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Spherical Resorcinol-Formaldehyde Resin Analysis Following Actual Hanford Tank Waste Processing

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LEGAL NOTICE

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6/7/2006
for W. Tamosaitis

ACCEPTED FOR
WTP PROJECT USE

May 2006

Test specification: 24590-PTF-TSP-RT-04-0005, Rev. 0
Test plan: TP-RPP-WTP-378, Rev. 0
Test exceptions: 24590-PTF-TEF-RT-05-00008
24590-PTF-TEF-RT-05-00011

R&T focus area: Pretreatment
Test Scoping Statement(s): A-235

Battelle—Pacific Northwest Division
Richland, Washington 99352

COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specification 24590-PTF-TSP-RT-04-0005, Rev. 0 and Test Plan TP-RPP-WTP-378, Rev. 0, as modified by test exceptions 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

Gordon H. Beeman

Gordon H. Beeman, Manager
WTP R&T Support Project

5/16/06
Date

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Terms and Abbreviations

AEA	alpha energy analysis
ASO	Analytical Support Operations
ASR	analytical services request
AV	apparatus volume
BNI	Bechtel National, Inc.
BS	blank spike
BV	bed volume
C/C_o	analyte concentration in column effluent divided by analyte concentration in feed
DI	deionized (water)
DOE	U.S. Department of Energy
EQL	estimated quantitation limit
GEA	gamma energy analysis
HLW	high-level waste
IBC	IBC Advanced Technologies, Inc., American Fork, Utah
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
KPA	kinetic phosphorescence analysis
LAW	low-activity waste
LDR	Land Disposal Restriction
LEPS	low-energy photon spectrometry
M	molarity, moles/liter
MDL	method detection limit
MRQ	minimum reportable quantity
MS	matrix spike
M&TE	measuring and test equipment
NA	not applicable
ND	not detected
ORP	Office of River Protection
PNWD	Battelle—Pacific Northwest Division

QA	quality assurance
QC	quality control
QAPjP	Quality Assurance Project Plan
QARD	Quality Assurance Requirements and Descriptions
RCRA	Resource Conservation and Recovery Act
RF	resorcinol-formaldehyde
RPD	relative percent difference
RPP-WTP	River Protection Project-Hanford Tank Waste Treatment and Immobilization Plant
RPL	Radiochemical Processing Laboratory (PNWD facility)
R&T	research and technology
SRNL	Savannah River National Laboratory
TCLP	Toxicity Characteristics Leach Procedure
TRU	transuranic
WTP	Waste Treatment Plant
WTPSP	Waste Treatment Plant Support Project

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Testing Summary

Spherical resorcinol-formaldehyde (RF) resin, Microbeads Lot 5E-370/641, was previously tested in a lead-lag column system on Hanford tank wastes. One shakedown test was conducted on the system with AP-101 simulated Hanford tank waste followed by two actual tank waste tests, one with AP-101 and one with AN-102. This report describes characterization of each resin bed following processing with these wastes. The resin characterization post-testing satisfied the requirements of Technical Scoping Statement A-235.

Objectives

The test objectives for actual waste processing were to:

- provide sufficient characterization data to evaluate ion exchange performance for spherical RF resin[a]
- perform column testing with AP-101 simulant and two actual waste samples (AP-101 and AN-102, both diluted to 5 M Na) and determine the loading and elution performance under nominal Hanford Tank Waste Treatment and Immobilization Plant (WTP) processing conditions in support of commissioning [a]
- analyze spent resin to provide information on the retention of metals on the resin.

The only test objective for this report is further discussed in Table S.1.

Table S.1. Test Objectives

Test Objective	Objective Met?	Discussion
Analyze spent resin to provide information on the retention of metals on the resin.	Yes	Spent resin was dried and analyzed in total for gamma-emitting radionuclides. Duplicate 0.6-g aliquots from each resin bed were acid digested and analyzed by inductively coupled plasma-mass spectrometry (ICP-MS), inductively coupled plasma-atomic emission spectrometry (ICP-AES), kinetic phosphorescence analysis (KPA) and radiochemical methods. Results are summarized in Table S2. • The Ag, As, Ba, Cd, Pb, and Se concentrations were measured at ≤10 μg/g.(1) • Total Cr was present at 340 μg/g in the spent resin and could possibly exceed the Toxicity Characteristic criteria of 40 CFR 261.24 (Resource Conservation Recovery Act [RCRA]). However, Cr retained on the resin after the 0.5 M HNO_3 elution was not expected to be solubilized with dilute acetic acid (per the Toxicity Characteristic Leach Procedure [TCLP] test). A TCLP test needs to be completed to evaluate Cr with respect to regulatory threshold.(2) • The spent resin contained insufficient transuranic (TRU) concentration (2 nCi/g) to characterize it as TRU waste (100-nCi/g threshold). • The Cs isotopic fractionation indicated a minor influence from the original simulant process test. • All sources of alpha and beta-emitting constituents were accounted for.(3)
(1) The RCRA-listed metal Hg was not analyzed. (2) The TCLP and regulatory analysis using spiked simulant processing is currently scheduled to be conducted at Savannah River National Laboratory (SRNL) under technical scoping statement A-205. (3) Excluding volatile and low-energy components such as ^{3}H, ^{129}I, and ^{63}Ni.		

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[a] Column performance testing with Hanford tank wastes AP-101 and AN-102 was reported previously.

Table S.2. Summary of Analyte Concentrations in Spent RF Resin

Analyte	Resin A, µg/g	Resin B, µg/g	Analyte	Resin A, µCi/g	Resin B, µCi/g
Ag	[3.1]	[3.4]	^{60}Co	7.87E-2	1.96E-2
As	<6.4	[6.9]	^{126}Sn/Sb	3.58E-4	<8E-4
Ba	[1.5]	[1.0]	^{137}Cs	3.52E-1	3.68E+0
Cd	[0.58]	[0.69]	^{238}Pu	3.61E-4	1.69E-4
Cr	338	337	$^{239+240}$Pu	1.72E-3	6.21E-4
Cs (total)	0.0204	0.187	^{241}Am	2.72E-4	2.41E-4
Na	[68]	[160]	^{242}Cm	<1E-5	<1E-5
Ni	26.1	[9.7]	$^{243+244}$Cm	<2E-5	<2E-5
Pb	[9.5]	[8.6]	Sum of alpha	2.35E-3	1.04E-3
Se	[8.8]	[9.5]	Total alpha	1.86E-3	9.32E-4
^{99}Tc	18.9	19.4	Sum of beta	7.53E-1	4.02E+0
Th	<75	<74	Total beta	6.60E-1	3.70E+0
U	193	101	**Cs isotopics**	**Resin A, %**	**Resin B, %**
			^{133}Cs	69.7	62.9
			^{135}Cs	12.1	16.2
			^{137}Cs	18.2	20.9

Notes:
Other gamma-emitting analytes were evaluated and were less than the method detection limit. Bracketed results were greater than the method detection limit (MDL) but less than the estimated quantitation limit (EQL). The less-than values (<) indicate that the results were less than the MDL; the MDL is the instrument detection limit multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within ±15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.
Reference date: 8/15/05.

Test Exceptions

Specific test details were modified in Test Exceptions 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. Table S.3 summarizes the test exceptions to the test plan and provides a discussion of the impacts on the tests.

Table S.3. Test Exceptions

Test Exception ID	Test Exceptions	Discussion
24590-PTF-TEF-RT-05-00008	Modify elution volumes to include range from 15 to 30 bed volumes (BVs). The actual volume will be determined in consultation with the research and technology (R&T) lead.	The R&T lead directed use of a final 30-BV 0.5 M nitric acid elution for both the lead and lag columns. (e-mail Meehan to Fiskum 9/1/05)
	For the shakedown test only, increase the water rinse to 8 BVs following elution. The water rinse solutions, collected in 1- to 2-BV increments, are to be analyzed for pH using pH paper indicating-strips.	This test was to determine how Cs elution was affected by water. This test exception had no effect on the spent resin analysis.
24590-PTF-TEF-RT-05-00011	The interval for lag column eluate sample collection after the last waste tested will be determined in consultation with the R&T lead.	Round the clock coverage for the AN-102 processing operation was estimated to last 1 week. However, the AN-102 load condition continued much longer than anticipated. The lag column elution had to be conducted on the following week with more limited resources. Eluate samples were collected in 1.4-BV increments from 0 to 10 BVs, one composite from 10 to 22 BVs, and then again in 1.4-BV increments from 22 to 29 BVs processed. The sample collection alteration did not affect the evaluation of the spent resin analysis.

Results and Performance Against Success Criteria

Success criteria for the River Protection Project-Hanford Tank Waste Treatment and Immobilization Plant (RPP-WTP) project specific to the spent resin analysis were to demonstrate that the elution performance meets process requirements and the spent resin analysis indicates no problems with the planned disposal pathway. These are further discussed in Table S.4.

Table S.4. Success Criteria

Success Criteria	Discussion
Elution performance meets process requirements.	Residual Cs concentration on the resin was back-calculated as a function of elution progress. The design-limit of 4.2 µg Cs per g resin was met after processing 8 BVs 0.5 M nitric acid.
Spent resin analysis indicates no problems with the planned disposal pathway.	Resins were analyzed for RCRA metals (except Hg), total Cs, and TRU analytes. • Total Cr was present in the spent resin at 340 µg/g and could possibly exceed the Toxicity Characteristic criteria of 40 CFR 261.24 (RCRA). • The Ag, As, Ba, Cd, Pb, and Se were quantified at ≤10 µg/g. • The highest resin TRU content was 2 nCi/g, <<100-nCi/g threshold classification for TRU waste. • The highest residual ^{137}Cs on the spent resin was 3.2 Ci/m^3, between the Category 1 and 3 ^{137}Cs limits defined in the *Hanford Site Solids Waste Acceptance Criteria* (4.4E-3 and 1.2E+4 Ci/m^3, respectively).

Quality Requirements

Battelle—Pacific Northwest Division (PNWD) implemented the RPP-WTP quality requirements by performing work in accordance with the PNWD Waste Treatment Plant Support Project Quality Assurance Project Plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization and the approved test plan, TP-RPP-WTP-378, Rev. 0[(a)] and Test Exceptions 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. This work was performed to the quality requirements of NQA-1-1989 Part I, "Basic and Supplementary Requirements," NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev. 13, *Quality Assurance Requirements and Descriptions* (QARD). These quality requirements were implemented through PNWD's *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual*. The analytical requirements were implemented through WTPSP's Statement of Work (WTPSP-SOW-005) with the Radiochemical Processing Laboratory (RPL) Analytical Service Operations (ASO).

Experiments that were not method-specific were performed in accordance with PNWD's procedures QA-RPP-WTP-1101 "Scientific Investigations" and QA-RPP-WTP-1201 "Calibration Control System," verifying that sufficient data were taken with properly calibrated measuring and test equipment (M&TE) to obtain quality results.

BNI's QAPjP, PL-24590-QA00001, Rev. 0, was not applicable because the work was not performed in support of environmental/regulatory testing, and the data should not be used as such.

PNWD addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNWD's procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the test-plan objectives. This review procedure is part of PNWD's *WTPSP Quality Assurance Requirements and Description Manual*.

R&T Test Conditions

This report summarizes the spent resin preparation and analysis of spherical RF resin, Lot 5E-370/641.

Two 11-mL resin beds denoted as Resin A and Resin B were set up in a lead-lag format and subjected to three complete process cycles. Resin A was in the lead column for the first two process cycles. AP-101 simulant (1.47 L) was processed first, and both resin beds were individually eluted with 15 BVs 0.5 M nitric acid. AP-101 diluted to 5 M Na (1.53 L) was processed next, and the lead resin only (Resin A) was eluted with 15.6 BVs 0.5 M nitric acid. The lead and lag column positions were reversed such that Resin B was in the lead position. A 2.25-L aliquot of AN-102 with Sr/TRU components removed (per the WTP flowsheet) and diluted to 5 M Na was processed. Both the lead and lag columns were eluted with 30 BVs 0.5 M nitric acid.

(a) SK Fiskum. 2004. *Column Performance Testing of Actual Wastes from Tanks AP-101 and AN-102 Using Spherical Resorcinol Formaldehyde Ion Exchange Resin*, Battelle—Pacific Northwest Division, Richland, WA.

The resins were removed from the columns, then removed from the hot cell, and then processed per the test plan. All test conditions delineated by the test plan and test exceptions were met. A summary of test conditions is provided in Table S.5.

Table S.5. R&T Test-Condition Summary

R&T Test Condition	Discussion
Resins will be removed from the columns with deionized (DI) water, maintaining the resin beds separately from each other.	Resins were slurried out of the resin beds with water. Resins were collected in individual bottles and then removed from the hot cell. The dose rate from Resin B was 2.5 mR/h at contact and that from Resin A was <0.5 mR/h at contact.
The resin contact solution will be removed and analyzed by ion chromatography (IC) for anions.	The contact solution was removed and analyzed for anions and ^{137}Cs.
Resins will be dried to constant mass at 50^{o}C under vacuum.	The damp resin was flushed with nitrogen for 1 to 2 days to obtain a free-flowing form. The resins were then dried at 50^{o}C under vacuum until a mass change of <0.5% was obtained in a 7-h interval. This required a drying time of 13 days. Each dried resin sample weighed 2.92 g (2.87 g was originally packed into each column).
Resins will be analyzed for gamma-emitting isotopes.	Each dried resin sample was directly measured by gamma spectrometry in its entirety. Resin B contained 3.68 µCi/g ^{137}Cs; Resin A contained 0.352 µCi/g ^{137}Cs. Also detected at lower concentrations were ^{60}Co and ^{126}Sn/Sb.
Aliquots of resin will be acid digested and analyzed for total alpha, total beta, Pu, Am, Cm, metals, U, Cs, and Cs isotopics.	Duplicate 0.6-g aliquots from each resin bed were acid digested and analyzed by ICP-MS (^{133}Cs, ^{99}Tc, ^{85}Rb, and Cs isotopics), ICP-AES (RCRA metals, Na and Ni), KPA (U) and radiochemical methods (Pu, Am, Cm, total alpha, and total beta). Results are summarized in Table S2.

Simulant Use

Simulants were not used for this testing scope.

Discrepancies and Follow-on Tests

None.

1.0 Introduction

The U.S. Department of Energy (DOE) Hanford Project was the site of plutonium production during World War II and the cold war. Processing the spent nuclear fuel to recover the Pu (and other ancillary activities) resulted in large quantities of liquid wastes. The wastes are a complex mixture composed mostly of sodium nitrate, nitrite, hydroxide, and sulfate, along with a broad spectrum of minor and trace metals, organics, and radionuclides stored in underground storage tanks. Bechtel National Incorporated (BNI) was contracted by the DOE Office of River Protection (ORP) to build a processing plant, the River Protection Project-Hanford Tank Waste Treatment and Immobilization Plant (RPP-WTP), that will chemically separate the highly radioactive components (specifically Cs/^{137}Cs and, in some cases, Sr/^{90}Sr and transuranics [TRUs]) of the tank waste from the bulk (non-radioactive) constituents and immobilize the wastes by vitrification.

