter.
- 5.7.20 Weigh the filter and record the filter gross weight to ± 0.1 mg in the applicable Laboratory Notebook.
- 5.7.21 Mount the filter onto a planchet for counting.
- 5.7.21.1 Use tweezers to center the filter in the planchet and gently tack the edges.
- 5.7.21.2 Place the planchet in a polystyrene petri dish for transport to the counting area.
- 5.7.22 Request beta radiation readings from an HPT to verify that each sample can be counted on the GFPC without contaminating the detector.
- 5.7.22.1 IF the reading obtained is high enough to contaminate the detector, THEN reprepare and separate the sample using a smaller aliquot size.

5.8 Gas-Flow Proportional Counting**5.8.1 Preparing Mass-Attenuated Efficiency Calibration Standards**

- 5.8.1.1 Label 12 small beakers No. 1 through No. 12.
- 5.8.1.2 Pipet 1.0 mL of the Calibration Efficiency Standard into each beaker.
- 5.8.1.3 Pipet from 0.20 to 1.30 mL of Sr Carrier into each successive beaker using 0.10-mL increments.
- 5.8.1.4 Add approximately 0.5 mL of Fe Carrier to each beaker and swirl to mix.

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- 5.8.1.5 Add approximately 10 mL of concentrated NH_4OH to each beaker and swirl to mix.
- 5.8.1.6 Filter the solution through a paper filter cone into a clean 50-mL plastic centrifuge tube containing 5–10 mL of saturated $(\text{NH}_4)_2\text{CO}_3$ solution.
- 5.8.1.6.1 Use 5–10 mL of water to facilitate quantitative transfer.
- 5.8.1.6.2 Wash the filter cone with 1–2 mL of water.
- 5.8.1.7 Allow approximately 5 minutes for complete precipitation.
- 5.8.1.8 Weigh a glass-fiber filter and record the tare weight to ± 0.1 mg in the applicable Laboratory Notebook.
- 5.8.1.9 Place the tared filter in a filter holder/funnel, mount on vacuum box, and apply vacuum.
- 5.8.1.10 Transfer the sample to the holder/funnel and allow the sample solution to pass through the filter.
- 5.8.1.11 Wash the centrifuge tube, filter chimney, and filter paper with approximately 10 mL water to ensure quantitative transfer.
- 5.8.1.12 Wash the filter chimney and filter paper with a small amount (approximately 5 mL) of absolute ethanol.
- 5.8.1.13 Carefully remove the filter from the holder/funnel and place it on a labeled glass drying plate.
- 5.8.1.14 Place the glass drying plate containing the filter under a heat lamp for 5–10 minutes to dry the filter.
- 5.8.1.15 Weigh the filter and record the filter gross weight to ± 0.1 mg in the applicable Laboratory Notebook.
- 5.8.1.16 Count the prepared standards for 30 minutes and prepare a mass-attenuated efficiency calibration.

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5.8.2 Counting Samples and Analyzing Data

- 5.8.2.1 Count the prepared samples.
- 5.8.2.2 Calculate and document the net weight of solid residue on the filter for each sample.
- 5.8.2.3 Evaluate the chemical yield per Subsection 5.9.4.
- 5.8.2.4 IF the gravimetric yield is >100%,
THEN consult with supervision to evaluate the cause of the problem and any appropriate corrective actions.
- 5.8.2.5 IF the net amount of solid residue on the filter exceeds the mass-attenuated calibration range of the GFPC,
THEN re-prepare and separate the sample using a smaller aliquot.

NOTE: *When the count rate exceeds 100,000 cpm, the likelihood of contaminating the detector and producing erroneous count data becomes unacceptably high.*

- 5.8.2.6 IF the count rate per sample exceeds 100,000 cpm,
THEN reprepare and reanalyze the sample using a smaller aliquot size.
- 5.8.2.7 IF the sample is NOT a positive detect
AND the sample MDA is >RDL,
THEN recount the sample using longer count time to achieve the RDL whenever possible.
- 5.8.2.8 Report all sample and QC sample activities with the associated measurement uncertainty (see Equation 1.1.6).
- 5.8.2.8.1 Report all negative activities as such.
- 5.8.2.9 IF the sum of the activity and the measurement uncertainty at $+3\sigma$ is a negative number,
THEN determine if the cause is systematic or random.
- 5.8.2.9.1 Correct systematic errors and discuss random errors in the data report narrative.

