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TEST PLAN FOR DEMONSTRATING PLUTONIUM EXTRACTION
FROM SOLUTIONS STORED AT PFP USING EICHROM
EXTRACTION CHROMATOGRAPHIC RESINS

Pages: 18

ENGINEERING CHANGE NOTICE

Page 1 of 21. ECN 629405Proj.
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13a. Justification (mark one) Criteria Change ☒ Design Improvement ☐ Environmental ☐ Facility Deactivation ☐ As-Found ☐ Facilitate Const ☐ Const. Error/Omission ☐ Design Error/Omission ☐			
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Page 2 of 2

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629405

15. Design Verification Required [] Yes [X] No	16. Cost Impact				17. Schedule Impact (days)	
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18. Change Impact Review: Indicate the related documents (other than the engineering documents identified on Side 1) that will be affected by the change described in Block 12. Enter the affected document number in Block 19.

SDD/DD	[]	Seismic/Stress Analysis	[]	Tank Calibration Manual	[]
Functional Design Criteria	[]	Stress/Design Report	[]	Health Physics Procedure	[]
Operating Specification	[]	Interface Control Drawing	[]	Spares Multiple Unit Listing	[]
Criticality Specification	[]	Calibration Procedure	[]	Test Procedures/Specification	[]
Conceptual Design Report	[]	Installation Procedure	[]	Component Index	[]
Equipment Spec.	[]	Maintenance Procedure	[]	ASME Coded Item	[]
Const. Spec.	[]	Engineering Procedure	[]	Human Factor Consideration	[]
Procurement Spec.	[]	Operating Instruction	[]	Computer Software	[]
Vendor Information	[]	Operating Procedure	[]	Electric Circuit Schedule	[]
OM Manual	[]	Operational Safety Requirement	[]	ICRS Procedure	[]
FSAR/SAR	[]	IEFD Drawing	[]	Process Control Manual/Plan	[]
Safety Equipment List	[]	Cell Arrangement Drawing	[]	Process Flow Chart	[]
Radiation Work Permit	[]	Essential Material Specification	[]	Purchase Requisition	[]
Environmental Impact Statement	[]	Fac. Proc. Samp. Schedule	[]	Tickler File	[]
Environmental Report	[]	Inspection Plan	[]		[]
Environmental Permit	[]	Inventory Adjustment Request	[]		[]

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Cog. Mgr. C. S. Sutter	2/7/96	QA	
QA		Safety	
Safety S. E. Nunn	2-9-96	Design	
Environ.		Environ.	
Other L. T. Nirider	2-7-96	Other	
Rad Con. S. C. Snyder	2/7/96		
JRG J. R. Carey	2/9/96		
		DEPARTMENT OF ENERGY	
		Signature or a Control Number that tracks the Approval Signature	
		ADDITIONAL	

Test Plan for Demonstrating Plutonium Extraction from Solutions Stored at PFP Using ElChrom Extraction Chromatographic Resins

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Westinghouse Hanford Company, Richland, WA 99352

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Abstract: Plutonium solutions stored at the Plutonium Finishing Plant must be treated to convert the plutonium to a safe, solid form for storage and to remove the americium so that radiation exposure can be reduced. Extraction chromatographic resins will be tested for separating plutonium from these solutions in the laboratory. Separation parameters will be developed during the testing for large scale processing of the solutions. Use of chromatographic resins will allow plutonium separation with minimum of chemical addition to the feed and without the need for plutonium valence adjustment. The separated plutonium will be calcined to plutonium oxide by direct solution calcination.

