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RELEASE AUTHORIZATION

Document Number: WHC-SD-CP-TC-033, REV 0

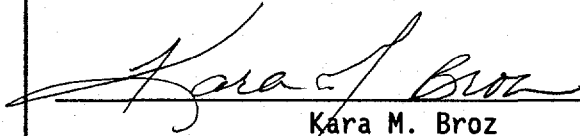
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This document was reviewed following the procedures described in WHC-CM-3-4 and is:

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WHC Information Release Administration Specialist:


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May 17, 1995

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SUPPORTING DOCUMENT

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2. Title

Test Procedure for Anion Exchange Testing with Argonne 10-L Solutions

3. Number

WHC-SD-CP-TC-033

4. Rev No.

0

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Plutonium, Ion Exchange, Anion Exchange, 10-L Solutions, Plutonium Nitrate

6. Author

Name: J. A. Compton

James A Compton 5-16-95
Signature

Organization/Charge Code 15F00/K6032

7. Abstract

Four anion exchange resins will be tested to confirm that they will sorb and release plutonium from/to the appropriate solutions in the presence of other cations. Certain cations need to be removed from the test solutions to minimize adverse behavior in other processing equipment. The ion exchange resins will be tested using old laboratory solutions from Argonne National Laboratory. The results will be compared to results from other similar processes for application to all plutonium solutions stored in the Plutonium Finishing Plant.

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DATE MAY 17 1995
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TEST PROCEDURE FOR ANION EXCHANGE TESTING WITH ARGONNE 10-L SOLUTIONS**1. TEST ITEM IDENTIFICATION**

Four separate anion exchange resins will be tested for their abilities to separate chemicals from plutonium solutions when those other chemicals might affect other processing equipment adversely. The resins are (1) Lewatit MP 500 SK¹, (2) Reillex HPQ², (3) Dowex MSA-1³, and (4) Amberlite IRA-900C⁴. Portions of the Argonne 10-L Solutions will be used as the plutonium source for these tests.

2. GENERAL DESCRIPTION

This test procedure is a companion document to the test plan, *Test Plan for Anion Exchange Testing with Argonne 10-L Solutions*, WHC-SD-CP-TP-085, Rev. 0. The test plan explains that the Argonne 10-L Solutions to be used in these tests are a portion of the solutions stored in the Plutonium Finishing Plant (PFP) awaiting conversion to a stable, storable oxide. Conversion to the oxide will occur in a direct denitration vertical calciner; however, some chemicals in the feed solutions may cause operating problems in the calciner. Use of ion exchange allows plutonium(IV) nitrate to be kept for calciner feed while other chemicals are discarded as raffinate. Calculations based on the test data will allow comparisons of these 4 anion exchange resins with other types of chromatography (Reference 1).

The tests will be performed in continuously operating ion exchange columns by Plutonium Process Support Laboratories (PPSL) staff using discrete batches of feed from different blends of the 10-L Solutions.

3. TEST CONDITION LIMITS

These tests are limited to normal radioactive laboratory room conditions. Required evacuations from the laboratory room requires that the test(s) in progress be stopped with resumption allowed after normal conditions are restored.

¹Lewatit MP500 SK is a registered trademark of Mobay Corporation, Pittsburgh, Pennsylvania.

²Reillex HPQ is a registered trademark of Reilly Industries, Inc., Indianapolis, Indiana.

³Dowex MSA-1 is a registered trademark of Dow Chemical Company, Midland, Michigan.

⁴Amberlite IRA-900C is a registered trademark of Rohm and Haas Corporation, Philadelphia, Pennsylvania.

The 10-L Solutions must be blended into categories chosen by PFP Process Engineering and delivered to the PPSL's Room 179 prior to starting testing.

4. INSTRUMENTS AND CALIBRATIONS

The pumps used in these tests are pre-calibrated by the manufacturer. Further calibration is not needed. No other equipment calibration is required. Feed volumes will be confirmed using graduated cylinders; the manufacturer's calibration marks are sufficiently accurate for test purposes.

Analyses performed by the PFP Analytical Laboratory will be done to their Quality Assurance Program requirements.

If used, the Liquid Scintillation Counter will be calibrated and operated per the manufacturer's operating manual.