The RPP-WTP will produce two waste streams: a high-volume low-activity waste (LAW) that is ^{137}Cs (and ^{90}Sr and TRU) depleted and a low-volume high-activity waste (HLW) (the ^{137}Cs, ^{90}Sr, and TRU-rich fraction). The wastes will be separated into LAW and HLW fractions in the pretreatment modules of the plant.

The RPP-WTP contract statement of work specifies ion exchange for removing Cs (^{137}Cs) from tank waste supernatant to ultimately achieve a ^{137}Cs loading of 0.3 Ci/m^3 or less in the immobilized LAW product.[a] Further, the contract specifies that cesium ion exchange will use the elutable SuperLig® 644 (SL-644) resin (registered trademark of IBC Advanced Technologies, Inc.) or the DOE-approved equivalent. SL-644 is solely available through IBC Advanced Technologies. To provide an alternative to this sole-source resin supply, DOE-ORP directed BNI to initiate a three-stage process for selecting and potentially implementing an alternative ion exchange resin for cesium removal in the RPP-WTP.[b]

BNI completed the first step of this process with the recommendation that resorcinol formaldehyde (RF) resin be pursued as a potential alternative to SL-644.[c] The RF resin is an organic-based resin developed at Westinghouse Savannah River Company in the late 1980s. It was selected as an alternative cesium ion exchange technology for the Initial Pretreatment Module project, and extensive testing was performed to support that project during the late 1980s to early 1990s (Bray et al. 1996; Brown et al. 1995 and 1996; Kurath et al. 1994). Both batch and column testing of the ground-gel RF resin was conducted at Battelle—Pacific Northwest Division (PNWD) and the Savannah River National Laboratory. The resin was found to have a high loading and selectivity for cesium from Hanford Site tank wastes. The cesium could be eluted from the resin under acidic conditions.

BNI completed the second step of this process by developing an implementation plan.[d] BNI completed the first stage of the implementation plan with a spherical RF resin providing the best combination of

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[a] DOE Contract No. DE-AC27-01RV14136 (DOE 2000) Section C.7.d.1.iii.

[b] CCN 030290, Letter from C. B. Reid, ORP, to R. F. Naventi, BNI, dated March 13, 2002.

[c] R. Peterson, H. Babad, L. Bray, J. Carlson, F. Dunn, A. Pajunen, I. Papp, and J. Watson. 2002. *WTP Pretreatment Alternative Resin Selection* 24590-PTF-RPT-RT-02-001, Rev. 0, Bechtel National, Inc., Richland, WA.

[d] M. Thorson. 2002. *Alternative Ion Exchange Resin Supplemental Research and Technology Plan – Case 20,* 24590-PTF-PL-RT-02-002, Rev. 0, Bechtel National, Inc., Richland, WA.

characteristics required for WTP operations. The WTP Pretreatment Alternative Resin Selection report[a] reviewed available literature on RF performance relative to the WTP plant design and identified areas to target additional testing such as:

- Assess column chemical performance with Hanford-typical feeds.
- Address potential for resin packing induced by swelling in the plant column with resultant high pressure drops, potential resin structural breakdown, and potential channeling.
- Determine elution performance for resin to achieve desired decontamination factor and obtain data on cesium bleed from subsequent loading cycles.
- Determine concentration of residual metals, cesium, and other contaminants on spent resin as a function of the last elution conditions that may affect disposal requirements.

As part of the first stage of RF testing, spherical RF resin performance for Cs-removal was assessed using batch contact and column testing under the A-222 scoping statement (Fiskum et al. 2004b). The spherical resin was found to have good capacity, good kinetics, and better elution than the ground gel RF resin.

Based on these results, DOE-ORP directed BNI to initiate second-stage testing designed to evaluate RF resin for cold commissioning in the WTP.[b] Technical scoping statements A-204, A-212, and A-235 from these plans address testing of the spherical RF resin with actual waste samples from Hanford waste tanks AP-101 and AN-102, as well as analysis of the spent resin. Two resin beds were prepared in a lead-lag format to assess the AP-101 and AN-102 processing characteristics. The results of processing these wastes were previously reported (Fiskum et al. 2006a, 2006b). The AP-101 tank waste (one of the Envelope A wastes) was selected for testing because it bounded the K interference effect and was more representative of the anticipated commissioning waste feed. A high K concentration presents a challenging matrix for RF resin because it interferes with Cs exchange. The AN-102 tank waste feed was selected for testing as a representative of the Envelope C tank wastes. Actual waste testing with an Envelope B waste was not conducted; simulant testing with an Envelope B waste matrix, AZ-102, is currently under investigation as part of Technical Scoping Statement A-225.

This report summarizes the spent resin analysis following processing of AP-101 simulant, AP-101 actual tank waste, and AN-102 actual tank waste (Sr/TRU removed) in support of the second stage testing A-235 technical scoping statement. The residual Pu, Am, and Cm were measured to provide an initial assessment of the TRU waste classification. The residual Resource Conservation Recovery Act (RCRA) metals Ag, As, Ba, Cd, Cr, Pb, and Se were measured to provide an initial indication of potential land disposal issues. Residual Cs and Cs isotopics were measured to assess resin dose rate and associated limitations with respect to spent resin handling.

(a) Ibid. footnote (c).

(b) Schepens. 2004. CCN 083069, Letter from R. J. Schepens, ORP, to J. P. Henschel, BNI, “Direction to Perform Required Cesium Ion Exchange Alternative Resin Testing”, 03-WEC-006, effective date February 25, 2004.

2.0 Experimental

This section describes the spherical RF resin, process history, post-processing resin handling, and resin analysis.

2.1 Spherical RF Resin

The spherical RF resin used for actual waste testing was manufactured by Microbeads (Skedsmokorset, Norway) Lot Number 5E-370/641. This resin lot was produced on 5/24/05 in an 80-gal (hydrogen form) production batch. General manufacturing conditions of this resin are protected by patent and non-disclosure agreement; specific lot preparation conditions were forwarded under separate letter to the BNI Research and Technology (R&T) lead by Microbeads. A nominal 1.75-L sample of the resin lot was received at PNWD on 6/6/05. The resin was provided in the hydrogen form under water in a glass 2-L bottle. The gaseous headspace was minimal.

2.2 Resin Process History

The RF resin was prepared for column testing per the protocol P1-RF.(a) The protocol delineated a shrink-swell cycle in a beaker format followed by an in-column shrink-swell cycle. The ion exchange process system was previously described by Fiskum et al. (2006a). Two columns were prepared containing 8.0-mL wet H-form pretreated resin (dry H-form mass equal to 2.87 g). The columns were connected in a lead-lag format for simulant and actual waste processing. One resin was denoted Resin A and was placed in the lead position for the first process cycle. The other resin was denoted Resin B and was placed in the lag position for the first process cycle. When the positions of the lead and lag resins were changed, Resin B was placed in the lead position.

The resin beds were exposed to a total of three process cycles. AP-101 simulant processing was conducted as a shakedown test, verifying that all parts were working (Fiskum et al. 2006a). The system was transferred to the hot cell for actual waste testing with AP-101 diluted to 5 M Na, followed by AN-102 actual waste diluted to 5 M Na with Sr and TRUs removed (Fiskum et al. 2006b). A summary of the process history is provided in Table 2.1.

(a) C. A. Nash and C. E. Duffey. August 17, 2004. *Hanford RPP-WTP Alternate Resin Program - Protocol P1-RF: Spherical Resin Sampling from Containers, Resin Pretreatment, F-Factor, and Resin Loading to Column,* WTP doc. no. 097893.

Table 2.1. Resin-History and Total Cs Loading onto Resin A and Resin B

Tank Waste (Lead Resin)	Cycle	Feed Volume, L (BV)(a)	Cs Break-through, % C/C_o(b)	Cs on Resin A, Moles	Cs on Resin B, Moles	^{137}Cs on Resin A, Ci	^{137}Cs on Resin B, Ci
AP-101 sim. (Resin A)	1	1.46 (131)	55.0	5.55E-5	9.06E-6	1.55E-4	2.53E-5
AP-101 (Resin A)	2	1.53 (137)	93.4	4.25E-5	2.12E-5	1.13E-1	5.67E-2
AN-102 (Resin B)	3	2.25 (202)	10.2	3.07E-6	1.45E-4(c)	7.84E-3	3.71E-1(c)
Total Cs loaded on columns during multiple processing.				1.01E-4	1.75E-4	1.21E-1	4.28E-1

(a) One bed volume (BV) = 11 mL and was the expanded Na-form resin volume in 0.5 M NaOH.
(b) C/C_o = analyte concentration in column effluent divided by analyte concentration in feed.
The Cs breakthrough represents the relative Cs concentration in the final load sample from the lead column.
(c) Total Cs included the Cs from AP-101 breakthrough from Resin A and retained on Resin B.

Cycle 1. The entire ion exchange system was used for a full shakedown test with 1.46 L AP-101 simulant. The lead column Cs breakthrough, measured from ^{137}Cs tracer, was 55% C/C_o; the lag column Cs breakthrough reached 0.35% C/C_o. Both the lead and lag columns were individually eluted with 15 BVs of 0.5 M HNO_3. The final lead column eluate Cs concentration reached 2.1E-3 C/C_o (equivalent to 1.2E-2 µg Cs/mL); the final lag column eluate Cs concentration reached 4.7E-4 C/C_o (equivalent to 2.8E-3 µg Cs/mL). The resin beds were individually rinsed with DI water, and the system was transferred to a hot-cell facility. The resins were regenerated to the Na-form by passing 10.8 BVs of 0.5 M NaOH sequentially through the lead and lag columns.

Cycle 2. The ion exchange system was used to process 1.53 L of AP-101 tank waste (diluted to 5 M Na). The lead and lag column positions were not switched from the simulant processing positions; Resin A remained in the lead position. The lead column Cs breakthrough was 93.4% C/C_o; the lag column Cs breakthrough reached 5.0% C/C_o. After waste processing, feed displacement, and water rinse, only the lead column was eluted with 15 BVs 0.5 M HNO_3 to an eluate ^{137}Cs concentration C/C_o of 2.0 E-3 (equivalent to a ^{137}Cs concentration of 0.22 µCi/mL and 1.2E-2 µg/mL total Cs). The lead column was then rinsed with DI water. The lag column contained an estimated 5.7E+4 µCi ^{137}Cs (~3.0-mg total Cs) calculated from integrating the lead-column Cs breakthrough and subtracting the lag column Cs breakthrough.

Cycle 3. The lead column was regenerated with 6 BVs 0.5 M NaOH. The lead and lag column positions were switched such that Resin B was in the lead position. A 2.25 L portion of AN-102 was processed through the dual column system. The lead column Cs breakthrough was 10.2% C/C_o; the lag column Cs breakthrough remained constant at 9.8E-4% C/C_o (1.4E-3 µCi/mL and 7.8E-5 µg/mL total Cs). Both the lead and lag columns were eluted with 30 BVs 0.5 M HNO_3. The final lead column eluate ^{137}Cs concentration reached 1.3E-3 C/C_o (equivalent to a ^{137}Cs concentration of 1.9E-1 µCi/mL and 1.0E-2 µg/mL total Cs). The lag column eluate ^{137}Cs concentration reached 9.1E-5 C/C_o, which was equivalent to a ^{137}Cs concentration of 1.3E-2 µCi/mL and 7.2E-4 µg/mL total Cs. Each column was individually rinsed with 3 BVs of DI water.

The total Cs mole and ^{137}Cs Ci loading on the two resin beds during the three process cycles are summarized in Table 2.1. Resin B was loaded with 73% more total Cs and 3.5 times more ^{137}Cs than Resin A. Most of the Resin B loading occurred during the third process cycle.

2.3 Spent Resin Preparation

The RF resins were stored undisturbed for 81 days. The solution in and above each resin bed (~15 mL) was defined as the storage solution. Although a water rinse was conducted following elution with 0.5 M HNO_3, the storage solution was still slightly acidic (see Section 3.5). Resin bed heights were recorded to be 4.9 cm; within measurement uncertainty, the height was unchanged since last processing. A 0.5-cm black band was observed at the top surface of both resin beds.

The resin was handled according to TI-RPP-WTP-433, *Preparation of Spent Spherical RF Resin for Analysis*.[a] The resin beds were slurried out of each column into respective tared glass bottles. Additional water was added as required to effect the transfer. The storage solution mixed with the additional water was defined as the contact solution. An average of 28 mL of contact solution was used to complete the resin transfers. A small amount of resin slurry (~1 to 2 drops each sample) was lost to the cell floor during the transfer process. No attempt was made to recover this material as it would have become severely contaminated from contact with the cell floor.

The resin slurry dose rates were assessed to verify that they could be safely removed from the hot cell and contact handled. Resin A and B dose rates were <0.5 and 2.5 mR/h, respectively (open window at contact). The 30-cm dose rates were <0.5 mR/h for both resins.

The resins were transferred to clean Erlenmeyer flasks. The contact liquids were physically removed from the slurries with transfer pipets, and sub-samples were submitted for gamma energy analysis (GEA) and ion chromatography (IC) analysis. A nitrogen flow was established into and out of the flasks to perform initial resin drying. Once the resins were freely flowing, they were transferred to 20-mL vials and placed in a 50°C vacuum oven. The gross mass was recorded mornings and evenings until the net mass change was <0.5% over a 7-h period. The dry resin masses for both Resin A and Resin B were 2.92 g.

2.4 Resin Analysis

The resins were analyzed by the Analytical Services Organization (ASO) according to analytical services request (ASR) 7603. The ASO was responsible to prepare and analyze appropriate analytical batch and instrument quality control (QC) samples and to provide any additional processing to the sub-samples that might be required (e.g., acid digestion, radiochemical separations). The dried samples were processed and distributed to analytical workstations as shown in Figure 2.1.

(a) S. K. Fiskum. 2005. Battelle—Pacific Northwest Division.

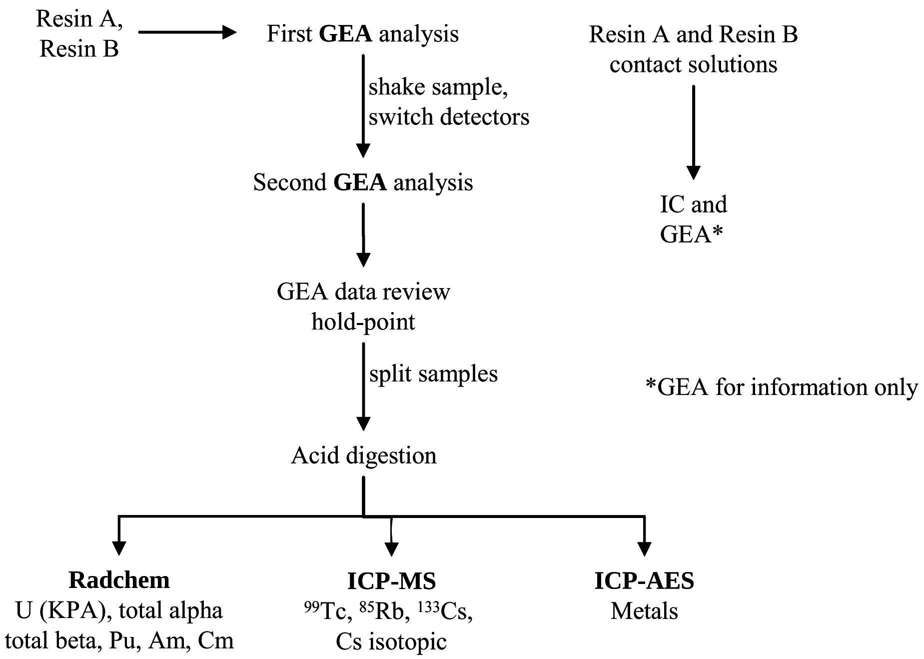

Figure 2.1. Spent Resin Analysis Processing

The following section describes the analytical methods. A summary of the ASO analytical procedures is provided in Table 2.2.