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TEST PLAN FOR DEMONSTRATING PLUTONIUM EXTRACTION FROM SOLUTIONS STORED AT PFP
USING EICHROM EXTRACTION CHROMATOGRAPHIC RESINS

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Date Published:
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TEST PLAN FOR DEMONSTRATING PLUTONIUM EXTRACTION FROM SOLUTIONS STORED AT PFP USING EICHROM EXTRACTION CHROMATOGRAPHIC RESINS

G. S. Barney

1.0 INTRODUCTION

The Plutonium Finishing Plant (PFP) received and generated plutonium-bearing solutions which have been stored for scrap processing. These solutions are highly acidic and are considered reactive and corrosive. The solutions contain a variety of components including various acids from laboratory waste, salts from processing [e.g. KNO_3 and $\text{Mn}(\text{NO}_3)_2$ from oxalate removal], etc. In order to store the plutonium contained in these solutions safely it is necessary to convert the plutonium to a solid form. The purpose of this testing is to develop the separation parameters required for large-scale processing of these (and similar) solutions. Separation of plutonium is required for solutions containing components that are incompatible with conversion of plutonium to a solid form.

Extraction chromatographic resins are used to selectively remove plutonium and other actinide elements from almost any acidic waste solution. This separation technique removes the reactive components from plutonium waste so that the separated plutonium can be solidified into a stable chemical form for long-term storage. The resin extracts plutonium from waste solutions as they are pumped through a resin bed. The concentrated plutonium is then removed from the resin by eluting with a solution containing a complexant such as citrate. The resulting plutonium solution can be solidified by calcination or precipitation of the plutonium from solution.

Extraction chromatographic resins combine the selectivity of solvent extraction processes with the simplicity of a column chromatographic system. While conventional solvent extraction is a liquid-liquid system, extraction chromatographic resins perform the same separations in a solid-liquid system. This allows use of much simpler equipment, easier material handling, and reduced capital cost in construction of process equipment.

The resins used in extraction chromatography are prepared by adsorption of a conventional or slightly modified extractant on a macroporous polymeric support. This preparation process immobilizes the extractant while retaining the chemical properties (distribution ratios, selectivities, etc.) of the original liquid solvent system. Thus, extractants known to selectively extract metal ions in a solvent extraction process (i.e. CMPO-TBP mixtures used in the TRUEX process for extraction of TRU elements) can be immobilized on a solid support and will retain their selectivity and extraction capacity.

2.0 OBJECTIVE

The primary objective of these tests is to evaluate extraction chromatographic resins for separating plutonium from solutions stored at the Plutonium Finishing Plant. Plutonium must be removed to levels below the TRU limit (100 nCi/g) for the resins to be considered successful. The waste effluent from the resin columns must also meet the requirements for disposal in high-level waste tanks. Removal of plutonium, americium, and uranium will be studied. The performance of EICrom resins (EICrom Industries, Inc.) and one laboratory-prepared resin will be evaluated. The various resins must be applied in a logical sequence to achieve optimum performance of the actinide removal system. This sequence will be determined from laboratory extraction parameters to be determined in these tests.

The tests will yield several types of extraction parameters. Distribution coefficients (K_d 's) for actinides will be determined under a range of conditions (acid concentration, waste composition, actinide concentration, and resin type) expected in the actinide removal system. These batch equilibrium tests will be used for setting experimental parameters for the column tests. Column testing will provide elution data that will be used to predict extraction rates for dissolved actinides, capacities, and regeneration efficiencies.

3. SCOPE

All tests will be performed on a laboratory scale. Both batch equilibration tests and flow-through column tests will be performed. The batch tests will use several grams of resin and less than 20 mLs of plutonium solution with a range of compositions. A number of these batch tests will be completed to cover the required range of conditions of acid concentration, waste composition, actinide concentration, and resin type. Laboratory columns filled with resins will have a bed volume of about 50 mLs. Several columns of each resin will be prepared. Volumes of wastes to be pumped through these columns will be up to about four liters.

Interfaces with laboratory coworkers conducting other testing with the test solutions (plutonium separations, precipitation, and calcination), laboratories performing TRUEX testing, and other waste partitioning activities will be important. Exchange of information with personnel performing these tests will be continued during these studies. Another important interface is with the operations group which sets overall goals for plutonium separation and waste disposal.