5. FACILITIES, EQUIPMENT AND MATERIALS

Four separate macroreticular anion exchange resins will be tested. The resins are (1) Lewatit MP 500 SK™, (2) Reillex HPQ™, (3) Dowex MSA-1™, and (4) Amberlite IRA-900C™. Portions of the Argonne 10-L Solutions will be used as the plutonium source for these tests.

The experimental work will be performed in Glovebox 179-6 in Room 179 and the hoods in Room 187 of the 234-5 Building. Non-radioactive chemical make-up will be performed in Room 191. Product from the tests will be stored in either Glovebox 179-6 or 179-9, depending on space availability and criticality safety. The work will be performed in normal room air at room temperature.

The following equipment is required:

- ▷ Ion exchange columns (4)
- ▷ Anion exchange resins (4)
- ▷ Glass sample vials
- ▷ Deionized water
- ▷ Pu(NO₃)₄ storage solutions ("10-L Solutions")
- ▷ Ultima Gold⁵ liquid scintillation counter cocktail
- ▷ Nitric acid solutions (1.5 M and 15.7 M)
- ▷ Sodium hydroxide solution (50 wt. percent)

6. SAFETY

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

⁵Ultima Gold is a registered trademark of Packard Instrument Company, Inc., Downer's Grove, Illinois.

6.1 CRITICALITY - The small amounts of plutonium in use during these experiments minimizes the possibility of a criticality. Administrative controls and inspections reduce the possibility even further.

Glovebox 179-6 is approved for fissile material handling under Criticality Prevention Specification CPS-L-114-00020, "Uniform Laboratory Specifications" (Reference 2). This glovebox is currently approved for Set D from that set of uniform specifications, which is for a single unit mass of 250 grams of plutonium with a maximum single container volume of 10.0 liters. This CPS Posting and a plutonium inventory are posted at one end for reference to approved plutonium handling conditions inside the glovebox. The glovebox receives monthly inspection by PPSL personnel and quarterly inspection by the PFP Criticality Safety Representative to assure compliance with the posted limits. The personnel performing the work are familiar with the posting limits and the methods used to stay within them.

Product solution storage may also require the use of Glovebox 179-9. This glovebox is also approved for fissile material handling under Set D of the uniform specifications.

6.2 RADIOLOGICAL - The small amounts of plutonium used in these experiments reduce the potential radiation dose to working personnel. These dose rates will be reduced further by allowing most of the radioactive work to proceed unattended except for periodic surveillance. All work and protective clothing will conform to the applicable Radiation Work Permit(s) (RWPs). Updated copies of the RWPs are posted outside the laboratory rooms for reference.

Radiation Control Technicians (RCTs) routinely measure and post the radiation dose rates for the glovebox. The RCTs also check the gloves and room for leakage of contamination. The RCTs are also required to be present during any movement of material in or out of the glovebox.

All personnel entering the PPSL radioactive laboratory area are required to wear dosimetry for measuring gamma exposure. The PPSL personnel working on the tests are also required to wear a Hanford Combination Neutron Dosimeter. Personnel working in the gloveboxes are required to wear a finger ring on each hand for measuring the dose to the extremities. Anyone receiving greater than 350 millirem in a calendar year is also required to wear a self-indicating "pencil" dosimeter issued by the RCTs. Any additional required dosimetry will be specified in the RWPs.

6.3 CHEMICAL - Heat will be generated by the reaction of 7M nitric acid with 19.1M (50 wt.%) sodium hydroxide (13.5M [50 wt.%] potassium hydroxide solution may be substituted for the sodium hydroxide) as the raffinate and wash solutions are neutralized. The heat generation rate and, therefore, the solution temperature, will be minimized by neutralizing as the raffinate and wash solutions enter the raffinate collection vessel. These flow rates are identical to the feed flow rate into the column and are positively controlled to a few mL/minute by the feed pump. Where possible, this vessel will be a stainless steel beaker to promote heat transfer from the solution to the

surrounding air; polyethylene will be used only if necessary. Boiling temperatures will not be reached and access to the hot vessels is not required. The beaker will not be handled without tongs or gloves suitable for high temperatures (230°C) until cooling for at least two hours.