2.4.1 Gamma Spectrometry

The entire quantity of each dried resin was submitted for analysis by GEA. Dried spherical RF resin has been found to hold a static charge. Sample splits of the dried spent resin were not attempted because of the potential for too much sample loss (and spread of contamination) during handling from the electrostatic repulsion. Each sample was counted for 14 h twice, once from the side and once from the bottom with shaking in between. The long (14-h) count times were used in an attempt to discern concentrations of low-activity and low-energy gamma emitters.

The sample resin geometries did not correlate exactly to the calibrated detector geometries. The detectors were not calibrated for resin with the average sample bulk density of ~0.58-g/mL and ~5-mL volume. However, calibrations at the same detector positions with a volume of 2 mL and density of 1 g/mL were available. There were two corrections that needed to be considered for the data evaluation, and estimates of these corrections were determined using the Gammatool software package (Amersham International, Version 2.0 1997). The larger volume of the resin compared to the standard (~5 mL vs. 2 mL) reduces the counting efficiencies by a few percent. Conversely, the lower absorption of the resin compared to the standard (~0.58 g/mL vs. 1 g/mL) increases the counting efficiencies by a few percent. These two effects were of the same order of magnitude, but in the opposite direction, and tended to cancel out. Consequently, no correction was applied to the data. The good agreement between the two counts on different detectors (side-looking and up-looking) provided good confirmation of the technical approach.

Table 2.2. ASO Analysis Procedure Identification

Analyte	Procedure	Title
Gamma emitters	RPG-CMC-450, Rev. 0	*Gamma Energy Analysis (GEA) and Low-Energy Photon Spectrometry (LEPS)*
Acid digestion	RPG-CMC-128, Rev, 0	*HNO_3-HCl Acid Extraction of Liquids for Metal Analysis Using a Dry-Block Heater*
^{133}Cs, Cs isotopics, ^{85}Rb, and ^{99}Tc	329-OP-SCO1, Rev. 0	*Inductively-Coupled Plasma Mass Spectrometry (ICP/MS)*
Metals (ICP-AES)	RPG-CMC-211, Rev. 0	*Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry (ICPAES).*
U (kinetic phosphorescence analysis [KPA])	RPG-CMC-4017, Rev. 0	*Analysis of Environmental Water Samples for Actinides and Strontium-90*
	RPG-CMC-4014, Rev. 1	*Uranium by Kinetic Phosphorescence Analysis*
Pu, Am, and Cm	RPG-CMC-417, Rev. 0	*Separation of U, Am/Cm, and Pu and Actinide Screen by Extraction Chromatography*
	RPG-CMC-496, Rev. 0	*Precipitation Plating of Actinides for High-Resolution Alpha Spectrometry*
	RPG-CMC-422, Rev. 1	*Solutions Analysis: Alpha Spectrometry*
Total alpha and total beta	RPG-CMC-4001, Rev. 1	*Source Preparation for Gross Alpha and Gross Beta Analysis*
	RPG-CMC-408, Rev. 1	*Total Alpha and Beta Analysis*
Anions	PNL-ALO-212, Rev. 1	*Determination of Inorganic Anions by Ion Chromatography.*

2.4.2 Acid Digestion

The samples were delivered for wet chemical analysis. Duplicate ~0.6-g aliquots were taken from each resin sample. Another ~0.6-g Resin A sample aliquot was taken and spiked with metals for ICP-AES analysis. Another ~0.6-g Resin B sample aliquot was taken and spiked with Cs, Tc, and Rb for inductively coupled plasma-mass spectrometry (ICP-MS) analysis. The aliquots were placed into Teflon screwcap centrifuge tubes. Two process blanks were prepared for parallel processing with the samples. Concentrated nitric acid was added, and the samples were heated to ~100°C for several hours. The samples were evaporated to dryness and then brought to volume with 20-mL 0.5 M HNO_3.

A sample of pretreated resin (no ion exchange processing) was digested in parallel for ICP-AES analysis; this sample was identified as the matrix blank. The matrix blank was processed in a non-radiological fume hood; one additional blank was prepared with the matrix blank.

A second acid digestion preparation was conducted to prepare blank spikes (BSs) of Rb and Cs. The first preparation resulted in BS failures because the spiked amount was too low for ICP-MS detection.

2.4.3 Inorganic Analysis

Anions in the contact solutions were directly analyzed using a Dionix ICS-2500 IC system equipped with a conductivity detector.

The ICP-AES analysis was conducted on the acid-digested and diluted sample aliquots using a Thermo Jarrell-Ash atomic emission spectrometer.

The ICP-MS analysis was conducted using a PQ-Excell (Thermo Elemental) to determine ^{133}Cs, ^{85}Rb, and ^{99}Tc from the acid-digested and diluted sample aliquots. BS recovery was calculated by subtracting the average blank concentrations. The Cs isotopic ratio was determined separately using an ion chromatograph equipped with a cation separator column before introducing samples to the ICP-MS. Relative Cs mass ratios from the isolated Cs fraction were determined by MS. These ratios were normalized to the measured ^{133}Cs and summed to determine total Cs concentration.

Uranium was determined on the acid-digested sample aliquots following purification by anion exchange using a Chemchek Instruments KPA.

2.4.4 Radiochemical Analysis

Total alpha was determined by evaporating sample aliquots onto counting planchets and counting with ZnS scintillation detectors. High solids content in samples mounted for total alpha may bias results low. Isotopes of ^{238}Pu, $^{239+240}$Pu, ^{241}Am, ^{242}Cm, and $^{243+244}$Cm in the acid-digested and diluted sample aliquots were determined by alpha spectrometry, after radiochemical separations. The sum of alpha was simply the summation of measured specific alpha-emitters and provided an alternate measure of the total alpha activity free of the mass loading bias. However, the alpha sum did not account for alpha activity associated with U. Total beta samples were similarly prepared and counted with gas flow proportional counters calibrated with ^{90}Sr/Y. The sum of beta was simply the summation of measured specific beta-emitters and provided an alternate measure of the total beta activity.

3.0 Results and Discussion

This section describes results from the spent resin and contact fluid analysis.

3.1 Analysis Results

The ICP-AES, ICP-MS, and KPA analytical results for both Resin A and Resin B are summarized in Table 3.1. The average results and relative percent differences (RPDs) for analytes greater than the estimated quantitation limit (EQL) are provided. The resin results were conservatively reported by not incorporating a process blank or matrix blank subtraction. The matrix blank (ICP-AES analysis only) and preparative blank results are shown for better resin analytical data evaluation, especially with regard to low-level analyte concentrations. In addition to the analytes of interest specified in the test plan, the analytes measured opportunistically by the ICP-AES are provided for information only as they have not been fully evaluated relative to QC.

Table 3.1. Metals Analysis Summary for Spent Spherical RF Resin

		Process Blank		**Matrix Blank**		**Resin A$^{(a)}$**		**Resin B$^{(a)}$**	
Analyte	**Analysis Method**	**Average µg/g$^{(b)}$**	**RPD %**	**Average µg/g$^{(b)}$**	**RPD %**	**Average µg/g$^{(b)}$**	**RPD %**	**Average µg/g$^{(b)}$**	**RPD %**
Analytes of Interest									
Ag	ICP-AES	<1.6	--	<0.54	--	[3.1]	--	[3.4]	--
As	ICP-AES	<6.3	--	<2.2	--	<6.4	--	[6.9]	--
Ba	ICP-AES	[0.73]	--	[0.41]	--	[1.5]	--	[1.0]	--
Cd	ICP-AES	[0.55]	--	<0.16	--	[0.58]	--	[0.69]	--
Cr	ICP-AES	[0.92]	--	[0.30]	--	338	2.1	337	1.5
^{133}Cs	ICP-MS	[4.1E-3]	--	na	na	0.0142	7.7	0.118	0
^{135}Cs	ICP-MS	na	na	na	na	0.00248$^{(c)}$	0.46	0.0303$^{(c)}$	1.03
^{137}Cs	ICP-MS	na	na	na	na	0.00371$^{(c)}$	1.14	0.0391$^{(c)}$	0.33
Cs(total)	ICP-MS	na	na	na	na	0.0204$^{(c)}$	5.1	0.187$^{(c)}$	0.10
Na	ICP-AES	[46]	--	301	5.7	[68]	--	[160]	--
Ni	ICP-AES	<1.1	--	[0.47]	--	26.1	1.2	[9.7]	--
Pb	ICP-AES	<7.0	--	<2.4	--	[9.5]	--	[8.6]	--
Se	ICP-AES	[8.2]	--	[3.0]	--	[8.8]	--	[9.5]	--
^{99}Tc	ICP-MS	<9.2E-3	--	na	na	18.9	1.6	19.4	1.6
Th	ICP-AES	<75	--	<26	--	<75	--	<74	--
U	ICP-AES	<210	--	<71	--	<207	--	<205	--
U	KPA	0.34	9.1	na	na	193	14.5	101	5.9
Opportunistic Analytes$^{(d)}$									
Al	ICP-AES	[78]	--	<3.6	--	[19]	--	[28]	--
B	ICP-AES	<1.4	--	15.7	3.2	17.0	2.4	22.2	2.3
Be	ICP-AES	<0.038	--	<0.01	--	<0.039	--	<0.038	--
Bi	ICP-AES	[6.4]	--	[2.7]	--	[10]	--	[8.5]	--

Table 3.1 (Contd)

		Process Blank		Matrix Blank		Resin A(a)		Resin B(a)	
Analyte	**Analysis Method**	**Average µg/g(b)**	**RPD %**	**Average µg/g(b)**	**RPD %**	**Average µg/g(b)**	**RPD %**	**Average µg/g(b)**	**RPD %**
Ca	ICP-AES	26	124	[6.7]	--	70.2	65	[32]	--
Ce	ICP-AES	<13	--	<4.6	--	<13	--	<13	--
Co	ICP-AES	<1.1	--	<0.39	--	[7.4]	--	[2.6]	--
Cu	ICP-AES	<2.9	--	[1.2]	--	70.0	1.1	29.6	11
Dy	ICP-AES	<2.0	--	<0.69	--	<2.0	--	<2.0	--
Eu	ICP-AES	<0.6	--	<0.22	--	<0.6	--	[0.65]	--
Fe	ICP-AES	17	48	[0.90]	--	[6.9]	--	[9.2]	--
K	ICP-AES	<340	--	<120	--	<350	--	<340	--
La	ICP-AES	<2.8	--	<0.96	--	<2.8	--	<2.8	--
Li	ICP-AES	<2.1	--	<0.71	--	<2.1	--	<2.0	--
Mg	ICP-AES	219	51	<1.8	--	<5.3	--	<5.2	--
Mn	ICP-AES	[0.37]	--	[0.08]	--	[0.29]	--	[0.33]	--
Mo	ICP-AES	[1.3]	--	[0.43]	--	[8.7]	--	16	5.6
Nd	ICP-AES	<13	--	<4.4	--	<13	--	<13	--
P	ICP-AES	[19]	--	[2.8]	--	[12]	--	[20]	--
Pd	ICP-AES	<35	--	<12	--	<35	--	<35	--
^{85}Rb	ICP-MS	[6.9E-3]	--	na	na	[5.0E-3]	--	[5.8E-3]	--
Rh	ICP-AES	<16	--	<5.5	--	<16	--	<16	--
Ru	ICP-AES	<4.1	--	<1.4	--	<4.2	--	[4.5]	--
Sb	ICP-AES	[10]	--	[3.3]	--	[8.6]	--	[8.9]	--
Si	ICP-AES	<11	--	<3.6	--	<11	--	<11	--
Sn	ICP-AES	<44	--	<15	--	[92]	--	[69]	--
Sr	ICP-AES	[0.29]	--	[0.18]	--	[0.44]	--	[0.57]	--
Te	ICP-AES	[9.60]	--	[3.4]	--	<9.0	--	[10]	--
Ti	ICP-AES	<0.62	--	<0.21	--	<0.6	--	<0.6	--
Tl	ICP-AES	<5.7	--	<2.0	--	<5.7	--	<5.7	--
V	ICP-AES	<1.2	--	<0.41	--	<1.2	--	<1.2	--
W	ICP-AES	<3.8	--	<1.3	--	[22]	--	41	7.6
Y	ICP-AES	<0.54	--	<0.18	--	<0.54	--	<0.54	--
Zn	ICP-AES	250	51	2.27	7.5	5.95	16.1	[4.4]	--
Zr	ICP-AES	<1.5	--	<0.52	--	[11]	--	[3.8]	--

(a) Results are presented on a dry resin mass basis.

(b) Bracketed results were greater than the method detection limit (MDL) but less than the EQL; RPDs are not reported for the bracketed results. The less-than values (<) indicate that the results were less than the MDL: reported values are the instrument detection limits multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.

(c) Based on ^{133}Cs analysis and isotopic analysis (provided in Table 3.7).

(d) Opportunistic analytes are reported as a courtesy. Results were not fully evaluated relative to QC criteria.

na = not applicable, not analyzed

Data are from ASR 7603.

The Cs, Cr, Ni, Tc, and U had concentrated to varying extents on the resin. Of the detected analytes, Cr was the highest at 338 μg/g. Cr also represented the highest analyte concentration on H-form SL-644 resin at 3990 μg/g, over 10× the concentration found with RF (Fiskum et al. 2004c). The Ba, Cd, Na, and Se analyte concentrations were less than the EQL and were also measured in the blank. Therefore, their concentrations on the resins should be considered a worst-case or bounding condition. The Ag and Pb analytes were measured in the resin below the EQL; they were undetected in the blank. Evaluating the opportunistic analytes showed that Cu, Mo, Sn, and W concentrations were elevated in the spent resin.

The total metals analysis should not be confused with metals solubilized during a toxicity characteristics leach procedure (TCLP) (EPA SW-846, Method 1311). The TCLP and associated analyses were not included in this scope of work because of the limitations in resin sample size. The resin sample sizes were small (2.9 g) to accommodate the safe handling of actual waste while generating useful ion exchange process data.(a) If a toxicity characteristic metal found in Resin A and/or Resin B were successfully solubilized in a TCLP and measured per the EPA methodology, then the measured analyte could be compared to the regulatory threshold. A comparison of analytical values to regulatory thresholds is provided in Table 3.2. A 20× concentration was applied to the regulatory threshold of each analyte converting from the liquid extraction phase concentration to the total metal solid phase concentration. For both resin beds, the Cr concentration apparently exceeded the total metal threshold. All other measured toxic metals appeared to be well below the total metal threshold.