4. DESCRIPTION OF TEST

4.1 Test Item

Extraction chromatographic resins to be tested were chosen for their known effectiveness in removing specific actinides from the test solutions. Specific removal of actinides is important so that the capacity of the resins is not used up by non-radioactive components of the wastes. It is also important that the actinides have high distribution coefficients for extraction onto the resins since it is important to concentrate them. A few commercially-prepared resins are available for this type of application. The resins to be tested and their probable applications are given in Table 1.

TABLE 1
RESINS TO BE TESTED FOR REMOVAL OF RADIONUCLIDES
FROM ACIDIC WASTES

Resin	Type	Manufacturer	Applicable Actinides
TEVA-Spec	didecyloctylmethylammonium nitrate extractant on a macroporous polymeric support	EIChrom Industries, Inc.	Pu(IV), Th(IV)
U/TEVA-Spec	Dipentyl Pentylphosphonate extractant on a macroporous polymeric support	EIChrom Industries, Inc.	Pu(IV), U(VI).
CMPO-Impregnated Resin	CMPO extractant on a macroporous polymeric support	Laboratory-prepared	Pu(IV), Pu(VI), U(VI), Am(III)

Two resins manufactured by EIChrom Industries, Inc. will be tested as well as a laboratory-prepared resin. TEVA-Spec resin is commercially available from EIChrom Industries, Inc. (Darien, Illinois) and is specifically designed to extract tetravalent actinide elements from acidic solutions. It contains an impregnated extractant, didecyloctylmethylammonium nitrate extractant on a polymeric resin substrate. Most other metal ions do not interfere with the extraction. Thorium, technetium, mercury and tetravalent neptunium are extracted by this resin, but are not expected to be present in significant concentrations in the test solutions. Dissolved metallic elements that make up the major portion of the solutions (i.e. Na, Al, Fe, Cr, Mg, and Ca) should not be extracted, according to EIChrom literature. The resin can be regenerated by eluting actinide elements with dilute acid or with

complexing agents. The useful loading capacity of this resin for plutonium is about 6 - 25 grams of plutonium/L of resin bed. The particle size range of resin beads to be tested is 100 to 150 μ .

The U/TEVA-Spec resin is an extraction chromatographic resin for the selective sorption of uranium and tetravalent actinides. It consists of a liquid extractant, dipentyl pentylphosphonate, adsorbed onto macroporous polymeric resin beads. The resin is an acrylic ester polymeric adsorbent. Unlike the TRU-Spec resin, this material will not extract americium. A separation of americium from other actinides can therefore be devised using these two resins. Very few metal ions interfere with the extraction of uranium and tetravalent actinides. Zirconium and ruthenium are extracted to a relatively small extent, but should not significantly affect actinide extraction. The resin can be regenerated by eluting the actinides with dilute nitric acid. It has a useful loading capacity of about 4 to 8 grams of actinide/L of resin bed. The particle size range of resin beads to be tested is 100 to 125 μ .

In addition to the EICrom resins, a resin prepared in our laboratory will be tested. It was prepared by loading liquid CMPO onto macroporous resin beads (Amberlite XAD-16) from a methanol solution (Barney and Cowan, 1992). The resulting material is about 50% by weight CMPO and appears to extract americium more strongly than the TRU-Spec EICrom resin. It should have similar selectivity to the TRU-Spec resin since the same extractant was used (except without the TBP solvent).

The test solutions have not been well characterized for solution composition. Only plutonium and americium concentrations have been reported and general descriptions of the acids that are present. These solutions shall be characterized thoroughly before the separations work with EICrom resins can begin.

The proposed testing will treat widely different compositions of the test solutions that are available. This will allow a determination of the general applicability of the extraction chromatographic method to different feed compositions. Depending on feasibility, two different test solutions will be tested initially that contain the laboratory wastes and concentrated filtrate solution from the oxalate precipitation process. About two liters of each solution (previously filtered through a membrane with a pore size of 0.3 μ m) will be passed through columns of resin at a constant flow rate. The plutonium and americium concentration in the column effluent will be monitored by sampling it periodically and counting the plutonium with a liquid scintillation analyzer. The volume of the column resin beds will be about 50 mLs and the flow rate will be about 15 mLs/min.