The possibility of a minor chemical hazard exists due to the use of an organic anion exchange resin with a concentrated nitric acid solution. The resin is composed of long-chain elements and/or ring structures that are normally resistant to attack by oxidizers such as nitric acid. Radiolysis and gradual chemical attack, however, can lead to resin degradation and the formation of smaller organic compounds that are more easily oxidized by concentrated nitric acid. The oxidation reaction can generate heat and gases, each of which would increase the pressure inside a sealed vessel.

The oxidation of small organic compounds is minimized in these experiments by the short test duration and the use of small amounts of fresh resin. The short test durations minimize the amount of radiolytic or chemical degradation that can occur, thereby minimizing the amounts of reactive chemicals formed. The use of small amounts of resin further reduces the amount of reactive chemicals that might be formed and, therefore, the amounts of heat and gases that can be generated. The use of fresh resin minimizes the amount of reactive chemicals that might be present at the beginning of the experiments. Finally, the ion exchange columns where the resin and concentrated nitric acid will be mixed are not sealed; thus, pressure would be released as it is generated.

Nitric and hydrochloric acids are also corrosive chemicals on skin and protective clothing is required for their handling. Acid-resistant gloves, coveralls, and goggles or face shield are the minimum protective apparel. Handling of any chemicals within Contamination Areas would be performed under the applicable Radiation Work Permits.

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

<u>Chemical</u>	<u>HEHF MSDS #</u>
Amberlite IRA-9000™ Ion Exchange Resin	25837
Dowex MSA-1™ Ion Exchange Resin	19902
Lewatit MP 500 SK™ Ion Exchange Resin	25894
Nitric Acid	1384
Potassium Hydroxide	1442
Reillex HPQ™ Ion Exchange Resin	35852
Sodium Hydroxide	1105
Ultima Gold™ Scintillation Cocktail	21700
Water	1848

Copies of these MSDSs are available for reference in Corridor 8 of the PPSL laboratory area. The appropriate MSDSs will be reviewed before beginning work with these chemicals.

6.4 THERMAL - The only thermal hazard occurs in the raffinate collection bottle while the acidic raffinate and wash solutions are being neutralized. This exothermic chemical reaction will be performed slowly enough to keep surface temperatures below boiling temperatures. Gloves required for handling hot containers are available; however, handling should not be necessary.

6.5 ELECTRICAL - The experiments require only small equipment with no electrical parts or only 120-VAC motors. Connections are made to power through standard plugs into standard outlets. All work involving the electrically powered equipment will occur in gloveboxes, where three sets of non-conductive gloves are required, with or without using electrical apparatus.

6.6 MECHANICAL - The experiments use small, lightweight equipment. Hazards of equipment falling over are minimal, especially given the minimal time required working directly with equipment. There are no exposed moving parts on any equipment.

6.7 UNATTENDED OPERATION - The ion exchange system may operate unattended due to the few moving parts and only a slight potential for damage to equipment. Periodic surveillance of the equipment occurs during sampling. Feed pumps must be shut off manually and individually if leaks or equipment malfunctions are observed. The columns will not be operated overnight.

7. MAINTENANCE AND FAILURES

Very little maintenance is expected. The pump(s) have the only moving parts and are not expected to require repairs in the relatively short duration of testing. The pump(s) will be replaced at failure. The ion exchange column(s) will be replaced if breakage or leakage occurs. Leaking or severed tubing will be replaced, as needed.

8. TEST DATA

Data to be 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 solutions volumes.
- ▶ ion exchange resin volume and weight.

9. PERSONNEL REQUIREMENTS

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, they will not necessarily be working with this equipment.

10. WITNESSES

Witnesses are not required for these tests.

11. 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 10-L 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. If needed, load the 10-L Solution portion into the glovebox per Operating Procedure Z0-170-313, "Use of Air Locks." 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. Label the 10-L 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 HNO₃:

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

to LOWER the acid concentration to 7.0 M using 1.5 M HNO₃:

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

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 10-L Solution. Make certain the combined 10-L Solution and fresh acid volume ($V_o + V_a$) will not exceed the volume of the feed bottle.

