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PFP Process Engineering 15530		Plutonium Process Support Laboratories 15F00		Date 05/10/95	
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Test Plan for Anion Exchange Testing with Argonne 10-L Solutions				ECN No. N/A	

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6. Author

Name: J. A. Compton

James A. Compton
Signature

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7. Abstract

The purpose of this test plan is to determine the performance characteristics of four anion exchange resins. This information is required to scale up an ion exchange process for removing undesirable chemicals from calciner feed at PFP. The performance characteristics will be judged by comparing the total exchange capacity, the sorption and desorption of plutonium, the distribution coefficient, and other operating information in the presence of various complexing anions. The results will be compared to a similar process using organic extractants to determine the best way of removing the undesirable chemicals from the plutonium solutions.

8. RELEASE STAMP

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TEST PLAN FOR ANION EXCHANGE TESTING WITH ARGONNE 10-L SOLUTIONS**1. INTRODUCTION**

Some of the plutonium in the Plutonium Finishing Plant (PFP) is contained in the plant as nitrate solutions, sludges, oxide, oxalates, and numerous other forms. This plutonium needs to be converted to a stable, storable plutonium dioxide (PuO_2) before placing it into vault storage. All plutonium not already converted to oxide by sustained exposure to high temperatures should be viewed as potentially reactive material and not suitable for long-term storage (i.e., storage that might exceed 50 years).

Of particular concern are 2,200 L of plutonium nitrate solutions containing 16.8 kg of plutonium. Some of these solutions are stored in polyethylene containers received from Argonne National Laboratory. These containers have a volume of 10 L and are surrounded by a stainless steel outer shell. These container assemblies are generally known as "10-L Containers" and their solutions as "10-L Solutions." The 10-L Solutions are mixtures of plutonium nitrate, concentrated nitric acid, hydrochloric acid, and other corrosive chemicals. Because of chemical oxidation and radiolysis, these plastic inner bottles are potentially brittle. Recent X-ray analysis shows no evidence that these bottles have failed; however, it is considered wise to (1) remove the 10-L Solutions before the containers fail and (2) convert the plutonium nitrate to solid PuO_2 as a final storage form. Other solutions are stored in all-stainless steel containers that are less subject to embrittlement, oxidation, and leakage. Certain chemicals, such as sodium, may need to be removed from the Pu solution for the nitrate to oxide conversion process to run properly. Ion exchange is one of the removal processes to be demonstrated by the Plutonium Process Support Laboratories (PPSL) in consideration for this mission.

Four ion exchange resins have been selected for testing. These resins have already been tested for Los Alamos National Laboratory (Reference 1) and shown to perform adequately in pure plutonium solutions.

2. OBJECTIVE

These tests are to obtain operating parameters for the four tested resins while confirming that plutonium will sorb onto and elute from the resins with additional chemicals in the feed. These test results can then be compared to similar results obtained using EICrom resins (Reference 2) to determine which resin is best at removing undesirable chemicals from feed for an experimental direct denitration calciner being tested separately. Operating parameters to be obtained are (1) plutonium loading rates, (2) raffinate solution volumes, (3) elution rates and concentrations, (4) wash solution volumes required, and (5) product purity.

3. SCOPE

Four separate anion exchange resins will be tested. Testing will be performed in ion exchange columns. Long-term stability of the resins and corresponding changes in capacity will not be determined. Thermal stability will not be determined.

The tests will use some of the 10-L Solutions as the source of plutonium. Twenty to forty liters of the available 250 liters are planned for use in the ion exchange testing; however, the amounts and specific sources (i.e., blends of 10-L containers' solutions) are uncertain and may change during the testing. Product from these tests and the remaining 10-L Solutions will be used for additional process demonstrations in the PPSL.

4. TEST DESCRIPTION

4.1 TEST ITEMS - Four separate anion exchange resins will be tested. 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.

4.2 TEST ENVIRONMENT - The experimental work will be performed in Glovebox 179-6 in Room 179 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.

4.3 EQUIPMENT AND FACILITIES - The following equipment is required:

- ▷ 147-mL ion exchange columns (4)
- ▷ Anion exchange resins (4)
- ▷ Glass sample vials
- ▷ Deionized water
- ▷ Pu(NO₃)₄ storage solutions ("10-L Solutions")

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.

- ▷ Ultima Gold⁵ liquid scintillation counter cocktail
- ▷ Nitric acid (HNO₃)

4.4 EXPERIMENTAL DESIGN - During these experiments, the following parameters will be varied:

- ▷ 10-L Solution blends (up to 10)
- ▷ ion exchange resin (4)
- ▷ feed flow rate (3 and 6 mL/min)
- ▷ eluant flow rate (5 and 10 mL/min)
- ▷ eluant concentration (0.5 and 0.7 M nitric acid)

Parameters to be held constant are:

- ▷ operating temperature (room temperature)
- ▷ column size (147 mL empty volume)
- ▷ resin volume (147 mL bulk)
- ▷ wash solution volume of 150 mL

4.5 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 volume.
- ▷ ion exchange resin volume and weight.

4.6 CRITERIA/CONSTRAINTS - There are no regulatory requirements or restraints that apply directly to these experiments. Raffinate solutions will be neutralized at collection, then stored in the Satellite Accumulation Area until the drain system is ready to accept them.

5. EXPECTED RESULTS

These tests will yield resin loading and plutonium concentration data that will allow these resins to be compared to previously tested or used ion exchange resins. The comparisons will be based on the resins' capacities and volumes of the raffinate and product streams per gram of plutonium recovered, calculated from the experimental data. Decisions regarding the selection of a calciner feed pretreatment system for use on remaining PFP plutonium solutions can then be made.