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5.9 Analytical Batch Quality Control

NOTE: *Documented project-specific quality control (QC) acceptance criteria may take precedence over criteria specified in this section.*

5.9.1 Laboratory Blanks

- 5.9.1.1 Process at least one laboratory blank with each analytical batch of samples.
- 5.9.1.2 Take the laboratory blank through all sample preparation, separation and counting steps along with the associated samples.
- 5.9.1.3 Count the laboratory blank for a time sufficient to meet the required detection limit (RDL).
- 5.9.1.4 Evaluate the laboratory blank results against the following acceptance criteria:
- Blank activity $< 2 \times$ blank uncertainty
 - Blank MDA $<$ RDL.
- 5.9.1.5 If result of the laboratory blank does not meet the above criteria, take appropriate corrective actions (e.g., recount, interferent cleanup, etc.) unless all associated sample results are $> 5 \times$ blank activity.
- 5.9.1.6 If the tracer recovery of the blank does not meet criteria specified in Step 5.9.1.4, re-prepare and reanalyze all associated samples if sufficient sample is available.

5.9.2 Laboratory Control Samples

- 5.9.2.1 Process at least one LCS with each analytical batch.
- 5.9.2.2 Take the LCS through all sample preparation, separation and counting steps along with the associated samples.
- 5.9.2.3 If the relative bias (see Equation 1.1.9) of the LCS is not within the range -0.25 to $+0.25$, re-prepare and reanalyze all associated samples if sufficient sample is available.

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5.9.3 Laboratory Duplicate

- 5.9.3.1 Process at least one sample in the analytical batch in duplicate.
- 5.9.3.2 Take the duplicate through all sample preparation, separation, and counting steps along with the associated samples.
- 5.9.3.3 If the normalized absolute difference (NAD, see Equation 1.1.10) between the original and duplicate sample is >3 , qualify the associated data in the data report.

5.9.4 Chemical Yield

- 5.9.4.1 Spike each sample with stable Sr carrier to determine chemical yield.
- 5.9.4.2 If the tracer percent yield for a sample is not between 40-110%, reprepare and reanalyze the sample whenever possible; otherwise, qualify the associated data in the data report.

6. Post-Performance Activities

- 6.1 Return dilutions/separations to origination point of parent solution.
- 6.2 Clean up and wipe down the area where the work was performed (hood, glovebox, or bench top).
- 6.3 After handling particularly hot samples, the background levels of the detector should be checked to check for contamination.
- 6.4 Dispose of waste materials into the proper waste stream per and Subsection 2.3 and MCP-3365.
- 6.5 Return samples to the originating point. If necessary, notify WGS personnel and/or the Material Balance Area (MBA) Custodian for a determination of the final disposition of samples.

7. Abnormal Operations

- 7.1 IF any abnormal conditions should arise during the performance of this procedure, THEN stop work, place the system in a stable configuration as applicable, and immediately notify the AL SS.

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8. RECORDS

Executed copies of Form 435.42, "Radioactive Waste Inventory Sheet."

NOTE: *LWP-1202, "Records Management," the INL Records Schedule Matrix, and associated record types list(s) provide current information on the retention, quality assurance, and/or destruction moratorium requirements for these records. Contact a Records Coordinator for assistance if needed.*

9. REFERENCES

Form 435.42, "Radioactive Waste Inventory Sheet"

MCP-3365, "Waste Management at the Analytical Laboratory"

MCP-3635, "Chemical Hygiene Plan"

10. APPENDIXES

Appendix A, Calculations

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Appendix A**Calculations****1. Calculations****1.1 Total Radiostrontium Activity****1.1.1 Observed Sample Activity**

$$A_{obs} = \frac{(cpm_s - cpm_{bkg})}{Y_t \times E \times S \times 60} \times DF$$

where

A_{obs} = observed sample activity (sum of radiostrontium activity plus ^{90}Y daughter activity), (dps/mL, dps/g, or dps/sample)

cpm_s = gross sample beta count rate (cpm)

cpm_{bkg} = background beta count rate (cpm)

DF = dilution factor

Y_t = fractional yield of carrier
(if fraction yield >100, use $Y_t = 1$)

E = detector efficiency (counts/decay)

S = sample size (kg for solids, L for liquids, 1 for filters)

60 = seconds/minute.

