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ANION EXCHANGE SEPARATION OF PLUTONIUM FROM VARIOUS SOLUTIONS STORED AT PFP

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E, S, Q, D or N/A (see WHC-CM-3-5, Sec.12.7)	1. Approval 2. Release 3. Information 4. Review 5. Post-Review 6. Dist. (Receipt Acknow. Required)	1. Approved 2. Approved w/comment 3. Disapproved w/comment 4. Reviewed no/comment 5. Reviewed w/comment 6. Receipt acknowledged

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(G) Reason	(H) Disp.	(J) Name	(K) Signature	(L) Date	(M) MSIN	(J) Name	(K) Signature	(L) Date	(M) MSIN	(G) Reason	(H) Disp.
1	1	Cog.Eng. S.A. Jones	[signature]	2-20-96	T5-12	Rad Con - SC Snyder	[signature]	2/26	T5-53	1	1
1	1	Cog. Mgr. L.S. Sutter	[signature]	2/20/96	T5-12	Crit. Safety - [illegible]	[signature]	2-26-96	T5-53	1	1
1	1	QA D.R. Groth	[signature]	2/23/96		JRG	[signature]	2/23/96		1	1
1	1	Safety S.C. Nunn	[signature]	2-23-96	T5-11						
1	1	Env. DJ McBride	[signature]	2-26-96	T5-54						
1	1	G. B. Chronister	[signature]	2/20/96							
1	1	J. F. Durnil									

18.	19.	20.	21. DOE APPROVAL (if required)
[signature] 2/23/96 Signature of EDT Originator / Date	Authorized Representative for Receiving Organization / Date	Caroline Sutter [signature] 2/26/96 Cognizant Manager / Date	Ctrl. No. [] Approved [] Approved w/comments [] Disapproved w/comments

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Anion Exchange Separation of Plutonium from Various Solutions Stored at PFP

S. A. Jones
Westinghouse Hanford Company, Richland, WA 99352
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Key Words: Plutonium, solution, ion exchange, anion exchange, separation

Abstract: Anion exchange has been demonstrated to effectively separate plutonium from other metals in solution. Specific PFP solutions types will be tested to determine if they are suitable candidates for anion exchange.

DATE:
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HANFORD RELEASE
ID:
FEB 26 1996

Release Approval — Date 2/26/96 — Release Stamp

Approved for Public Release

TABLE OF CONTENTS

ANION EXCHANGE SEPARATION OF PLUTONIUM FROM VARIOUS SOLUTIONS STORED AT PFP

S. A. Jones

1.0 TEST ITEM IDENTIFICATION

Reillex HPQ[a], a macroporous polyvinylpyridine resin, will be used to separate plutonium from other materials in solution using nitrate anion exchange. The solutions to be tested include, but are not limited to, Plutonium Reclamation Facility filtrate solution and analytical laboratory concentrator solutions. These solutions contain metals and anions which may interfere with later efforts to calcine the plutonium and would certainly reduce the concentration of plutonium in the calcined solid.

2.0 GENERAL DESCRIPTION

Reillex HPQ has been demonstrated[1] to provide rapid Pu(IV) sorption kinetics from nitric acid solutions and to be strongly resistant to chemical attack by nitric acid. This resin has been shown to be more resistant to the effects of *in situ* alpha particle irradiation[2] compared to other resins tested.

There are solutions in the Plutonium Finishing Plant (PFP) that were to have been processed in the Plutonium Reclamation Facility (PRF). The PRF will not be operated and for safe storage of the plutonium these solutions must be converted to a solid form. Nitrate anion exchange will be tested as a method of separating the potentially reactive components of these solutions from the plutonium. This testing will develop the separation parameters required for large-scale processing of these and similar solutions.

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[a]Reillex HPQ is a registered trademark of Reilly Industries, Inc., Indianapolis, Indiana.

3.0 CRITICALITY SAFETY LIMITS

No special test conditions are required. Liquid testing will be performed in a glovebox with a 250 g limit and a 10 L/container maximum volume with no maximum total volume.

4.0 INSTRUMENTS AND CALIBRATION

Pump volumes will be confirmed using graduated cylinders. Analyses performed by the PFP Analytical Laboratory will be done to their quality assurance requirements. The liquid scintillation counter will be calibrated and operated per the manufacturers operating manual.

5.0 FACILITIES, EQUIPMENT, AND MATERIALS

Experimental work will be performed in Glovebox 179-6 in Room 179 and the hoods in Room 187 of the 234-5Z Building. Nonradioactive chemical preparations will be performed in Room 191. Product from these tests will be stored in either Glovebox 179-6 or 179-9.