After the columns are loaded with plutonium, they will be eluted with an ammonium citrate solution at pH 4 or other complexant or dilute nitric acid. Again, the effluent will be periodically monitored for plutonium concentration to determine the required volume of eluant. The eluted solution will be

directly calcined or precipitated and calcined to produce the stabilized plutonium oxide product.

The information to be obtained in the tests will be (1) loading capacity of the resins, (2) effects of waste composition on the plutonium loading curves, (3) loading and regeneration efficiency of the resins, (4) required volume of stripping solution, and (5) concentrations of reactive impurities in the stabilized PuO_2 product and product density. These data will allow evaluation of the method and provide information for scale-up of the process.

4.2 Test environment

The testing will be performed in PPSL laboratory rooms equipped with open-faced hoods for handling the test solutions. Solution preparation, extraction chromatographic experiments, and counting of the solutions containing alpha emitters (plutonium, americium, and uranium) will be performed in Rooms 191, 187 (Hoods 1 to 4), and 183 of the PPSL Laboratory. Samples generated from the testing will be sealed and counted using a liquid scintillation analyzer in Room 183. Non-radioactive solutions, such as the eluant will be prepared in the cold lab (Room 191 of 234-5 Building).

4.3 Equipment and Facilities

At least two open-faced hoods in the PPSL must be available for preparing test solutions and performing the extraction chromatographic experiments. Space in the cold labs and in the counting room will also be used. Appropriate alpha survey equipment must be available for contamination control. Applicable Radiation Work Permits for the laboratory rooms used in this work must be approved and followed. Current RWP's do not need revision because of this study.

Equipment required for these tests includes apparatus for batch equilibration, column tests and radioactive analysis. The batch measurements will be performed using 20 ml glass bottles with screw caps to contain the resin-solution mixtures. These bottles will be shaken with a variable speed flat bed shaker to speed equilibration. The solutions will be sampled periodically and the actinide concentrations measured to determine the required equilibration time. Chromatography columns (Spectrum Medical Industries, Inc.) will be used to contain the resin in the column tests. The resin bed dimensions can be adjusted with these columns. Precision metering pumps (Eldex Laboratories, Inc., model E-120-S) will be used to pump solutions through the columns at a known, constant rate. Effluents from the columns will be sampled automatically using a fraction collector (Haake Buchler Instruments, Inc., model LC 200) that will collect constant volume fractions. Actinide concentrations will be measured using liquid scintillation counting (Packard 1500 Tri-Carb liquid scintillation analyzer). The required laboratory equipment for these tests already exists in the PPSL.

The extraction chromatographic resins to be tested are listed in Table 1.

The two solutions to be tested will be obtained from PFP Operations. Two liters of each solution will be required for the testing.

4.4 Data

The important experimental conditions for batch equilibrium measurements of radionuclide distribution include actinide concentrations (plutonium, uranium and americium), solution chemical composition, resin type, and acid concentration. In addition, since actinide elution will be important, the types of solutions used as elutants and their concentration are important. For the column experiments, flow rate and bed volume are important experimental parameters, in addition to those given above. The ranges of experimental parameters for each type of measurement are given in Table 2.

TABLE 2
EXPERIMENTAL PARAMETERS AND RANGES

Measurement Type	Parameter	Range
Batch Equilibration (Distribution Coefficients)	Actinide Type	Plutonium, Uranium, Americium
	Actinide Concentration	1 to 20 g/L
	Waste Solution Chemical Composition	HCl, HNO ₃ , H ₂ SO ₄ , HF, HClO ₄ , Metal Salts
	Resin Type	See Table 1
	Acid Concentration	1.0 to 6.0 M
Column Tests (Adsorption Rates, Capacity)	Above Parameters	Above Ranges
	Flow Rate	0 to 15 ml/min
	Bed Volume	1 to 50 mls

The experimental concentration ranges are based on estimates of waste solution composition which is the feed for the adsorption columns. Actinide concentrations will cover the range of expected concentrations in the solutions.