Table 3.2. Projected Worst-Case Toxicity Characteristic

Toxic Metal	Threshold		Resin A		Resin B	
	Regulatory, mg/L TCLP	Total Metal, μg/g (a)	Analyte Conc., μg/g(b, c)	Ratio Analyte to Total Metal Threshold	Analyte Conc., μg/g(b, c)	Ratio Analyte to Total Metal Threshold
Ag	5.0	100	[3.1]	0.031	[3.4]	0.034
As	5.0	100	<6.4	<0.064	[6.9]	0.069
Ba	100	2000	[1.5]	0.00073	[1.0]	0.00048
Cd	1.0	20	[0.58]	0.029	[0.69]	0.034
Cr	5.0	100	338	**3.38**	337	**3.37**
Pb	5.0	100	[9.5]	0.095	[8.6]	0.086
Se	1.0	20	[8.8]	0.44	[9.5]	0.48

(a) Bounding condition analyte concentration in the resin if the entire quantity were solubilized during a TCLP test (20× concentration from the TCLP extract).

(b) Results are presented on a dry-resin-mass basis.

(c) Bracketed results were greater than the MDL but less than the EQL. The less-than values (<) indicate that the results were less than the MDL; reported values were the instrument detection limits multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.

Notes: Bolded and shaded values indicate potential exceedance of regulatory threshold.

The analyte to total metal threshold ratios in this table may be compared to the previously reported ratios for SL-644 generated by using equivalent assumptions (Fiskum et al. 2004c, Table 4.8).

(a) A RCRA-metals analysis following TCLP is planned under Technical Scoping Statement A-205 using spiked simulant.
CA Nash. 2006. *Batch Contact and Column Testing of Spherical Resorcinol Formaldehyde* WSRC-RP-2004-00565, Rev. 1, Westinghouse Savannah River Company, Aiken, SC.

These data showed that spherical RF contained lower RCRA total metal concentrations on the spent resin than were found with SL-644. Characterization reported by Fiskum et al. (2004c) showed that SL-644 contained significant quantities of Ag (potentially near the threshold), Cr (20 to 40× threshold), and Pb (1.7× threshold) held by the resin (where threshold assumes that all of the analyte is solubilized in the TCLP procedure). The SL-644, however, was exposed to eight process cycles, seven of which were actual tank waste, which was significantly more waste contact than the RF test. Bounding test conditions using simulants (Arm et al. 2003) showed that the SL-644 would still pass the land disposal restrictions (LDRs) because these elements were not sufficiently solubilized during a TCLP test modified to support a small sample size.

Results from the radioisotopic characterization are listed in Table 3.3. The major radioisotopic constituent was ^{137}Cs, averaging 0.352 µCi/g for Resin A and 3.68 µCi/g for Resin B, nearly a factor of 10 difference between the resin beds. The total quantity loaded onto these resins during the last process cycle differed by a factor of 47 (Resin B was loaded with 47× more ^{137}Cs than Resin A).

The ^{137}Cs determined by ICP-MS agreed within 8% of the GEA determination (converted from µCi to µg basis). The 8% difference was within the reported uncertainties. The total beta activity measured on Resin A (0.66 µCi/g) was approximately equal to the sum of ^{137}Cs, ^{60}Co, and ^{99}Tc concentrations (0.75 µCi/g), indicating that all beta-activity sources were identified.[a] The total beta activity measured on Resin B (3.69 µCi/g) was again approximately equal to the sum of identified beta emitters, ^{60}Co, ^{99}Tc, and ^{137}Cs (4.0 µCi/g), indicating the total beta activity source(s) were identified. The ~10% difference in the sum of beta emitters and the total beta determination may be attributed to the variation in counting efficiency for the isotopes on the gas-flow proportional counter (^{99}Tc has a lower counting efficiency than the calibration standard ^{90}Sr/Y).

The ^{99}Tc concentrations on Resins A and B were virtually identical. This indicated that the resin beds were probably equally saturated with Tc under the process conditions. Tc is anticipated to exist as an anion in the caustic wastes (Lavrukhina and Pozdnyakov 1970) and was thus not expected to exchange onto the cation exchange resin. Although the specific Tc complexation in these wastes was not known, a significant fraction (~50%) of the AN-102 Tc was expected to be present as Tc(IV) (Burgeson et al. 2004a). Each resin bed retained ~0.5% of the total AN-102 feed Tc.[b]

Similarly, Cr concentrations on the two resins were virtually identical and relatively high at 340 µg/g. Like Tc, Cr is expected to exist largely as an anionic complex in tank waste (CrO_4^{2-}) and is not expected to exchange onto RF resin. The mechanism for Cr retention on the resin may be similar to that of the Tc retention.

The sum of alpha-activity measurements was higher than the total alpha-activity measurements (26% for Resin A and 11% for Resin B). Two reasons may have contributed to this discrepancy: 1) the total alpha activity may have been biased low by solids loading on the counting planchet (alpha absorption effects), and 2) the total alpha measurement uncertainty was high at 28% and 40%, and the sum of alpha activity measurements were within these uncertainties. Resin A resulted in about a factor of 2 higher Pu

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(a) Total beta sources were measured as dry preparations using gas-flow proportional counting. Thus, volatile and low-energy beta sources are excluded from this comparison.

(b) The total Tc in the AN-102 feed was calculated from the Urie-reported (2002) value (0.148 µCi/mL) and the 1.9× dilution factor.

Table 3.3. Radioisotopic Analysis of Spent Resin

Analyte	Analysis Method	Resin A(a)			Resin B(a)		
		Average µCi/g	2-σ error %	RPD %	Average µCi/g	2-σ error %	RPD %
^{60}Co	GEA	7.87E-2	6	5	1.96E-2	6	6
^{99}Tc	ICP-MS	3.22E-1	15	1.6	3.29E-1	15	1.6
^{106}Ru/Rh	GEA	<1E-3	--	--	<3E-3	--	--
^{125}Sb	GEA	<6E-4	--	--	<2E-3	--	--
^{126}Sn/Sb	GEA	3.58E-4	26	6	<8E-4	--	--
^{134}Cs	GEA	<1E-4	--	--	<8E-5	--	--
^{137}Cs	GEA	3.52E-1	8	5	3.68	8	4
^{152}Eu	GEA	<9E-5	--	--	<7E-5	--	--
^{154}Eu	GEA	<2E-4	--	--	<1E-4	--	--
^{155}Eu	GEA	<4E-4	--	--	<3E-3	--	--
^{226}Ra	GEA	<3E-4	--	--	<1E-3	--	--
^{231}Pa	GEA	<5E-3	--	--	<1E-2	--	--
^{241}Am	Separations and α spec.	2.72E-4	14	21	2.41E-4	16	6
^{238}Pu	Separations and α spec.	3.61E-4	12	2	1.69E-4	24	12
$^{239+240}$Pu	Separations and α spec.	1.72E-3	6	7	6.21E-4	12	14
^{242}Cm	Separations and α spec.	<1E-5	--	--	<1E-5	--	--
$^{243+244}$Cm	Separations and α spec.	<2E-5	--	--	<2E-5	--	--
Sum of alpha	Separations and α spec.	2.35E-3	6	8	1.04E-3	8	14
Total alpha	Scintillation count	1.86E-3	28	2	9.32E-4	40	14
Sum of beta	GEA + ^{99}Tc	7.53E-1	8	3	4.02E+0	8	4
Total beta	Gas-flow prop. count	6.60E-1	8	6	3.70E+0	8	4

(a) Results are presented on a dry-resin mass basis.
Data are from ASR 7603. Reference date is 8/15/05.

concentration than Resin B; Am was generally equally distributed between the two resins. Overall, the measured alpha-specific radionuclides (Am and Pu) appeared to account for all alpha activity in the samples. The U contribution to the total alpha activity was evaluated based on the AN-102 U isotopic ratios reported by Urie et al. (2002) in the wet centrifuged solids and the measured U values reported in Table 3.1.(a) The U was calculated to contribute ~13% of the reported total alpha activity.

(a) The applied U isotopic ratio was 0.0088% ^{233}U, 0.7% ^{235}U, 0.029% ^{236}U, and 99.3% ^{238}U; U isotopics was not reported for the supernatant.

3.2 Total Metal and Radionuclide Recoveries

The fractions of the AN-102 and AP-101 (actual and simulated waste) feed analytes found on the spent resins are provided in Table 3.4 and Table 3.5.(a) The analyte fractions are provided based on the following: 1) total analyte in the AN-102 feed; 2) total analyte in the combined AN-102 and AP-101 (actual waste) feeds; and 3) total analyte in the combined AN-102, AP-101 actual, and AP-101 simulant waste feeds.(b)

The highest recovered analyte was U at 3.1 and 1.6% of the AN-102 feed U mass (Resin A and B, respectively). Chromium represented the second highest recovered analyte on the RF resin at 1.3% (both resins) of the AN-102 feed Cr mass. Note that the Cr fractions on both resins were equivalent, indicating that the Cr may have reached a saturation concentration on the resin. This observation was also supported by the fact that the actual concentrations of Cr reported in Table 3.1 were nearly identical. Approximately 1% of the feed Ag concentrated on the RF (the Ag result had >15% uncertainty). All other feed analytes were present at <1% on the spent resin.

3.3 TRU Waste Considerations

In both cases, the total alpha activities on the resin beds (1.0 and 2.4 nCi/g) were << 100 nCi/g threshold defining TRU waste. The current test should not be considered definitive for TRU waste characterization purposes. The AN-102 is a high-complexant waste and contained a fairly high Pu concentration even after Sr/TRU precipitation, 7.6E-4 µCi/mL Pu (Fiskum et al. 2006b). The presence of complexants in the last waste processed could potentially affect Pu distribution on the resin. However, Pu fractionation from AP-101 processing indicated that the spherical RF resin had little affinity for Pu where ~9% of the $^{239+240}$Pu was recovered in the eluate (Fiskum et al. 2006a). In contrast, the AP-101 processing with SL-644 resulted in 72% of the Pu recovery in the eluate fraction (Fiskum et al. 2004a), demonstrating SL-644 affinity for Pu under the same load process conditions. Table 3.6 compares the RF and SL-644 final process parameters and spent resin TRU results. A Pu elution profile from SL-644 would need to be generated to determine if the additional 14 BVs of eluate would remove the excess Pu from Column 2 to reach equivalency with Column 1.

(a) It should be noted that the final analyte concentrations on the spent resins will be a complex function of the analyte concentrations in the feeds, the analyte ion exchange equilibrium and rate characteristics, and the analyte speciation.

(b) The total feed mass basis is provided in Appendix B, along with the fractionation of the various analytes into the effluent and eluate process streams.

Table 3.4. Total Analyte Quantities Recovered on the Eluted Resins and Relative Analyte Fractions from Three Processed Wastes

Analyte(a)	Resin A Analyte Recoveries				Resin B Analyte Recoveries			
	Total recovered	Relative to AN-102	Relative to AN-102 + AP-101 Actual	Relative to AN-102 + AP-101 Actual + Simulant	Total recovered	Relative to AN-102	Relative to AN-102 + AP-101 Actual	Relative to AN-102 + AP-101 Actual + Simulant
	µg	%	%	%	µg	%	%	%
Ag	[9.1]	[0.93]	[0.52]	[0.52]	[9.8]	[1.01]	[0.56]	[0.56]
As	<19	<0.48	<0.29	<0.29	[20]	[>0.52]	[>0.31]	[>0.31]
Ba	[4.2]	[0.54]	[0.21]	[0.17]	[2.8]	[0.356]	[0.141]	[0.11]
Cd	[1.7]	[0.0030]	[0.0028]	[0.0027]	[2.0]	[0.0035]	[0.0034]	[0.0033]
Cr	986	1.32	0.327	0.192	983	1.32	0.326	0.192
K	<1008	<0.043	<0.0022	<0.0012	<998	<0.043	<0.0022	<0.0012
Mn	[0.85]	[0.080]	[0.071]	[0.063]	[0.96]	[0.090]	[0.080]	[0.071]
Na	[197]	[7.7E-5]	[4.5E-5]	[3.3E-5]	[453]	[1.8E-4]	[1.0E-4]	[7.5E-5]
Ni	76.1	0.019	0.019	0.019	[28]	[0.0072]	[0.0071]	[0.0071]
Pb	[28]	[0.022]	[0.019]	[0.017]	[25]	[0.020]	[0.017]	[0.016]
Se	[26]	[0.46]	[0.27]	[0.27]	[28]	[0.50]	[0.29]	[0.29]
Sr	[1.3]	[5.0E-4]	[5.0E-4]	[4.9E-4]	[1.7]	[6.4E-4]	[6.4E-4]	[6.4E-4]
Th	<220	na	na	na	<218	na	na	na
U KPA	564	3.11	0.68	0.68	295	1.63	0.354	0.354
Rb-85	[1.5E-2]	[4.1E-4]	[1.8E-4]	[1.1E-4]	[1.7E-2]	[4.8E-4]	[2.1E-4]	[1.3E-4]
Cs-133	0.0415	3.77E-4	2.50E-4	1.65E-4	0.345	3.13E-3	2.08E-3	1.37E-3
Opportunistic Analytes								
Al	[55]	[0.0010]	[3.4E-4]	[2.2E-4]	[80]	[0.0014]	[5.0E-4]	[3.1E-4]
B	49.6	0.105	0.074	0.057	64.7	0.136	0.096	0.074
Be	<0.11	<0.478	<0.007	<0.007	<0.11	<0.47	<0.0069	<0.0067
Bi	[29]	[0.56]	[0.29]	[0.29]	[25]	[0.48]	[0.25]	[0.25]
Ca	205	0.078	0.073	0.071	93.4	0.036	0.033	0.032
Co	[21]	[0.49]	[0.40]	[0.40]	[7.4]	[0.17]	[0.14]	[0.14]
Cu	204	0.85	0.70	0.67	86.4	0.361	0.296	0.282
Dy	<5.9	<0.48	<0.26	<0.26	<5.8	<0.47	<0.25	<0.25
Eu	<1.9	<0.31	<0.17	<0.17	[1.9]	[0.32]	[0.17]	[0.17]

Table 3.4 (Contd)

Analyte(a)	Resin A Analyte Recoveries				Resin B Analyte Recoveries			
	Total recovered	**Relative to AN-102**	**Relative to AN-102 + AP-101 Actual**	**Relative to AN-102 + AP-101 Actual + Simulant**	**Total µg recovered**	**Relative to AN-102**	**Relative to AN-102 + AP-101 Actual**	**Relative to AN-102 + AP-101 Actual + Simulant**
	µg	**%**	**%**	**%**	**µg**	**%**	**%**	**%**
Fe	[20]	[0.27]	[0.14]	[0.11]	[27]	[0.35]	[0.18]	[0.14]
La	<8.2	<0.23	<0.16	<0.16	<8.1	<0.23	<0.16	<0.16
Li	<6.0	<0.26	<0.13	<0.11	<6.0	<0.26	<0.13	<0.11
Mg	<15	na	na	na	<15.3	na	na	na
Mo	[25]	[0.050]	[0.035]	[0.028]	46.6	0.092	0.065	0.051
Nd	<38	<0.23	<0.15	<0.15	<38	<0.23	<0.15	<0.15
P	[35]	[0.0020]	[0.0016]	[0.0013]	[58]	[0.0034]	[0.0026]	[0.0021]
Pd	<102	<0.34	<0.17	<0.17	<102	<0.33	<0.17	<0.17
Rh	<47	<0.27	<0.16	<0.16	<47	<0.27	<0.16	<0.16
Ru	<12	<0.04	<0.03	<0.03	[13]	[0.046]	[0.037]	[0.037]
Sb	[25]	[0.53]	[0.33]	[0.33]	[26]	[0.55]	[0.34]	[0.34]
Si	<31	<0.02	<0.01	<0.01	<31	<0.017	<0.009	<0.006
Sn	[267]	[0.82]	[0.17]	[0.17]	[200]	[0.61]	[0.13]	[0.13]
Te	<26	<0.23	<0.14	<0.14	[29]	[0.26]	[0.15]	[0.15]
Ti	<1.8	<0.40	<0.21	<0.21	<1.8	<0.40	<0.21	<0.21
Tl	<17	na	na	na	<17	na	na	na
V	<3.5	<0.29	<0.19	<0.19	<3.4	<0.29	<0.18	<0.18
W	[63]	[0.045]	[0.036]	[0.030]	120	0.087	0.068	0.057
Y	<1.6	<0.28	<0.20	<0.20	<1.6	<0.28	<0.20	<0.20
Zn	17.4	0.35	0.13	0.07	[13]	[0.25]	[0.10]	[0.051]
Zr	[31]	[1.1]	[0.45]	[0.41]	[11]	[0.41]	[0.16]	[0.14]

(a) Assumes the feed "less-than" values were actual analyte concentrations (bounding condition).