The following materials and equipment are required:

- Liquid chromatography column
- Reillex HPQ anion exchange resin
- Sample vials
- Deionized water
- Test solutions
- Ultima Gold[b] liquid scintillation counter cocktail
- Nitric acid solutions (0.5 M, 1.5 M and 15.7 M)

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[b]Ultima Gold is a registered trademark of Packard Instrument Company, Inc., Downers Grove, Illinois

6.0 SAFETY

A Job Hazard Analysis Checksheet is attached at the end of this test plan.

Criticality and radiological safety considerations are largely those attendant to glovebox operations with plutonium materials. Safety will be ensured by rigid adherence to Criticality Prevention Specifications CPS-L-114-00010[3], -00020[4], -00030[5], -00040[6], and -01900[7], and Plutonium Finishing Plant Radiation Work Permits.

The chemicals to be used in these experiments and their corresponding HEHF-assigned Material Safety Data Sheet (MSDS) numbers are:

Chemical	HEHF MSDS #
Nitric Acid	1384
Reillex HPQ™ Ion Exchange Resin	35852
Ultima Gold™ Scintillation Cocktail	21700
Water	1848

Additional safety items are covered in the PPSL operating procedures.

7.0 MAINTENANCE AND FAILURES

No special maintenance is required. Failures are not anticipated; however, failures can be corrected, and testing, if interupted, can be resumed from any point without adverse consequences.

8.0 TEST DATA

Data to obtained during these experiments are plutonium concentrations in the feed, raffinate, and eluant streams; acid concentrations in the feed, wash raffinate, and eluant streams; other metal cations and their concentrations in the feed, raffinate, and eluant streams; feed, wash, and eluant solution volumes; and, ion exchange volume and weight.

9.0 PERSONNEL

The PPSL staff will perform all test operations. Operation of the system requires only one person except when items must be sealed into or out of the glovebox. Security regulations require additional personnel in the room; however, the additional personnel will not necessarily be working with this equipment.

10.0 WITNESSES

Witnesses are not required for these tests.

11.0 PROCEDURE

Items required for performing each run are listed below. Column and bottle sizes, tubing lengths, and solution volumes may be adjusted by operating personnel, as needed, during set-up and operation.

1. One ion exchange column with resin already loaded (150 mL).
2. One metering pump for flows between 0 and 10 mL/min.
3. Three sections of 1/16-inch tubing, one of which has a filter attached at one end. Tubing sections need to be at least two feet long.

4. Feed, wash, eluant, and product bottles: 1-L polybottles.
5. One bottle of test solution, as furnished.
6. One bottle of wash solution: 1-L polybottle with at least 150 mL of 7M nitric acid.
7. One eluant bottle: 1-L polybottle with 1 L of 0.5M or 0.7M nitric acid, depending on run conditions.
8. One product bottle: 1-L polybottle.
9. One raffinate bottle: 4-L polybottle.
10. Sample bottles: 25-mL glass or poly, as needed.
11. Pink-bottomed SNM labels (for H/X>20), as needed.

A. Column Set-Up

1. Record the plutonium content on the glovebox inventory before the solution enters the glovebox and be certain that the Criticality Prevention Posting will not be violated by the material entry. Load test solution into the glovebox. Label the test solution bottle with a pink-bottomed SNM label and place an identical label on the glovebox exterior near where the bottle will be stored.

2. Check to be certain the intended ion exchange column is connected to the feed pump.

3. Check the acid concentration in the feed supply. If the acid concentration is outside the range of 6.5-7.5 M, adjust the acidity by the applicable equation below:

to RAISE the acid concentration to 7.0 M using 15.7 M HNO_3:

$$V_a = (7-[H]_o)*V_o/8.7$$

to LOWER the acid concentration to 7.0 M using 1.5 M HNO_3:

$$V_a = ([H]_o-7)*V_o/5.5$$

where V_a is the amount of fresh nitric acid to be added (liters), V_o is the intended amount of feed to be adjusted (liters), and $[H]_o$ is the original acid concentration in the test solution. Make certain the combined test solution and fresh acid volume (V_o + V_a) will not exceed the volume of the feed bottle.

4. Add the correct amounts of test solution and the proper fresh acid solution to the feed bottle. Mix the feed solution. Remove a 10-15 mL sample and place it in a labeled sample bottle. Proceed with the remainder of this procedure; fill out a sample analysis request form (for acid concentration only) and submit the sample to the PFP Analytical Laboratory as time permits.

5. Fill out and place a pink-bottomed SNM label on the glovebox wall or window nearest the feed bottle to indicate the amount and identity of the solution. Correct the elemental weight entries on the original test solution bottle labels for its decreased content or fill out replacement labels.

6. Place the filtered end of the feed line to the pump into the feed bottle.

7. Fill out and place another pink-bottomed SNM label on the glovebox wall or window nearest the raffinate bottle to indicate its identity and contents after the feed passes through the column. Record this bottle's presence on the glovebox inventory.