4.5 Criteria/Constraints

The criteria for successful application of this testing are (1) that plutonium separation from 10-L solutions using extraction chromatographic resins is essentially complete, leaving the reactive components of these

solutions in the waste along with plutonium concentrations below the TRU limit of 100 nCi/g, (2) the plutonium is concentrated by at least a factor of two before calcination, and (3) the waste will be acceptable for disposal in high-level waste tanks. The separated plutonium will be in a form that can easily be calcined to plutonium oxide or precipitated and then calcined. Data generated in these tests will allow prediction of the performance of the resins in large-scale process application.

Some constraints on these tests are as follows:

(1) The chemical compositions of these solutions are not known with a great amount of confidence. Obviously, before any chemical separations are attempted, the solutions shall be analyzed for cations and anions. Adjustment of the feed solution composition will be required in some cases for the separation to be successful. An example is when hydrofluoric acid is present. Aluminum must be added to prevent the formation of plutonium fluoride complexes which are not extractable.

(2) Working volumes of test solutions will be limited due to the radiological limits on the open-faced hoods. This may require storage of some of the solutions in hoods other than those used in the extraction chromatography tests.

5.0 EXPECTED RESULTS

The tests will likely show that several resins can be applied to removal of plutonium from stored solutions at the PFP. Based on available data from EICrom, data from other laboratories (i.e. Los Alamos National Laboratory) performing work on extraction chromatographic resins, and on data obtained in this laboratory, commercially-available adsorbents can be found that will fulfill the requirements of plutonium removal and separation from reactive solution components. Sizing the treatment columns for the quantities of solutions to be processed will determine if a given resin is practical to use. The size of the columns will be estimated from expected solution flow rates, rates of extraction and extraction capacities of the resins measured in these tests. One of the most important unknown factors in these tests is the effect of various dissolved salts in the test solutions. These salts may compete with plutonium for extraction on the resins and may reduce extraction capacity.

6.0 TEST PROCEDURE

Procedures for measuring actinide distribution coefficients (K_d values), extraction rates, and extraction capacity are summarized in the

following paragraphs. There are two general types of procedures to be used: batch equilibration measurements and column flow-through measurements.

In the batch equilibration measurements of radionuclide K_d values, a weighed amount of resin is equilibrated with a measured volume of solution containing known concentrations of actinide and non-radioactive components. The acid concentration will be adjusted with HNO_3 and measured after equilibration. The time required for equilibration is determined by sampling the solution periodically and measuring the actinide concentration. When a constant actinide concentration is reached, equilibrium has been attained. The concentration of actinide in the solution after equilibration is used to calculate the amount of actinide adsorbed on the resin. A K_d value is then calculated using this data.

For the column experiments, commercially-available liquid chromatography columns will be filled with the resin(s). Solutions containing known concentrations of actinide and inert components will be pumped through the columns at a constant, known flow rate. Samples of the effluents from the columns will be counted for actinide concentration. Breakthrough curves will be plotted to show actinide concentration in the effluent versus effluent volume. For extraction rate experiments, the flow rate will be varied and the effect of flow rate on the actinide concentration in the effluent will be evaluated. Resin capacities will be determined by measuring the volume passed through the column when the actinide is first observed in the effluent.

The size of the columns to be used will be determined by results of the batch equilibration measurements. Large distribution coefficients will require that smaller columns be used in order to reach the capacity of the adsorbent. Other considerations in determining column size are height to diameter ratio (must be large enough to prevent channeling), and distribution of the influent solution to the bed diameter.

The effects of acid concentration, actinide concentrations, and concentrations of interfering ions will be determined in both the batch and column tests by changing their values over the specified range of these parameters and measuring the effects on adsorption.