Notes: Bracketed results were greater than the MDL but less than the EQL. The less-than values (<) indicate that the results were less than the MDL: reported values were the instrument detection limits multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.

"na" indicates all feed values and resin values were less than the instrument detection limit.

Table 3.5. Total Analyte Quantities Recovered and Relative Influence from Two Processed Wastes

Analyte	Resin A Analyte Recoveries				Resin B Analyte Recoveries			
	Total recovered	Relative to AN-102	Relative to AN-102 + AP-101 Actual	Relative to AN-102 + AP-101 Actual + Simulant	Total recovered	Relative to AN-102	Relative to AN-102 + AP-101 Actual	Relative to AN-102 + AP-101 Actual + Simulant
	μCi	%	%		μCi	%	%	
^{60}Co	2.30E-1	0.587	0.525	na	5.72E-2	0.146	0.131	na
^{137}Cs	1.03	3.09E-4	2.01E-4	na	10.7	3.23E-3	2.10E-3	na
^{238}Pu	1.05E-3	0.235	0.203	na	4.93E-4	0.110	0.095	na
$^{239+240}$Pu	5.02E-3	0.398	0.355	na	1.81E-3	0.144	0.128	na
^{241}Am	7.94E-4	0.00328	0.00325	na	7.04E-4	2.91E-3	2.88E-3	na
^{242}Cm	<3.E-5	<3.E-2	<3.E-2	na	<3.E-5	<3.E-2	<3.E-2	na
$^{243+244}$Cm	<6.E-5	<5.E-3	<5.E-3	na	<6.E-5	<5.E-3	<5.E-3	na
Total alpha	5.43E-3	0.0289	0.0277	na	2.72E-3	0.0145	0.0139	na
Sum of alpha	6.89E-3	0.0254	0.0250	na	3.04E-3	0.0112	0.0110	na

Reference date is 8/15/05.

Table 3.6. Final Process Cycle Comparison for RF and SL-644 and Spent Resin TRU Concentrations

Parameter for Last Process Cycle	Spherical RF Resin A	 Resin B	SL-644[(a)] Column 1	 Column 2
Position	lag	lead	lead	lag
Tank waste feed	AN-102		AP-104/SY-101	
TOC in waste feed,[(b)] M	1.3[(c)]		0.35	
Pu in waste feed, μCi/mL	7.66E-4		2.95E-4	
Waste feed volume, BV	202		182	
Elution volume, BV	29	30	30	16
Eluate TRU, nCi/g	8.7[(d)]	14[(d)]	164[(e)]	not measured
Regenerated and rinsed?	no	no	yes	no
Spent resin form	H-form	H-form	Na-form	H-form
TRU concentration in spent resin,[(f)] nCi/g	2.4	1.0	11	103

(a) Data reported by Fiskum et al. 2004c.

(b) The total organic carbon (TOC) is an indicator of the potential complexant concentration.

(c) Not measured, calculated from AN-102 analysis (Urie et al. 2002) hot persulfate method, 1.9× dilution factor, and assumed no organic destruction during Sr/TRU precipitation.

(d) Eluate TRU concentration is the sum of alpha emitter concentrations, multiplied by composited volumes, and divided by 2.92 g dry H-form resin.

(e) Eluate TRU concentration in SL-644 is the sum of alpha emitters (1.81 E-3 μCi/mL) multiplied by composited volume (200 mL) divided by 2.20 g dry Na-form resin mass (Fiskum et al. 2004c).

(f) Sum of Pu, Am, and Cm concentrations.

The total TRU concentrations in the RF resin processing eluates were measured and normalized to the spent resin mass and are provided in Table 3.6. The eluates associated with AN-102 processing showed that TRU concentrations equivalent to 8.7 and 14 nCi/g (for Resin A and B, respectively) were exchanged onto, and eluted from, the resin. These values, in combination with the residual spent resin TRU values, were well below the TRU waste classification threshold. The SL-644 Column 1 eluate following AP-104/ SY-101 processing was similarly evaluated and found to be equivalent to 164 nCi/g, greater than the TRU threshold. The Resin A eluate associated with AP-101 processing through RF resin resulted in an equivalent TRU loading of 7.9 nCi/g (corresponding to ~9% Pu exchange onto the resin). The similarity of exchanged TRU concentrations between AP-101 and AN-102 indicated that the complexant concentration in the feed did not have a large effect on TRU exchange characteristics. In both cases, the combined TRU component from the eluate and the TRU component on the spent resin remained well below the TRU threshold. Extended RF resin elution was not required to reduce the TRU concentrations below the TRU waste classification limit; extended SL-644 resin elution may be critical to reducing TRU concentrations below the TRU waste classification limit.

3.4 Cs Isotopic Distribution

Table 3.7 presents the Cs isotopic distributions for each resin bed and the last waste processed (AN 102) (Fiskum et al. 2006b). The isotopic distributions were different between the two resin beds and the AN-102 waste. Resin A had a higher ^{133}Cs fraction than Resin B. Both resin beds had a higher ^{133}Cs fraction than AN-102. This indicated that ^{133}Cs from the front-end processing still contributed a percentage of the residual Cs on the resin even after processing two additional cycles. The SL-644 isotopic distribution from "Column 1" is also presented for comparison; it was heavily influenced by the first cycle Cs processing even after seven additional waste cycles were processed (Fiskum et al. 2004c).

Table 3.7. Cs Isotopic Distribution in Resin A and B

	Mass Abundance			
Isotope	**AN-102 Feed**	**Resin A**	**Resin B**	**SL-644**[(a)]
^{133}Cs	0.616	0.697	0.629	0.784
^{135}Cs	0.170	0.121	0.162	0.094
^{137}Cs	0.214	0.182	0.209	0.122

(a) Column 1 results after one simulant and seven actual tank waste process cycles.

The higher abundance of stable Cs (^{133}Cs) relative to the last waste matrix processed appeared to be a holdover from initial processing with AP-101 simulant, the first waste matrix processed. The three tank waste feeds were evaluated for their relative influence on the spent resin Cs isotopic compositions. Weighting factors[(a)] were applied to the feeds in an iterative process to obtain a match to the measured isotopic fractionation in the resins. The weighting factors for each processed waste and associated Cs contribution on the spent resins are summarized in Table 3.8.

Table 3.8. Cs Source Contribution in Spent Resin A and B

		Weighting Factor, %	
Source	**Cycle**	**Resin A**	**Resin B**
AP-101 simulant	1	20.5	3.3
AP-101 actual waste	2	79.5	18.0
AN-102 actual waste	3	<0.1	78.7

Despite the excellent elutability observed for RF resins (Fiskum et al. 2004b), Cs holdover from the first process run was apparent. Resin A, which was the lead column for both simulant and actual AP-101 tests, demonstrated significant Cs contribution from the simulant (20.5%) even though the resin bed had been eluted following simulant and actual waste processing. The residual simulant AP-101 weighting factor on Resin B (3.3%) was proportionate to the simulant breakthrough onto this resin bed (14% of the AP-101 simulant feed broke through Resin A onto Resin B). Resin B contained a fraction of AP-101 actual waste before loading with AN-102 commenced; the weighting factors reflected this mixture. In contrast, the SL-644 resin isotopic weighting factor was 51% for the first waste process cycle (Fiskum et al. 2004c) even though it was subjected to seven subsequent actual waste process cycles and higher Cs mass and ^{137}Cs Ci loading than the RF resin. These results indicate that the isotopic distribution of the residual Cs on the spherical RF resin will be a function of the composition of all wastes processed through the resin beds.

(a) The weighting factor is the estimated fraction of residual Cs on the resin bed associated with each feed processed.

The spent resin residual ^{137}Cs concentration was evaluated on the basis of actual waste first being processed through the resin bed instead of simulant (100% ^{133}Cs) to assess handling considerations of spent resin in the WTP. Because the slightly low ^{137}Cs isotopic concentration was a result of initial processing with the simulant, the final isotopic Cs concentrations can be expected to be driven in part by the first waste type processed on the resin. Thus, if actual tank waste had been processed first on the spherical RF resin, then the spent resin would likely have contained a higher ^{137}Cs concentration. Depending on the actual waste first processed on a resin bed, the ^{137}Cs concentration could have been closer to 31% (Envelope B) or 22% (Envelope A) of the total Cs. The ^{137}Cs concentrations, corrected for initial waste type, on the spent resin are summarized in Table 3.9. The highest ^{137}Cs was calculated at 5.5 µCi/g or 3.2 Ci/m^3 where bulk dry resin density was estimated at 0.58 g/mL. The calculated activity concentrations were between the *Hanford Site Solids Waste Acceptance Criteria* (McDowell 2002) Category 1 and 3 limits (4.4E-3 and 1.2E+4 Ci/m^3, respectively). The spherical RF ^{137}Cs loading concentrations were much less than previously reported for SL-644 (Arm et al. 2003) where an estimated 37 µCi/g and 50 µCi/g ^{137}Cs would remain on the SL-644 resin following Envelope A and Envelope B processing, respectively.

Table 3.9. Corrected ^{137}Cs Concentrations on Spent Resin

Resin ID	**Condition**	**^{137}Cs, µCi/g**	**^{137}Cs Isotopic Fraction, %**	**Ci/m^3 (a)**
Resin A	as found	3.52E-1	18.2	0.20
	as corrected for Env. A	4.3E-1	22	0.25
	as corrected for Env. B	6.0E-1	31	0.35
Resin B	as found	3.68E+0	20.9	2.13
	as corrected for Env. A	3.9E+0	22	2.25
	as corrected for Env. B	5.5E+0	31	3.17
(a) Where the bulk dry resin density was estimated at 0.58 g/mL.				

3.5 Contact Solution

The anionic composition of the contact solution is summarized in Table 3.10. Only nitrate was present to any significant extent. Because the resin contact solution was diluted with water (~2×) during the course of resin removal, the actual nitrate concentration that contacted the resin during the storage period was higher than the reported average of 1120 µg/mL (0.018 M). Incorporating the dilution factor of ~2, the storage solution nitrate concentration was calculated to be ~0.037 M. The H^+ concentration was expected to equal the NO_3^- concentration, from which the calculated pH was ~1.4. This was consistent with previous results where pH was tested as a function of water rinse volume (Fiskum et al. 2006a). In this case, the 3-BV(a) (equal to 1.5-apparatus volume [AV]) water rinse was expected to still be quite acidic (between pH <1 and 3). About 93% of the eluate was removed from the column with the water rinse, leaving about 7% of the eluate still in contact with the resin. In the WTP, the residual eluate in contact

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(a) One BV was defined as the resin BV when expanded in the Na-form (11 mL). The H-form resin volume contracted to 9 mL. The fluid head above the 9 mL resin (H-form) resin bed was ~10 mL. The sum of fluid-filled parts before the column was 8.3 mL.

with the resin will be a function of WTP-specific system design, such as mixing volumes above the resin bed.

Cesium continued to leach or desorb from the resins during the storage period. The ^{137}Cs concentrations in the Resins B and A contact solutions were 0.533 µCi/mL and 0.0442 µCi/mL, respectively, and commensurate with the differences in residual ^{137}Cs loading on the two resin beds. The measured Cs values were obtained after a 2× dilution factor from the resin removal process; thus, the storage solution was actually twice the measured concentration. The last water rinse samples from Resins B and A resulted in ^{137}Cs concentrations of 0.0963 µCi/mL and 0.00973 µCi/mL, respectively. Each final water rinse solution was nearly 10× less concentrated in ^{137}Cs than the storage solution. The low pH (~1.4) continued to favor Cs elution.

Table 3.10. Anionic Composition of Resin Contact Solutions

Contact Solution	**Anion, µg/mL**			
	Chloride	**Nitrite**	**Sulfate**	**Nitrate**
Resin A	<0.5	<0.75	<1	1,060
Resin B	[1.3]	<0.75	<1	1,180
Trip blank	<0.05	<0.75	<1	<0.12

The RPP-WTP flowsheet indicates the resin will be removed immediately following the elution and water rinse. The residual Cs on the spent resin (with no post-elution leaching) was estimated by mathematically adding the leached Cs (in the contact fluid) back to the dry resin mass. The residual ^{137}Cs concentrations would have increased ~30% with concentrations of 0.454 and 4.93 µCi/g for Resin A and Resin B, respectively. In this case, the calculated Resin B ^{137}Cs concentration would have attained a maximum of 7.4 Ci/m^3 (applying the ^{137}Cs isotopic ratio of 31%, consistent with Envelope B wastes).

3.6 Resin Mass Recovery

A total mass of 2.92-g dry H-form resin was recovered from each column. This mass was ~50 mg (or 1.8%) higher then the dry H-form mass added to the columns. The additional Cr, Tc, and U concentrations in the resin material were not sufficient to account for the mass difference. Several factors could account for the ~50-mg mass discrepancy. The ingoing mass was determined from a replicate resin sample measured by volume in a graduated cylinder and then taken to dryness. The readability of the 10-mL graduated cylinder was close to 0.05 mL; for the 8-mL measured volume, this introduced an uncertainty of 0.6%. The final dry mass, defined as <0.5% resin mass change over a 7-h time interval at 50°C under vacuum, may be affected by environmental factors, such as humidity, which were not specifically controlled during the drying process. Finally, the mass drying geometry was different. The spent resin was not spread out over a large beaker during the drying process but instead was contained in a 20-mL vial. The reduced surface area exposure may have limited the drying, despite the resin mixing after each mass measurement.