B. Column Operation: Extraction and Washing

Sampling frequency may be adjusted as testing proceeds. The values listed in the following steps are intended as initial guidlines.

1. Turn on the feed pump. Observe the liquid rising in the column to be certain the pump is working. Record the feed flow rate in the laboratory notebook.

2. Periodically return to observe the color rising in the column and sample the raffinate from the column as in steps a-d, below. The feed pump may be shut off if the laboratory must be evacuated; however, leaving plutonium loaded onto the resin is generally to be avoided.

   a. Label a sample vial and record its number in the lab notebook.

b. Move the raffinate solution tube from the raffinate bottle into the sample vial. Leave the tube in the sample vial until the necessary 10-15 mL are collected.
c. When enough sample has been collected, place the raffinate tube back into the raffinate bottle. Screw the cap onto the sample vial.
d. Fill out a sample analysis request form to have the samples analyzed for plutonium and acid concentrations. Alternatively, the sample may be retained within PPSL for plutonium analysis in the Liquid Scintillation Counter.

3. Refill the feed bottle, as needed, from the test solution bottle and adjust the acid concentration as in steps A3 and A4, above. Correct all SNM labels to indicate the new contents or replace them, as needed.

4. When the green color of the solution reaches the top of the column (or when the feed solution is gone), sample the column effluent again per steps 2a-2d, then shut off the feed pump.

5. Turn off the feed pump and move the feed line and filter from the feed bottle into the wash solution bottle.

6. Restart the feed pump. Run 150 mL of wash solution through the column.

7. Remove a 10-15-mL sample of the composite raffinate/wash solution and place it in a labeled sample bottle. Fill out a sample analysis request form to have the sample analyzed for plutonium, acid, and all other metal cations concentrations.

8. Shut off the feed pump and prepare for column elution.

9. Move the raffinate/wash solution collection bottle and appropriate labels to the Satellite Accumulation Area. Record the entry of the bottle on the glovebox inventory.

C. Column Operation: Elution and Product Storage

1. Rearrange the tubing to move the pump outlet line to the top of the column and the column outlet line to the bottom. Place the column outlet line into the product bottle.

2. Fill out and attach a pink-bottomed SNM label for the product bottle. Attach it to the glovebox wall or window nearest the product bottle. Record this bottle's presence on the glovebox inventory and be certain that the Criticality Prevention Posting will not be violated.

3. Place the pump's inlet line and filter into the eluant bottle and turn on the pump.

4. Periodically observe the column operation and progress of the elution in removing the plutonium from the resin.

5. Sample the product solution every 15 minutes as in steps B2a-d, above, but with the product bottle substituted for the feed bottle.

6. When the green-colored band of concentrated plutonium approaches the bottom of the column, sample every 2 minutes as in steps B2a-d, above.

7. After the plutonium appears to be completely removed from the column, wait 5 minutes, then shut the pump off. Remove a 10-15-mL sample and place it in a labeled sample bottle. Fill out a sample analysis request form to have the sample analyzed for plutonium, acid, and all other metal cations concentrations.

8. Seal all samples out of the glovebox.

9. Cap the product bottle. Determine where the bottle will be stored to await calcination. Move the pink-bottomed SNM label to the glovebox wall or window nearest the bottle storage location.

10. If necessary, seal the bottle out of Glovebox 179-6. Record the entry of the bottle onto the Glovebox 9 inventory before placing it

inside and be certain that the Criticality Safety Posting will not be violated. Remove the bottle listing from the Glovebox 179-6 inventory.

12.0 DISPOSITION OF TEST ITEM

Ion exchange resin will be disposed of as contaminated waste after testing. Solid Waste Operations and Environmental Engineerting will assist in waste designation and disposal.

Pumps and other mechanical equipment will be saved and reused, if possible. If necessary, these items will be designated as waste and managed accordingly.

Raffinate and wash solutions will be sent to Tank Farms via the building radioactive drain system. Environmental Engineering and Process Engineering will assist in the disposal of these wastes.

Product solutions will be calcined in the direct denitration vertical calciner being demonstated in Room 188 of the PPSL or otherwise converted to a stablized solid material.

13.0 REPORTS

Status will be reported in Plutonium Process Support Laboratories Weekly Reports. Following the completion of testing, a test report will be issued as a supporting document.

14.0 DATA SHEETS

Data from these tests will be obtained in the form of computer printouts, instrument readings, and measurements. This data will be recorded in a laboratory notebook and in a computer spreadsheet for data analysis.

15.0 REFERENCES

1. S. F. Marsh, Evaluation of a New, Macroporous Polyvinylpyridine Resin for Processing Plutonium Using Nitrate Anion Exchange, Los Alamos National Laboratory report LA-11490 (April 1989).