7.0 SAFETY

All standard laboratory safety measures will be observed. All radiation work procedures will be adhered to, as described in HSRCM-1, "Hanford Site Radiological Control Manual." The following specific permits will apply: Z-34, "Low Radiological Risk Activities in Radiological Controlled Areas (RCA's)," and Z-12, "Routine Work Inside Gloveboxes and Hoods in SCA's." All work with chemicals will follow the Westinghouse Hanford Company Chemical Hygiene Plan (WHC-SD-CP-HSP-001).

Wastes containing extraction chromatographic resins, inorganic adsorbents, synthetic wastewaters, and actinides (Pu, Am, and U), will be generated from these tests. The resins are not likely to be classified as hazardous wastes because of their relatively high flash point (165 °F), unreactiveness, and low toxicity. The organic resins will contact a weak oxidizer (dilute HNO₃) but will not be an explosive hazard. They will, however, generally be contaminated with radioactive tracers. The aqueous waste solutions will be neutralized with NaOH solution before disposal to neutral pH and will be radioactive. These solutions can be disposed of in the D-4 drain. PFP Environmental Compliance will approve the disposal of any of these wastes. Material Safety Data Sheets are available in the laboratory for all of the resins and reagents that will be used.

8.0 QUALITY ASSURANCE

These tests are assigned Approval Designator SQ, as per Section 12.7 of the WHC-3-5 Manual. The quality assurance implementation is under the direction of the cognizant chemist. All procedures performed by the PPSL will be handled in accordance with the Westinghouse Hanford Company manual "Laboratories Administration" WHC-CM-5-4. All work will be documented in laboratory notebooks according to Section 3.6 of the WHC-CM-5-4 manual.

All analyses will be performed according to written procedures and will be verified with standard samples before use. Other chemical analyses will be performed by Analytical Services which maintains a Laboratory Measurement Control System (LMCS). The LMCS provides for analysis of standards and replicate analyses to assure quality control.

9.0 ORGANIZATION AND FUNCTIONAL RESPONSIBILITIES

Organizational responsibilities are summarized in Table 3.

Table 3. Organizational Responsibilities

Organization	Responsibilities
Plutonium Process Support Laboratories	Test Planning
	Test Execution
	Data Analysis
	Documentation
PFP Process Engineering	Technical Consultation
	Approval
	Data Evaluation
Analytical Services	Sample Analysis
Standards Laboratory	Standards Preparation
PFP Safety/Quality Assurance	Review Test Plan And Reports

10.0 ESTIMATED SCHEDULE AND COSTS

A summary of the estimated costs is shown below:

Exempt	\$40,000	
Nonexempt	\$30,000	
Materials	\$10,000	
Analyses	<u>\$10,000</u>	(Analytical Services)
Total	\$90,000	

Laboratory studies are scheduled to begin immediately after approval and release of this test plan and receipt of the test solutions. The work will continue through the end of March, 1996 with a final test report due March 31, 1996.

11.0 REPORTS

Periodic status reports will be issued via the organizational weekly reports. Letter reports will be issued as tasks are completed. A summary report will be issued March 31, 1996.

12.0 REFERENCES

1. WHC-CM-5-4, Rev. 0, Laboratory Administration, Westinghouse Hanford Company, Richland, Washington.
2. Barney, G. S. and R. G. Cowan, Separation of Actinide Ions From Radioactive Waste Solutions Using Extraction Chromatography, WHC-SA-1520-FP, Westinghouse Hanford Company, Richland, Washington.
3. WHC-CM-3-5, Document Control and Records Management Manual, Westinghouse Hanford Company, Richland, Washington.
4. Criticality Prevention Specifications, CPS-L-114, Westinghouse Hanford Company, Richland, Washington.
5. Radiological Work Permits, Z-012, Rev. #1, "Routine Work Inside Gloveboxes and Open-face Hoods, Z-034, "Low Radiological Risk Activities in Radiological Controlled Areas," Westinghouse Hanford Company, Richland, Washington.

13.0 DATA SHEETS

Detailed, step by step test instructions will be prepared before any laboratory work is initiated. These instructions will be entered in a controlled laboratory notebook in sufficient detail to allow operation by trained laboratory personnel according to WHC-CM-5-4, Section 5.4. 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.