Within the measurement uncertainties, no resin mass loss was observed. SL-644 has been shown to lose about 1.2% mass per process cycle (Arm et al. 2003).

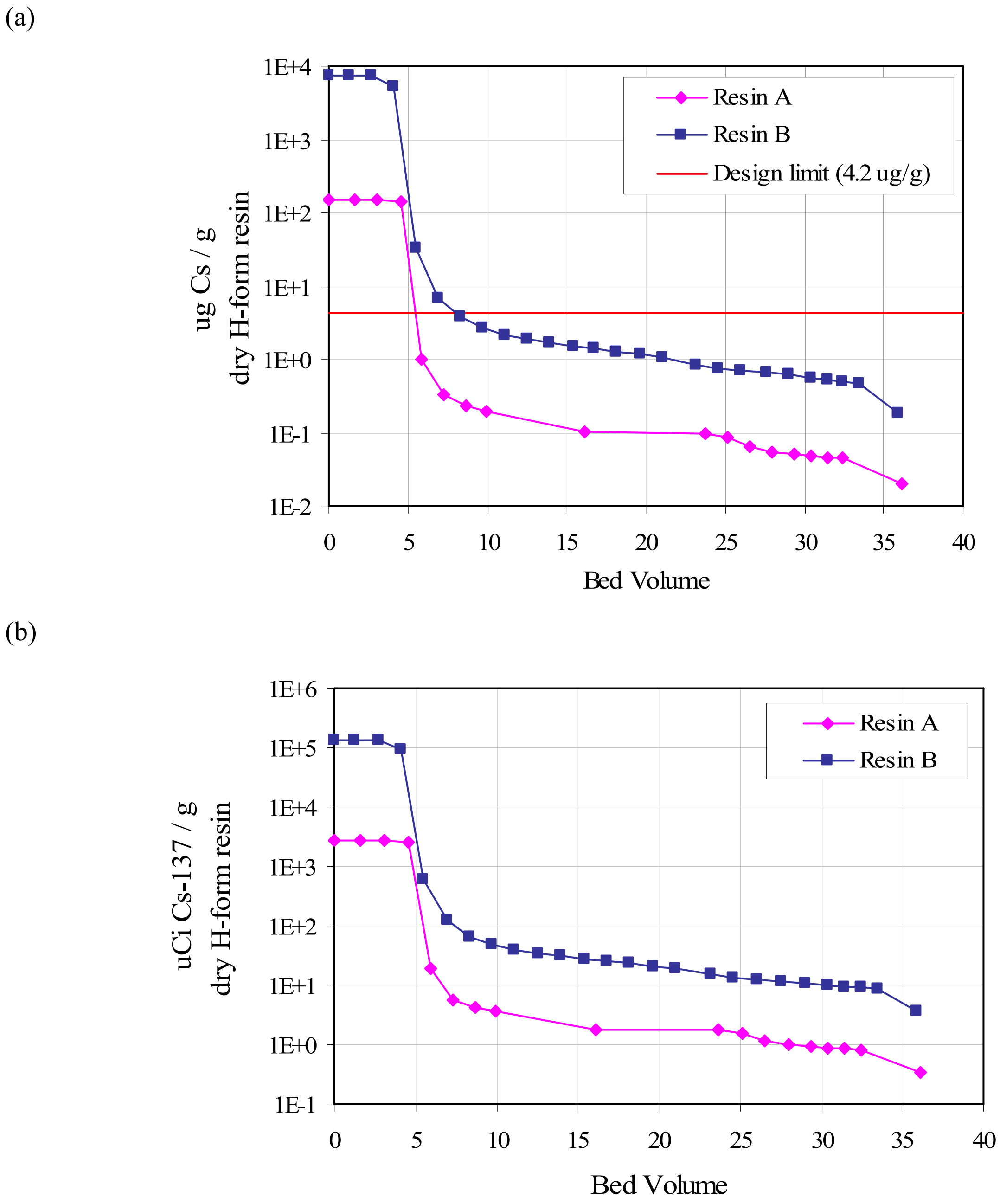
3.7 Elution Profiles

The Cs concentration on the resin (per g dry H-form basis) was evaluated as a function of eluate BV. The eluates and water rinses had been collected in 1- to 1.4-BV increments, and the ^{137}Cs was measured as part of the AN-102 Cs elution evaluation (Fiskum 2006b). Working backward through the water rinse and elution process, the total Cs on the resin was calculated by adding back the measured Cs from each rinse and eluate sample. The Resin A and B elution profiles are presented in Figure 3.1. The 0.5 M HNO_3 eluate was processed from 0 to 30 BVs, water rinse was processed from 30 to 33 BVs; and the final data point at 36 BVs represented Cs desorbed into the contact solution. The eluate and water rinse flowrates were maintained at 1.4 BV/h. The raw data are presented in Table 3.11.

Between 0 and 10 BVs of eluate, Cs concentration on the resin dropped about three orders of magnitude. The next 20 BVs of elution resulted in a Cs concentration drop by only a factor of 5 (Resin A) and 8 (Resin B), demonstrating significant tailing effects. The dip at the final processing point indicated that Cs continued to desorb from the resin while in contact with the storage solution. This soak in the storage solution removed an additional ~50% of residual Cs from the resin.

The back-calculated ^{137}Cs loading (defined as Cs concentration at 0 BVs) agreed, within experimental uncertainty, with the calculated loading from the AN-102 feed and breakthrough evaluations (shown in Table 2.1). The Resin B (lead column) contained a maximum of 7.4 mg Cs per gram of resin (dry H-form). Since the final Cs breakthrough on Resin B was only to 10% C/C_o, the resin was shown to load >7.4 mg Cs/g when processing a low-K matrix, such as AN-102.

The WTP design limit for spent resin residual Cs was defined as 4.2 µg residual Cs per g spent resin (Burgeson et al. 2004b). The elution design limit could have been met after processing 10 BVs eluate for Resin B (loaded with 0.16 Ci/g) and 6 BVs eluate for Resin A (loaded with 2.7E-3 Ci/g). Higher Cs loading on the resin beds may require higher elution volumes to reach the design limit.



Conditions:

- Spherical RF Microbeads 5E-370/641
- BV (0.5 M NaOH feed condition) = 11 mL
- Process temperature = 25 to 27°C
- Flow Rate = 1.41 BV/h
- Eluant = 0.5 M HNO_3 0 to 30 BVs
- Water rinse 31 to 33 BVs
- Contact solution 30 to 36 BVs
- Final resin condition plotted at 36 BVs

Figure 3.1. Elution Profiles for Resin A and Resin B as a Function of (a) Cs Mass and (b) ^{137}Cs Activity

Table 3.11. Cs on Resin as a Function of Processed BV

Resin A			Resin B		
BV	**µg Cs/g Resin(a)**	**µCi ^{137}Cs/g Resin(a)**	**BV**	**µg Cs/g Resin(a)**	**µCi ^{137}Cs/g Resin(a)**
0	1.55E+2	2.79E+3	0	7.40E+3	1.33E+5
1.64	1.55E+2	2.79E+3	1.31	7.40E+3	1.33E+5
3.10	1.55E+2	2.79E+3	2.69	7.40E+3	1.33E+5
4.52	1.39E+2	2.50E+3	4.10	5.13E+3	9.26E+4
5.89	1.03E+0	1.85E+1	5.51	3.36E+1	6.07E+2
7.26	3.21E-1	5.79E+0	6.92	6.66E+0	1.20E+2
8.66	2.36E-1	4.25E+0	8.32	3.73E+0	6.73E+1
9.99	2.01E-1	3.62E+0	9.72	2.71E+0	4.89E+1
16.16	1.02E-1	1.83E+0	11.14	2.17E+0	3.91E+1
23.71	9.71E-2	1.75E+0	12.53	1.90E+0	3.43E+1
25.17	8.68E-2	1.57E+0	13.94	1.70E+0	3.06E+1
26.54	6.39E-2	1.15E+0	15.39	1.52E+0	2.75E+1
28.00	5.50E-2	9.92E-1	16.76	1.39E+0	2.51E+1
29.39	5.11E-2	9.23E-1	18.18	1.27E+0	2.29E+1
30.44(b)	4.88E-2	8.81E-1	19.62	1.16E+0	2.09E+1
31.43(b)	4.67E-2	8.43E-1	21.04	1.07E+0	1.93E+1
32.43(b)	4.47E-2	8.06E-1	23.15	8.49E-1	1.53E+1
36.16(c)	2.04E-2	3.52E-1	24.56	7.66E-1	1.38E+1
No additional data.			26.00	7.07E-1	1.27E+1
			27.55	6.54E-1	1.18E+1
			28.99	6.10E-1	1.10E+1
			30.44	5.53E-1	9.98E+0
			31.46(b)	5.22E-1	9.41E+0
			32.45(b)	4.98E-1	8.98E+0
			33.46(b)	4.77E-1	8.61E+0
			35.91(c)	1.87E-1	3.68E+0

(a) Dry, H-form resin.
(b) Water rinse samples.
(c) Contact solution.

4.0 Quality-Assurance Requirements

PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the PNWD Waste Treatment Plant Support Project Quality Assurance Project Plan (QAPjP) approved by the RPP-WTP QA organization. This work was performed to the quality requirements of NQA-1-1989, Part I, “Basic and Supplementary Requirements,” NQA-2a-1990, Part 2.7, and DOE/RW-0333P, Rev. 13, *Quality Assurance Requirements and Descriptions* (QARD). These quality requirements were implemented through PNWD’s *Waste Treatment Plant Support Project (WTPSP) Quality Assurance Requirements and Description Manual* and to the approved Test Plan, TP-RPP-WTP-378, Rev. 0, and Test Exceptions 24590-PTF-TEF-RT-05-00008 and 24590-PTF-TEF-RT-05-00011. The analytical requirements were implemented through WTPSP’s Statement of Work (WTPSP-SOW-005) with the RPL ASO.

Experiments that were not method-specific were performed in accordance with PNWD’s procedures QA-RPP-WTP-1101 “Scientific Investigations” and QA-RPP-WTP-1201 “Calibration Control System,” verifying that sufficient data were taken with properly calibrated measuring and test equipment (M&TE) to obtain quality results.

BNI’s QAPjP, PL-24590-QA00001, Rev. 0, was not applicable because the work was not performed in support of environmental/regulatory testing, and the data should not be used as such.

PNWD addressed internal verification and validation activities by conducting an independent technical review of the final data report in accordance with PNWD’s procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the test-plan objectives. This review procedure is part of PNWD’s *WTPSP Quality Assurance Requirements and Description Manual*.

Analytical data quality and QC are in Appendix A. All raw and reduced data are maintained and/or cross-referenced in data files under Project 42365 at PNWD.

5.0 Conclusions

The spent spherical RF resin (used in three process cycles, two of which were actual tank waste) has been characterized with respect to residual metals and radionuclides.

- The residual ^{137}Cs concentrations were determined to be 0.352 and 3.68 µCi/g for Resin A and Resin B, respectively. The total Cs concentrations measured 0.0204 and 0.187 µg/g for Resin A and Resin B, respectively. The difference between the final concentrations on the two resin beds was attributed to the higher Cs loading on Resin B during the last process cycle.
- Elution to a resin Cs concentration of 4.2 µg/g (design limit) was accomplished within 10 BVs given a final Cs load concentration of 7.4 mg/g.
- The spent resin ^{137}Cs isotopic fractions were slightly low (18.2% for Resin A and 20.9% for Resin B) with respect to AN-102 (21.5%) and AP-101 (22.4%), indicating minor hold-over of Cs from the first material processed (^{133}Cs in the simulant).
- The following metals concentrated to some extent on the resin: Cs, Cr, Ni, Tc, and U (analytes of interest) and Cu, Mo, Sn, and W (opportunistic analytes).
- The Ag, As, Ba, Cd, Pb, and Se concentrations were measured at ≤10 µg/g. Total Cr was present at 340 µg/g in the spent resin and could possibly exceed the Toxicity Characteristic criteria of 40 CFR 261.24 (RCRA). However, Cr retained on the resin after the 0.5 M HNO_3 elution was not expected to be solubilized with dilute acetic acid (per the TCLP test). A TCLP test needs to be completed to evaluate Cr with respect to regulatory threshold.(a)
- The following radionuclides were quantified on the resin: ^{60}Co, ^{99}Tc, ^{126}Sn/Sb, ^{137}Cs, ^{241}Am, ^{238}Pu, and $^{239+240}$Pu. All major sources of alpha and beta isotopes were identified.
- A maximum of 2-nCi/g total alpha activity from TRU components was measured on the RF resin, nearly two orders of magnitude below the TRU waste threshold definition of 100 nCi/g.
- No resin mass loss was observed after processing through the three cycles.

(a) The TCLP and regulatory analysis using spiked simulant processing is currently scheduled to be conducted at SRNL under technical scoping statement A-205.

Appendix A

Analytical Results QC Summary

Appendix A: Analytical Results QC Summary

Analytical results and batch quality control (QC) results are summarized in Table A.1 through Table A.3 along with QC, i.e., duplicate precision (relative percent difference [RPD]), matrix spike (MS) recovery, and blank spike (BS) recovery. The RPD was evaluated only for analytes >EQL (estimated quantitation limit).

Gamma Energy Analysis

Gamma energy analysis was conducted directly and non-destructively on each resin sample in its entirety. Therefore, laboratory control samples as well as BS, and MS QC samples were not required. All instrument QC requirements were met. Analyte concentration uncertainty represented the total propagated uncertainty from all sources, including calibration and counting uncertainty. Only analytes greater than the detection limit were included in the QC data table.

The resin density and volume did not exactly match the standards. Measurement response differences from these two factors were evaluated and determined to cancel each other out. The samples were measured twice, once with the up-looking detector and once with the side-looking detector. The RPD was based on the results from the two detectors. The precision between these two counting geometries was good, supporting the analytical approach. The ^{137}Cs measured by gamma energy analysis (GEA) was compared to the independently-measured inductively coupled plasma-mass spectrometry (ICP-MS) ^{137}Cs; the results agreed within 8%, further supporting the accuracy of the GEA data analysis.

Acid Digestion

Each resin sample was processed in duplicate by acid digestion. Two MS samples were prepared. A Resin A sample was spiked with inductively coupled plasma-atomic emission spectrometry (ICP-AES) analytes; a Resin B sample was spiked with ICP-MS analytes and low-level U (for kinetic phosphorescence analysis [KPA]). Two preparation blanks were prepared with the resin samples. A resin matrix blank was prepared with one preparation blank in a parallel process in a non-radioactive fume hood.

Inductively Coupled Plasma-Atomic Emission Spectrometry

Overall analyte concentration uncertainties were within ± 15% for analytes >EQL. All instrument and batch QC met acceptance criteria.