4. Add the correct amounts of 10-L 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 10-L 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 and be certain that the Criticality Prevention Posting will not be violated.

8. Calculate the amount of sodium hydroxide solution needed to neutralize the acid in the expected raffinate and wash solution volumes. Make sure the combined solution volume will not exceed the volume of the raffinate bottle. Add the calculated amount of sodium hydroxide solution to the raffinate collection bottle. Label this bottle as hazardous waste and corrosive.

B. Column Operation: Extraction and Washing

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 10-L 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 neutralized 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 neutralized raffinate/wash solution collection bottle and appropriate labels to the Satellite Accumulation Area. Record the entry of the bottle on the glovebox inventory, if needed, and be certain that the Criticality Prevention Posting will not be violated.

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 per Operating Procedure Z0-170-299, "Seal Out." Submit the samples to the PFP Analytical Laboratory.
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 and into Glovebox 179-9 per Operating Procedures Z0-170-299 and Z0-170-313. 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. DISPOSITION OF TEST ITEMS

The ion exchange resins will be disposed as contaminated waste after testing. Solid Waste Operations and Environmental Engineering 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.

Neutralized raffinate/wash solutions from the tests 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 demonstrated in Room 188 of the PPSL.

13. REFERENCES

1. Dr. G. S. Barney, *Test Plan for Demonstrating Plutonium Extraction from 10-L Solutions Using EICrom Extraction Chromatographic Resins*, WHC-SD-CP-TP-078, Revision 0, Westinghouse Hanford Company, Richland, WA, August 22, 1994.

14. DATA SHEETS

Test data will be recorded in a laboratory notebook.

HANFORD JOB HAZARD ANALYSIS CHECKLIST			Page 1 of <u>1</u>
Prepared By J. A. Compton	Date 05/15/95	Area 200-W	Bldg. 234-5Z
Scope/Description: This checklist applies to anion exchange testing in the Plutonium Process Support Laboratories as described in test procedure WHC-SD-CP-TC-033.			<input checked="" type="checkbox"/> New <input type="checkbox"/> 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		Yes	No	✓	●	Reference
1. Radiation Area Work	X		✓	●	RWP Z-012	10. Respiratory Hazards	X		✓	●	RWP Z-005
2. Hazardous Waste Operations	X		✓	●	CM-4-3 / W-12	11. Electrical Hazards		X	✓		
3. Confined Space Entry		X	✓	●		12. Lock and Tag		X	✓	●	
4. Cutting/Welding		X		●		13. Scaffolding		X			
5. Roof Work		X				14. Aerial Lifts		X	✓		
6. Fall Hazards (> = 10')		X				15. Asbestos Removal		X	✓	●	
7. Excavation/Trenching		X		●		16. Other (see JHA Sht. 2):		X			
8. Asbestos Inspection Report		X		●		✓ = Formal training required. ● = Items that require a permit/form/report.					
9. Hazardous Materials	X		✓		CM-5-8 / 7.1						

Other Hazards	Yes	No	Control Measures
1. Temperature Extremes	X		Hot surface contact not normally needed; gloves available.
2. Noise		X	N/A
3. Poor Lighting		X	N/A
4. Animals/Insects		X	N/A
5. Process Chemicals/Steam		X	N/A
6. Dust		X	N/A
7. Flammable/Combustible Materials		X	N/A
8. Ladders		X	N/A
9. Wet/Slippery Floors		X	N/A
10. Uneven Terrain		X	N/A
11. Open Excavations/Trenches		X	N/A
12. Adjacent Water Hazard		X	N/A
13. Vehicle Traffic		X	N/A
14. Heavy Equipment		X	N/A
15. Rigging Operation		X	N/A
16. Manual Lifting		X	N/A
17. Power Tools		X	N/A
18. Pinch Points		X	N/A
19. Falling Objects		X	N/A
20. Sharp Objects		X	N/A
21. Overhead Obstructions		X	N/A
22. Site Control (Signs/Barricades)		X	N/A
23. Remote Work Area		X	N/A
24. Other (see JHA Sht. 2):		X	N/A

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 J. A. Compton (Signature) <i>James A. Compton</i>	Industrial Safety/Hygiene (Signature) A. G. Pines <i>A. G. Pines</i>
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