2. S. F. Marsh, The Effects of *in situ* Alpha-Particle Irradiations on Six Strong-Base Anion Exchange Resins, Los Alamos National Laboratory report LA-12055 (April 1991).

3. Criticality Prevention Specification, CPS-L-114-00010, Westinghouse Hanford Company, Richland, Washington.

4. Criticality Prevention Specification, CPS-L-114-00020, Westinghouse Hanford Company, Richland, Washington.

5. Criticality Prevention Specification, CPS-L-114-00030, Westinghouse Hanford Company, Richland, Washington.

6. Criticality Prevention Specification, CPS-L-114-00040, Westinghouse Hanford Company, Richland, Washington.

7. Criticality Prevention Specification, CPS-L-114-01900, Westinghouse Hanford Company, Richland, Washington.

HANFORD JOB HAZARD ANALYSIS CHECKLIST

Prepared By S. A. Jones | Date 01/26/96 | Area 200W | Bldg. 234-5Z

Scope/Description: This checklist applies to anion exchange testing in the Plutonium Process Support Laboratories as described in test plan WHC-SD-CP-TP-089.

☒ New
☐ Revised

Emergency Contact Person(s):

Primary: PFP Building Emergency Director

Secondary: PFP Central Alarm Station

Emergency Radio/Phone Number: PAX 227 859360

JHA Number (not required):

Specific Work Location(s): 234-5Z Building, Room 179, Gloveboxes 179-6 and 179-9.

KNOWN OR POTENTIAL HAZARDS

	Yes	No	✓	●	Reference
1. Radiation Area Work	X		✓	●	RWP Z-012
2. Hazardous Waste Operations	X		✓	●	CM-4-3/W-12
3. Confined Space Entry		X	✓	●	
4. Cutting/Welding		X		●	
5. Roof Work		X			
6. Fall Hazards (> = 10')		X			
7. Excavation/Trenching		X		●	
8. Asbestos Inspection Report		X		●	
9. Hazardous Materials	X		✓		CM-5-8 / 7.1
10. Respiratory Hazards	X		✓		RWP Z-005
11. Electrical Hazards		X	✓		
12. Lock and Tag		X	✓	●	
13. Scaffolding		X			
14. Aerial Lifts		X	✓		
15. Asbestos Removal		X	✓	●	
16. Other (see JHA Sht. 2):		X			

✓ = Formal training required.
● = Items than require a permit/form/report.

Other Hazards	Yes	No	Control Measures
1. Temperature Extremes		X	
2. Noise		X	
3. Poor Lighting		X	
4. Animals/Insects		X	
5. Process Chemicals/Steam		X	
6. Dust		X	
7. Flammable/Combustible Materials		X	
8. Ladders		X	
9. Wet/Slippery Floors		X	
10. Uneven Terrain		X	
11. Open Excavations/Trenches		X	
12. Adjacent Water Hazard		X	
13. Vehicle Traffic		X	
14. Heavy Equipment		X	
15. Rigging Operation		X	
16. Manual Lifting		X	
17. Power Tools		X	
18. Pinch Points		X	
19. Falling Objects		X	
20. Sharp Objects		X	
21. Overhead Obstructions		X	
22. Site Control (Signs/Barricades)		X	
23. Remote Work Area		X	
24. Other (see JHA Sht. 2):		X	

MINIMUM DRESS REQUIREMENTS: Per applicable RWP

APPROVALS

Does further evaluation of the job steps, associated hazards, or safety measures need to be performed? ☐ Yes ☒ No
If Yes, continue job hazard analysis on the following pages.

Supervisor, Person in Charge (Signature) S. A. Jones [signature]

Industrial Safety/Hygiene (Signature) A. G. Pines [signature] 2-23-96

BC-3000-717 (01/95)

DISTRIBUTION SHEET

To	From	Page 1 of 1
PFP Process Engineering 15530	Plutonium Process Support Laboratories	Date February 20, 1996
Project Title/Work Order		EDT No. 609889
Anion Exchange Separation of Plutonium from Various Solutions Stored at PFP		ECN No. NA

Name	MSIN	Text With All Attach.	Text Only	Attach./ Appendix Only	*EDT/ECN Only
G. S. Barney	T5-12	X			
T. D. Cooper	T5-12	X			
J. F. Durnil	T5-55	X			
F. D. Fisher	T5-12	X			
M. W. Gibson	T5-55	X			
D. R. Groth	T4-15	X			
S. A. Jones	T5-12	X			
D. J. McBride	T5-54	X			
L. T. Nirider	T5-53	X			
S. E. Nunn	T5-11	X			
A. G. Pines	T5-11	X			
R. D. Redekopp	T5-15	X			
S. C. Snyder	T5-03	X			
C. S. Sutter	T5-12	X			
E. C. Vogt	T5-50	X			
Central Files	A3-88	X			
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