Inductively Coupled Plasma-Mass Spectrometry

ICP-MS was used to determine the ^{133}Cs and ^{99}Tc concentrations and the Cs isotopic distribution. Both Cs and Tc results demonstrated high precision. The ^{135}Cs and ^{137}Cs concentrations were calculated from the sample isotopic ratio and the ^{133}Cs concentration. Insufficient Cs and Tc were added to the MS sample to evaluate recovery. The post-spike, conducted at the work station, recovered 89% and 102%, respectively. Insufficient Cs was added to the BS to evaluate recovery. An additional BS sample was prepared separately from the analytical batch using the same procedure, and it recovered 100%. Overall analyte concentration uncertainties were within ± 15%. All instrument QC requirements were met.

Uranium by Kinetic Phosphorescence Analysis

The U content was relatively high in the samples, and the MS prepared for the KPA contained insufficient additional U to evaluate recovery. The RPDs of duplicate samples were found to be within the acceptance criterion of <15%, and the BS recovery met the acceptance criteria. All instrument QC requirements were met.

Americium, Curium, and Plutonium

The BS and MS QC samples were prepared after sample digestion. The Am chemistry closely follows Cm chemistry, and the Am spike satisfied the MS and BS recovery requirements for Cm. All analyte concentrations were well below the minimum reportable quantity (MRQ). The Pu and Am BS and MS recoveries met the acceptance criteria. The duplicate sample RPDs met the acceptance criterion, except the Resin A Am result, which resulted in 21% RPD. Given the experimental error of 14% (2-σ), the Resin A Am results were within 1.3-sigma agreement. All instrument QC requirements were met. Reported analyte concentration uncertainty represented the total propagated uncertainty from all sources, including sub-sampling, calibration, and counting uncertainty.

Total Alpha and Total Beta

All batch and instrument QC requirements were met.

The sum of alpha (summation of Pu, Am, and Cm activities measured by alpha energy analysis [AEA]) was slightly higher than the total alpha measure and was determined to be a better measure of the total alpha activity in the spent resin samples.

The sum of beta emitters (summation of ^{137}Cs, ^{60}Co, and ^{99}Tc activities) was slightly higher than the total beta measurement. The slight bias was attributed to and consistent with the higher energy calibration source for the gas-flow proportional counters (^{90}Sr/^{90}Y). The sum of beta was a better measure of the total beta activity in the spent resins.

Ion Chromatography

All batch and instrument QC requirements were met.

Table A.1. ICP-AES, ICP-MS, KPA, and IC QC Results of Spent Resin

				Process	Matrix	Resin A			Resin B			BS	MS	Serial
	Analysis	MRQ	EQL	Blank	Blank	Sample	Dup.	RPD	Sample	Dup.	RPD	Recovery	Recovery	Dilution
Analyte	Method	µg/g	µg/g	µg/g(a)	µg/g(a)	µg/g(a)	µg/g(a)	%	µg/g(a)	µg/g(a)	%	%	%	%
Criteria:								<15%			<15%	80 - 120	75 - 125	<10
Target Analyte														
Ag	ICP-AES	14	1.6	<1.6	<0.54	[3.2]	[3.0]	[6.5]	[3.2]	[3.5]	[9.0]	98	120	—
As	ICP-AES	72	6.5	<6.3	<2.2	<6.4	<6.3		<6.1	[6.9]		106	101	—
Ba	ICP-AES	78	0.45	[0.73]	[0.41]	[1.0]	[1.9]	[62]	[0.82]	[1.1]	[29]	101	100	—
Cd	ICP-AES	7.5	0.46	[0.55]	<0.16	[0.63]	[0.52]	[19]	[0.58]	[0.79]	[31]	106	106	—
^{133}Cs	ICP-MS	10	6.5E-3	[4.1E-3]	na	1.37E-2	1.48E-2	7.7	0.118	0.118	0	(d)	(d)	—
^{135}Cs	ICP-MS	nmrq		na	na	2.49E-3	2.47E-3	0.46	3.05E-2	3.02E-2	1.0	na	na	—
^{137}Cs	ICP-MS	nmrq		na	na	3.73E-3	3.69E-3	1.1	3.91E-2	3.92E-2	0.33	na	na	—
Cr	ICP-AES	15	0.66	[0.92]	[0.30]	334	341	2.1	334	339	1.5	101	99	1.6
Na	ICP-AES	180	44	[45]	301	[67]	[68]	[1.5]	[150]	[160]	[6.5]	106	104	—
Ni	ICP-AES	30	1.2	<1.1	[0.47]	25.9	26.2	1.2	[9.4]	[10]	[6.2]	101	105	—
Pb	ICP-AES	300	7.2	<7.0	<2.4	[9.6]	[9.3]	[3.2]	[7.9]	[9.2]	[15]	97(c)	100	—
Se	ICP-AES	50	7.2	[8.2]	[3.0]	[8.9]	[8.6]	[3.4]	[9.0]	[10]	[11]	116	104	—
^{99}Tc	ICP-MS	23	4.6E-2	<9.2E-3	na	19.1	18.8	1.6	19.5	19.2	1.6	98	(d)	0.92
Th	ICP-AES	500	77	<75	<26	<76	<75	—	<72	<77	—	100	98	—
U	ICP-AES	1000	210	<210	<71	<210	<210	—	<200	<210	—	114	104	—
U	KPA	50	nr	0.34	na	207	179	14.5	104	98	5.9	93	(d)	—
Opportunistic Analytes(b)														
Al	ICP-AES	nmrq	11	[78]	<3.6	[18]	[20]	[11]	[27]	[28]	[3.6]	105	106	—
B	ICP-AES	nmrq	1.4	<1.4	15.7	16.8	17.2	2.4	21.9	22.4	2.3			—
Be	ICP-AES	nmrq	0.039	<0.038	<0.013	<0.039	<0.038		<0.037	<0.039		107	103	—
Bi	ICP-AES	nmrq	6.4	[6.4]	[2.7]	[9.9]	[10]	[1.0]	[7.8]	[9.1]	[15]	115	110	—
Ca	ICP-AES	nmrq	3.9	26	[6.7]	47.4	92.9	65	[20]	44.0	75	103	103	—
Ce	ICPAES	nmrq	14	<13	<4.6	<13	<13		<13	<14				—
Co	ICP-AES	nmrq	1.2	<1.1	<0.39	[7.4]	[7.3]	[1.4]	[2.4]	[2.7]	[12]			—
Cu	ICP-AES	nmrq	3.0	<2.9	[1.2]	69.6	70.4	1.1	[28]	31.2	11	101	104	—
Dy	ICP-AES	nmrq	2.1	<2.0	<0.69	<2.0	<2.0	—	<1.9	<2.1	—			—
Eu	ICP-AES	nmrq	0.65	<0.63	<0.22	<0.64	<0.63	—	<0.61	[0.69]	—			—
Fe	ICP-AES	nmrq	1.9	17	[0.90]	[6.7]	[7.1]	[5.8]	[9.3]	[9.0]	[3.3]	101	102	—
K	ICP-AES	nmrq	350	<340	<120	<350	<340	—	<330	<350	—	106	104	—
La	ICP-AES	nmrq	2.9	<2.8	<0.96	<2.8	<2.8	—	<2.7	<2.9	—	104	103	—

Table A.1 (Contd)

Analyte	Analysis Method	MRQ µg/g	EQL µg/g	Process Blank µg/g(a)	Matrix Blank µg/g(a)	Resin A			Resin B			BS Recovery %	MS Recovery %	Serial Dilution %
						Sample µg/g(a)	Dup. µg/g(a)	RPD %	Sample µg/g(a)	Dup. µg/g(a)	RPD %			
Criteria:								<15%			<15%	80 - 120	75 - 125	<10
Li	ICP-AES	nmrq	2.1	<2.1	<0.71	<2.1	<2.0	—	<2.0	<2.1	—	107	106	—
Mg	ICP-AES	nmrq	5.4	219	<1.8	<5.3	<5.2	—	<5.1	<5.4	—	97	101	—
Mn	ICP-AES	nmrq	0.19	[0.37]	[0.082]	[0.30]	[0.28]	[6.9]	[0.32]	[0.33]	[3.1]	103	105	—
Mo	ICP-AES	nmrq	1.2	[1.3]	[0.43]	[8.6]	[8.7]	[1.2]	15.5	16.4	5.6			—
Nd	ICP-AES	nmrq	13	<13	<4.4	<13	<13	—	<12	<13	—	105	103	—
P	ICP-AES	nmrq	7.6	[19]	[2.8]	[12]	[12]	[0]	[18]	[22]	[20]			—
Pd	ICP-AES	nmrq	36	<35	<12	<35	<35	—	<34	<36	—			—
^{85}Rb	ICP-MS	nmrq	1.9E-2	[7.0E-3]	na	[3.7E-3]	[6.2E-3]	[50]	[5.1E-3]	[6.4E-3]	[23]	(d)	110	—
Rh	ICP-AES	nmrq	16	<16	<5.5	<16	<16	—	<15	<16	—			—
Ru	ICP-AES	nmrq	4.2	<4.1	<1.4	<4.2	<4.1	—	<4.0	[4.5]	—			—
Sb	ICP-AES	nmrq	7.4	[10]	[3.3]	[8.6]	<7.2	—	[7.8]	[10]	[25]			—
Si	ICP-AES	nmrq	11	<11	<3.6	<11	<11	—	<10	<11	—			—
Sn	ICP-AES	nmrq	45	<44	<15	[93]	[90]	[3.3]	[62]	[75]	[19]			—
Sr	ICP-AES	nmrq	0.18	[0.29]	[0.18]	[0.43]	[0.44]	[2.3]	[0.54]	[0.59]	[8.8]	102	102	—
Te	ICP-AES	nmrq	9.2	[9.6]	[3.4]	<9.1	<8.9	—	[9.1]	[11]	[19]			—
Ti	ICP-AES	nmrq	0.63	<0.62	<0.21	<0.62	<0.61	—	<0.59	<0.63	—			—
Tl	ICP-AES	nmrq	5.8	<5.7	<2.0	<5.8	<5.7	—	<5.5	<5.8	—			—
V	ICP-AES	nmrq	1.2	<1.2	<0.41	<1.2	<1.2	—	<1.1	<1.2	—	100	101	—
W	ICP-AES	nmrq	3.9	<3.8	<1.3	[21]	[22]	[4.7]	39.5	42.6	7.6			—
Y	ICP-AES	nmrq	0.55	<0.54	<0.18	<0.55	<0.54	—	<0.52	<0.55	—			—
Zn	ICP-AES	nmrq	0.51	250	2.27	5.47	6.43	16	[4.1]	[4.6]	[11]	113	106	—
Zr	ICP-AES	nmrq	1.6	<1.5	<0.52	[10]	[11]	[9.5]	[3.6]	[3.9]	[8.0]			—

(a) The overall error for values without brackets was estimated to be within ±15% (analytes greater than the EQL). Bracketed values identify sample concentrations that were <EQL but >MDL, and errors likely exceeded 15%. The MDL was typically a factor of 10 lower than the EQL.

(b) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

(c) Value from 3× dilution.

(d) The spike concentration was too low relative to the sample concentration for proper evaluation.

BS = blank spike; EQL = estimated quantitation limit; MRQ = minimum reportable quantity; MS = matrix spike; nmrq = no minimum reportable quantity; RPD = relative percent difference

nr = not reported; na = not analyzed

"—" indicates calculation was not required

The bolded and shaded result indicates non-compliance with BNI acceptance criteria; see discussion.

Table A.2. Radionuclide QC Results of Spent Resin

		MRQ	Process Blank	Resin A			Resin B			BS Recovery %	MS Recovery %
Analyte	**Method**	**µCi/g**	**µCi/g ± 1-σ**	**Sample µCi/g ± 1-σ**	**Duplicate µCi/g ± 1-σ**	**RPD %**	**Sample µCi/g ± 1-σ**	**Dup µCi/g ± 1-σ**	**RPD %**		
Criteria:						*<15*			*<15*	*80 - 120*	*75 - 125*
^{60}Co	GEA	0.50	(a)	8.07E-2 2%	7.66E-2 3%	5	2.01E-2 2%	1.90E-2 3%	6	(a)	(a)
^{99}Tc	ICP-MS	0.40	<2E-4	3.25E-1	3.20E-1	1.6	3.32E-1	3.26E-1	1.6	98	(b)
^{126}Sn/Sb	GEA	67	(a)	3.48E-4 15%	3.68E-4 13%	6	<8E-4	<8E-4	--	(a)	(a)
^{137}Cs	GEA	0.050	(a)	3.60E-1 4%	3.43E-1 4%	5	3.75E+0 4%	3.60E+0 4%	4	(a)	(a)
^{238}Pu	Separations and α spec.	0.010	<3E-6	3.65E-4 6%	3.58E-4 6%	2	1.59E-4 12%	1.79E-4 12%	12	--	--
$^{239/240}$Pu	Separations and α spec.	0.010	<3E-6	1.78E-3 3%	1.66E-3 3%	7	5.77E-4 6%	6.66E-4 6%	14	100	90
^{241}Am	Separations and α spec.	0.010	2.26E-6 26%	3.00E-4 7%	2.44E-4 7%	**21**	2.34E-4 9%	2.48E-4 8%	6	98	90
^{242}Cm	Separations and α spec.	0.010	<8E-7	<1E-5	<9E-6	--	<1E-5	<1E-5	--	--	--
$^{243+244}$Cm	Separations and α spec.	0.010	<1E-6	<2E-5	<1E-5	--	<2E-5	1.99E-5 29%	--	--	--
Sum of alpha	Separations and α spec.	na	--	2.45E-3 3%	2.26E-3 3%	8	9.69E-4 5%	1.11E-3 4%	14	--	--
Total alpha	Scintillation count	0.010	<4E-5	1.84E-3 14%	1.87E-3 14%	2	9.98E-4 20%	8.65E-4 23%	14	99	120
Sum of beta	GEA + ^{99}Tc	na	--	7.66E-1 4%	7.40E-1 3%	3	4.10E+0 4%	3.95E+0 4%	4	--	--
Total beta	Gas-flow prop. count	0.001	<9E-6	6.41E-1 4%	6.79E-1 4%	6	3.63E+0 4%	3.77E+0 4%	4	90	(b)

(a) Direct measurement by GEA did not require a process blank, BS, or MS.
(b) The analyte spike was <5% of the analyte concentration; recovery could not be evaluated.
Analyses were conducted according to ASR 7603, reference date 8/15/05.
BS = blank spike; MRQ = minimum reportable quantity; MS = matrix spike; RPD = relative percent difference.
Gamma-emitting radionuclides less then the instrument detection limit were not included in the QC evaluation.

Table A.3. IC QC Results of Contact Solution

				Dilution	Resin A Contact Solution			Resin B Contact Solution			BS	MS
	Analysis	**MRQ**	**EQL**	**Blank**	**Sample**	**Dup.**	**RPD**	**Sample**	**Dup.**	**RPD**	**Recovery**	**Recovery**
Analyte	**Method**	**µg/mL**	**µg/mL**	**µg/mL**	**µg/mL**	**µg/mL**	**%**	**µg/mL**	**µg/mL**	**%**	**%**	**%**
Criteria:							<15%			<15%	80 - 120	75 - 125
chloride	IC	10	3	<0.05	<0.5	<0.5	--	[1.3]	[1.1]	--	98	96
nitrite	IC	3000	2	<0.75	<0.75	<0.75	--	<0.75	<0.75	--	101	95
sulfate	IC	2300	2.8	<0.28	<1.0	<1.0	--	<1.0	<1.0	--	99	94
nitrate	IC	3000	16	<0.32	1,060	1,070	0.9	1,180	1,180	0	97	99

Analyses were conducted according to ASR 7603.