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1.1.2 Fractional ⁹⁰Y Daughter Ingrowth

$$FI = \frac{\lambda_Y \times A_{0_{Sr}} \times (e^{-(\lambda_{Sr} \times t)} - e^{-(\lambda_Y \times t)})}{(\lambda_Y - \lambda_{Sr}) \times A_{0_{Sr}}} = 1.0002494 \times (e^{-(0.0000027 \times t)} - e^{-(0.0108304 \times t)})$$

where

FI = fractional ingrowth of ⁹⁰Y at t hours after separation

λ_Y = decay constant for ⁹⁰Y = 0.0108304 hour⁻¹

λ_{Sr} = decay constant for ⁹⁰Sr = 0.0000027 hour⁻¹

t = time (hours) lapsed from separation.

1.1.3 Total Radiostrontium Activity

$$A_{Sr} = \frac{A_{obs}}{1 + FI}$$

where

A_{Sr} = total radiostrontium activity in sample (dpm/mL, dpm/g or dpm/sample)

A_{obs} = total observed activity from Equation 1.1.1

FI = fractional ⁹⁰Y ingrowth from Equation 0.

1.1.4 Activity Unit Conversions

$$\text{pCi} = \text{dpm} \times 0.45045 \text{ pCi/dpm}$$

$$\text{pCi} = \text{dps} \times 27.027 \text{ pCi/dps}$$

$$\text{dps} = \text{dpm} \times 0.01337 \text{ dps/dpm}$$

$$\text{Bq} = \text{dps}.$$

1.1.5 Decay Correction

$$A = A_o \times e^{-\lambda t}$$

where

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A = activity

A_o = original activity

e = natural log

λ = $0.693/(t_{1/2})$

t = elapsed time from A_o to present (must be in same units as $t_{1/2}$)

$t_{1/2}$ = half-life of the isotope (must be in same units as t).

1.1.6 Total Propagated Uncertainty (TPU)

$$TPU = \sqrt{\sigma_a^2 + \sigma_b^2 + \sigma_c^2 + \sigma_d^2 + \sigma_e^2}$$

where

TPU = total propagated uncertainty (relative)

σ_a = counting (relative) uncertainty

σ_b = uncertainty (relative) of carrier concentration

σ_c = uncertainty (relative) of detector efficiency

σ_d = uncertainty (relative) of sample aliquot measurement (mass or volume)

σ_e = uncertainty (relative) of carrier amount added.

The absolute TPU is calculated by multiplying the relative TPU (expressed as a fraction) times the sample activity.

NOTE: *This equation propagates uncertainty for a result using the five most significant error factors in the measurement process. Smaller error factors, such as counting time uncertainty or branching ratio uncertainty, do not contribute significantly to the TPU and are therefore omitted from the equation.*

1.1.7 Counting Uncertainty

$$\sigma_a = \frac{\sqrt{C_g - C_{bkg}}}{C_n}$$

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where

σ_a = counting uncertainty (relative)

C_g = gross sample counts

C_{bkg} = background counts (for same count time as the sample)

C_n = net sample counts (gross minus background).

1.1.8 Carrier Yield

$$Y = \frac{W_g - W_t}{C \times V_c} = \frac{W_n}{W_a}$$

where

Y = yield

W_g = gross mass of filter and residue (mg)

W_t = tare mass of filter (mg)

C = concentration of Sr carrier (mg Sr/mL)

V_c = volume of Sr carrier added (mL)

W_n = net mass of precipitate on filter after separation (mg)

W_a = mass of carrier added (mg).

If tracer percent recovery is needed, multiply Y times 100.

1.1.9 Relative Bias (RB)

$$RB = \frac{A_m - A_k}{A_k}$$

where

RB = relative bias

A_m = measured activity of the LCS, decay-corrected to time of LCS standard solution preparation

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A_k = known activity of the LCS at time of LCS standard solution preparation.

1.1.10 Normalized Absolute Difference

$$NAD = \frac{|S - D|}{\sqrt{U_s^2 + U_D^2}}$$

where

S = sample result

D = duplicate result

U_s = sample uncertainty

U_D = duplicate uncertainty.

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1.1.11 Minimum Detectable Activity (MDA)

$$MDA = \frac{3 + 4.65\sqrt{C_{bkg} \times T_C \times DF}}{T_C \times E \times Y \times S \times 60 \text{ sec/min}}$$

where

MDA = minimum detectable activity (dps/g, dps/L, dps/smpl)

C_{bkg} = background count rate (cpm)

T_C = sample count time (minutes)

E = detector efficiency (cpm/dpm)

Y = fractional chemical yield (if fractional yield >1.00, use Y=1)

S = sample size (g, L, or 1 for filters)

DF = dilution factor.