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

6. TEST PROCEDURE

Procedure WHC-SD-CP-TC-033, "Test Procedure for Anion Exchange Testing with Argonne 10-L Solutions" has been written separately for these tests. The general order of operational steps will be:

- A. Determine the blend of 10-L Solutions and volume to be used.
- B. Add the desired resin(s) to the column(s).
- C. Calculate the necessary amount of sodium hydroxide solution for neutralizing the expected amounts of raffinate and wash solutions. Add the sodium hydroxide solution to the raffinate collection bottle.
- D. Start feed flow upward through column. Refill feed container, as needed, during operation.
- E. Sample raffinate stream periodically, especially when green color of dissolved plutonium gets near the top of the resin.
- F. Wash the column with 7 M nitric acid.
- G. Move the neutralized raffinate solution to the Satellite Accumulation Area.
- H. Elute the plutonium from the columns with dilute nitric acid using downward flow. Collect the product in plastic bottles, sampling frequently.
- I. Store the product solution until needed for other process demonstrations.

7. SAFETY

7.1 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, "Uniform Laboratory Specifications" (Reference 3). 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 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.

7.2 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 and high neutron 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.

7.3 CHEMICAL - 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 eye protection 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
Reillex HPQ™ Ion Exchange Resin	35852
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.

7.4 THERMAL - There are no heated objects or surfaces to present thermal hazards. Exothermic chemical reactions will be performed slowly enough to preclude dangerous surface temperatures. Gloves required for handling these chemicals also are a barrier minimizing the consequences of contact with hot surfaces.

7.5 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.

7.6 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.

7.7 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.

8. QUALITY ASSURANCE

Quality work in this project is assured by:

- ◆ peer review of this test plan and the results report by other PPSL members and PFP Engineering organizations.
- ◆ using reviewed and approved procedures for all experimental and ancillary work.

- ◆ the use of established standards, duplicate analyses, and reviewed/approved procedures in the PFP Analytical Laboratory.

9. ORGANIZATIONAL RESPONSIBILITIES

9.1 PLUTONIUM PROCESS SUPPORT LABORATORIES - The PPSL writes, reviews, and approves this test plan, any required procedures, and the results report. All experimental assembly, work, and disassembly are performed by PPSL personnel. The PPSL also assists Process Engineering and Design Engineering with setting up the system to be used for processing the remainder of PFP's plutonium solutions.

9.2 PFP PROCESS ENGINEERING - The PFP Process Engineering group will review and approve this test plan and the results report. Process Engineering may also elect to observe portions of the testing. Finally, Process Engineering will apply the test results to a larger-scale system for purifying the plutonium from additional solutions.

9.3 PFP ANALYTICAL LABORATORY - The PFP Analytical Laboratory performs routine analyses on samples generated during these experiments. These analyses include plutonium concentrations before and after ion exchange, acid concentrations, and metal impurities before and after ion exchange.

9.4 PFP DESIGN ENGINEERING - The PFP Design Engineering Group applies the test results into the mechanical design of the system that Transition Operations will use on the remainder of the PFP's liquid solutions.

9.5 PFP TRANSITION OPERATIONS - The PFP Transition Operations Group furnishes the 10-L Solutions to be used during testing. Transition Operations also accepts any raffinate solutions disposed to the building drain system.

9.6 PFP SOLID WASTE OPERATIONS - Solid Waste Operations will dispose of waste materials generated during testing.

9.7 PFP NUCLEAR SAFETY - The PFP Nuclear Safety Representative for the PPSL will review and approve this test plan and any related procedures. The Nuclear Safety Representative also makes periodic inspections of the laboratory area for criticality prevention and other purposes.

9.8 PFP CRITICALITY SAFETY - The PFP Criticality Safety Representative will review and approve this test plan and any related procedures. The Criticality Safety Representative also inspects the laboratory area periodically for compliance with criticality prevention specifications and postings.

9.9 PFP HEALTH PHYSICS - The PFP Health Physics Group writes the Radiation Work Procedures covering the tasks required by this test plan. The Radiological Control Technicians (RCTs) also determine the dose rates present at the work sites and on containers, check for contamination at the work

locations, and check for contamination in the laboratory rooms. The RCTs also are present whenever any materials enters or exits a glovebox and whenever uncontrolled contamination is located.

9.10 PFP QUALITY ASSURANCE - The PFP Quality Assurance Group reviews and approves this test plan and related procedures.

10. SCHEDULE

The testing activities will require considerable time due to the inherently slow operation of ion exchange processes. The need to remove certain impurities from feed to the direct denitration calciner also requires that the ion exchange testing continue throughout calciner testing. Predicted dates for various phases of the testing are:

Issue test plan	May 3, 1995
Issue test procedure	May 5, 1995
Complete apparatus assembly	May 5, 1995
Complete testing	August 15, 1995
Issue results report	September 22, 1995

11. REPORTS

Progress on these experiments will be reported in the PPSL Weekly Report. Results will be published in a Supporting Document after completion of testing.

12. REFERENCES

1. Marsh, S. F., *The Effects of Ionizing Radiation on Reillex™ HPQ, a New Macroporous Polyvinylidene Resin, and on Four Conventional Polystyrene Anion Exchange Resins*, LA-11912, Los Alamos National Laboratory, Los Alamos, NM, December, 1991.
2. Barney, G. S., *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.
3. Criticality Prevention Specification CPS-L-114-00020, Rev./Mod. C-2, issued March 15, 1995.

13. DATA SHEETS

The data will be recorded in a controlled laboratory notebook.