BS = blank spike; EQL = estimated quantitation limit; MRQ = minimum reportable quantity; MS = matrix spike; RPD = relative percent difference.

Appendix B

Analyte Fractionation Through Ion Exchange Processing

Appendix B: Analyte Fractionation Through Ion Exchange Processing

The total amounts of various metals and radionuclides fed to the ion exchanger for the three process feeds are shown in Tables B.1 through B.4. Also shown are the fractionations through two ion exchange tests (AP-101 actual waste and AN-102 actual waste). Fractionation through the AP-101 simulant shakedown test was not assessed. The composite data were derived from previously-reported test data (Fiskum et al. 2006a and 2006b) and simulant formulation (Russell et al. 2003).

The composition of the AP-101 actual waste effluent was estimated. Only the first 60-BV composite of AP-101 effluent was analyzed. Therefore, the total effluent AP-101 analyte masses and activities (except ^{137}Cs) were estimated by attributing the entire effluent analyte composition to that obtained for the 60-BV composite. The AP-101 composite ^{137}Cs activity was evaluated based on the measured concentration in the entire effluent. Except for Cs, most analytes remained with the LAW effluent.

Tables B.5 and B.6 summarize the analyte recoveries relative to total mass of analyte (from combined feeds) that contacted the resins.

Table B.1. AP-101 Analyte Load and Fractionation to the Effluent and Eluate

Analyte	AP-101 Simulant Feed(a)	AP-101 Actual Waste				
					Resin A Eluate	
		Feed	Effluent(b)	Effluent Recovery(b)	Eluate	Recovery
	µg	µg	µg	%	µg	%
Ag	0	[7.7E+2]	[7.4E+2]	[97]	<5.9E+2	<77
As	0	<2.6E+3	<2.8E+3	na	<2.4E+3	na
Ba	[4.7E+2]	[1.2E+3]	[6.9E+2]	[58]	[3.4E+2]	[28]
Cd	[1.6E+3]	2.74E+3	2.62E+3	95.5	[1.9E+2]	[6.87]
Cr	2.11E+5	2.27E+5	2.32E+5	102	[8.0E+2]	[0.35]
K	3.88E+7	4.41E+7	4.33E+7	98.2	[1.6E+5]	[0.36]
Mn	[1.5E+2]	[1.3E+2]	[1.6E+2]	[125]	<6.8E+1	<51
Na	1.64E+8	1.81E+8	1.84E+8	102	2.50E+5	0.14
Ni	<305	5.14E+3	6.09E+3	118	<4.2E+2	<8.2
Pb	[1.4E+4]	[1.8E+4]	[1.3E+4]	[69]	[4.8E+3]	[26]
Se	0	[4.0E+3]	[4.4E+3]	[112]	[2.9E+3]	[72]
Sr	[7.2E+1]	[4.9E+2]	[4.0E+2]	[81]	[1.1E+2]	[23]
Th	0	<1.5E+3	<1.7E+3	na	<1.4E+3	na
U KPA	0	6.52E+4	6.14E+4	94.1	1.65E+3	2.5
Rb-85	5.15E+3	4.46E+3	3.92E+3	87.9	9.67E+1	2.2
Cs-133	8.55E+3	5.60E+3	[1.6E+0]	[2.9E-2]	3.76E+3	67.1
Opportunistic Analytes(c)						
Al	9.65E+6	1.04E+7	1.05E+7	101	[4.0E+3]	[0.038]
B	2.02E+4	1.97E+4	1.76E+4	89	[1.3E+3]	[6.6]
Be	[5.2E+1]	1.59E+3	1.54E+3	97	[2.3E+1]	[1.4]
Bi	0	[4.7E+3]	[4.0E+3]	[83]	<2.3E+3	<49
Ca	<7.2E+3	[2.1E+4]	[1.3E+4]	[64]	[2.4E+3]	[12]
Co	0	[1.0E+3]	[1.1E+3]	[104]	<4.2E+2	<40
Cu	[1.4E+3]	[5.2E+3]	[4.8E+3]	[92]	<1.1E+3	<21
Dy	0	[1.1E+3]	[1.1E+3]	[100]	<7.4E+2	<69
Eu	0	[5.1E+2]	[5.1E+2]	[101]	<2.4E+2	<47
Fe	[3.9E+3]	[7.0E+3]	[7.6E+3]	[108]	<6.8E+2	<10
La	0	[1.5E+3]	[1.5E+3]	[98]	<1.0E+3	<68
Li	[8.0E+2]	[2.3E+3]	[2.1E+3]	[93]	<7.6E+2	<33
Mg	0	<2.2E+3	<2.3E+3	na	<2.0E+3	na
Mo	1.85E+4	2.13E+4	2.19E+4	103	[5.5E+2]	[2.6]
Nd	0	[8.3E+3]	[8.1E+3]	[98]	<4.8E+3	<58
P	5.56E+5	5.33E+5	5.47E+5	[103]	[3.0E+3]	[0.56]
Pd	0	[3.1E+4]	[3.0E+4]	[97]	<1.3E+4	<42
Rh	0	[1.1E+4]	[1.1E+4]	[97]	<5.9E+3	<52
Ru	0	[7.0E+3]	[7.2E+3]	[103]	<1.5E+3	<22
Sb	0	<3.0E+3	<3.2E+3	na	[3.8E+3]	[>128]
Si	1.73E+5	1.56E+5	1.36E+5	87.0	<3.9E+3	<2.5

Table B.1 (Contd)

Analyte	AP-101 Simulant Feed	AP-101 Actual Waste				
		Feed	Effluent$^{(a)}$	Effluent Recovery$^{(a)}$	Resin A Eluate	
					Eluate	Recovery
	µg	µg	µg	%	µg	%
Sn	0	[1.2E+5]	[1.3E+5]	[109]	<1.6E+4	<13
Te	0	[8114]	[8.9E+3]	[110]	[3.4E+3]	[42]
Ti	0	[4.0E+2]	[3.6E+2]	[91]	<2.3E+2	<57
Tl	0	<2.3E+3	<2509	na	<2.1E+3	na
V	0	[6.7E+2]	[6.7E+2]	[100]	<4.4E+2	<65
W	3.37E+4	3.69E+4	3.74E+4	101	<1.4E+3	<3.9
Y	0	2.30E+2	<2.4E+2	[<104]	<2.0E+2	<87
Zn	1.18E+4	8.05E+3	8.99E+3	112	2.51E+3	31.1
Zr	[7.2E+2]	[4.1E+3]	[4.3E+3]	[104]	<5.6E+2	<14

(a) The simulant processing effluent and eluates were not analyzed.
(b) The first 60-BV effluent was analyzed. The effluent recovery was estimated by applying the 60-BV concentration (after correction for dilution) to the entire process volume.
(c) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

Notes:

AP-101 simulant feed: "0" indicates that the analyte was neither in the feed formulation nor was it detected in analysis.

Bracketed results were greater than the MDL but less than the EQL. The less-than values (<) indicate that the results were less than the MDL: reported values were the instrument detection limits multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.

Data were derived from previous report (Fiskum et al. 2006a).

Table B.2. AP-101 Radionuclide Load and Fractionation to the Effluent and Eluate

Radionuclide$^{(a)}$	AP-101 Actual Waste				
	Feed	Effluent$^{(b)}$	Recovery$^{(b)}$	Resin A Eluate	
				Eluate	Recovery
	µCi	µCi	%	µCi	%
^{60}Co	<5.E+0	2.12E+0	46.2	<1.4E+0	na
^{137}Cs	1.79E+5	8.14E+2	0.45	1.22E+5	68.3
^{238}Pu	7.27E-2	1.26E-1	173	3.25E-3	4.46
$^{239+240}$Pu	1.53E-1	1.76E-1	115	1.36E-2	8.85
^{241}Am	2.17E-1	3.11E-1	143	5.22E-3	2.40
^{242}Cm	<6.E-4	<8.E-4	134	<8.E-4	na
$^{243+244}$Cm	9.42E-3$^{(c)}$	9.73E-2$^{(c)}$	1033$^{(c)}$	9.99E-4	10.6
Total alpha	<8.E-1	<8.E-1	na	<6.1E-1	na
Sum of alpha	4.53E-1	7.11E-1	157	2.38E-2	5.25

(a) Reference date is 8/15/05.
(b) The first 60-BV effluent was analyzed. The effluent recovery was estimated by applying the 60-BV concentration (after correction for dilution) to the entire process volume.
(c) High blank

Data were derived from previous test report (Fiskum et al. 2006a).

Table B.3. AN-102 Analyte Load and Fractionation to the Effluent and Eluate

	AN-102 Actual Waste						
			Effluent	Resin B Eluate		Resin A Eluate	
Analyte	Feed	Effluent	Recovery	Eluate	Recovery	Eluate	Recovery
	µg	µg	%	µg	%	µg	%
Ag	<9.7E+2	<9.8E+2	na	<1.2E+2	na	<1.1E+2	na
As	<3.9E+3	<3.9E+3	na	<4.7E+2	na	<4.5E+2	na
Ba	[7.9E+2]	[3.4E+2]	[44]	[1.9E+2]	[24]	[1.3E+2]	[16]
Cd	5.68E+4	5.77E+4	102	[1.0E+2]	[0.18]	[5.6E+1]	[0.10]
Cr	7.45E+4	7.61E+4	102	[2.4E+2]	[0.32]	[1.7E+2]	[0.23]
K	2.34E+6	2.48E+6	106	<2.5E+4	<1.1	[2.7E+4]	[1.1]
Mn	[1.1E+3]	[1.1E+3]	[102]	[2.9E+1]	[2.7]	[2.6E+1]	[2.4]
Na	2.55E+8	2.55E+8	99.9	3.14E+5	0.123	3.55E+5	0.14
Ni	3.94E+5	3.95E+5	100	1.18E+3	0.299	2.14E+3	0.54
Pb	1.27E+5	1.11E+5	87.3	1.31E+4	10.3	1.10E+4	8.65
Se	[5.5E+3]	[6.4E+3]	[117]	<5.2E+2	<9.4	<5.0E+2	<9.0
Sr	2.57E+5	2.62E+5	102	2.5E+2	0.10	1.92E+2	0.075
Th	<2.3E+3	<2.3E+3	na	<2.8E+2	na	<2.7E+2	na
U KPA	1.81E+4	1.80E+4	99.5	7.83E+2	4.32	3.62E+2	1.99
Rb-85	3.53E+3	3.06E+3	86.6	1.99E+2	5.63E+0	na	na
Cs-133	1.10E+4	[4.1E+0]	[0.038]	1.17E+4	1.07E+2	na	na
Opportunistic Analytes(a)							
Al	5.66E+6	5.75E+6	102	[2.39E+3]	[0.042]	[1.23E+3]	[0.022]
B	4.75E+4	3.72E+4	78.5	2.2E+3	4.67	[6.87E+2]	[1.4]
Be	<2.4E+1	[2.53E+1]	[107]	[5.08E+0]	[22]	<2.7E+0	na
Bi	[5.18E+3]	[4.83E+3]	[93]	<4.6E+2	<8.9	<4.4E+2	<8.6
Ca	2.61E+5	2.57E+5	98.7	[6.10E+2]	[0.23]	[7.69E+2]	[0.29]
Co	[4.39E+3]	[4.37E+3]	[100]	<8.3E+1	<1.9	<8.0E+1	<1.8
Cu	2.40E+4	2.26E+4	94.5	2.9E+3	12.3	2.96E+3	12.4
Dy	<1.2E+3	<1.2E+3	na	<1.5E+2	na	<1.4E+2	na
Eu	[5.96E+2]	[5.75E+2]	[96]	<4.7E+1	<7.9	<4.5E+1	<7.6
Fe	[7.54E+3]	[6.67E+3]	[88]	[3.22E+2]	[4.3]	[2.00E+2]	[2.6]
La	[3.60E+3]	[3.45E+3]	[96]	<2.1E+2	<5.7	<2.0E+2	<5.5
Li	[2.33E+3]	[2.23E+3]	[96]	<1.5E+2	<6.5	<1.5E+2	<6.3
Mg	<3.2E+3	<3.2E+3	<1.0E+2	<3.9E+2	<1.2E+1	<3.8E+2	<1.2E+1
Mo	5.09E+4	5.20E+4	102	[9.66E+1]	[0.19]	[9.98E+1]	[0.20]
Nd	[1.64E+4]	[1.63E+4]	[99]	<9.5E+2	<5.8	<9.2E+2	<5.6
P	1.71E+6	1.74E+6	102	[9.15E+2]	[0.053]	[5.89E+2]	[0.034]
Pd	[3.04E+4]	[2.99E+4]	[98]	<2.6E+3	<8.5	<2.5E+3	<8.2
Rh	[1.72E+4]	[1.68E+4]	[98]	<1.2E+3	<6.9	<1.1E+3	<6.6
Ru	2.85E+4	2.90E+4	102	<3.1E+2	<1.1	<3.0E+2	<1.0
Sb	4.73E+3	[4.60E+3]	[97]	[5.93E+2]	[13]	[6.05E+2]	[13]
Si	1.80E+5	[3.91E+4]	[22]	[4.74E+3]	[2.6]	[2.98E+3]	[1.7]

Table B.3 (Contd)

Analyte	AN-102 Actual Waste						
			Effluent	Resin B Eluate		Resin A Eluate	
	Feed	Effluent	Recovery	Eluate	Recovery	Eluate	Recovery
	µg	µg	%	µg	%	µg	%
Sn	[3.26E+4]	[3.68E+4]	[113]	<3.2E+3	<10	<3.1E+3	<9.6
Te	[1.13E+4]	[1.17E+4]	[104]	<6.6E+2	<5.9	<6.4E+2	<5.7
Ti	[4.50E+2]	[3.91E+2]	[87]	<4.5E+1	<10	<4.4E+1	<9.7
Tl	<3.5E+3	<3.5E+3	<1.0E+2	<4.2E+2	<12	<4.0E+2	<12
V	[1.19E+3]	[1.17E+3]	[98]	<8.7E+1	<7.3	<8.4E+1	<7.1
W	1.38E+5	1.41E+5	102	<2.8E+2	<0.20	<2.7E+2	<0.20
Y	[5.63E+2]	[5.52E+2]	[98]	<4.0E+1	<7.1	<3.8E+1	<6.8
Zn	5.03E+3	3.70E+3	73.6	1.94E+3	38.5	1.78E+3	35.5
Zr	[2.70E+3]	[2.53E+3]	[94]	<1.1E+2	<4.2	<1.1E+2	<4.0

(a) Opportunistic analytes are reported for information only; QC requirements did not apply to these analytes.

Notes:

Bracketed results were greater than the MDL but less than the EQL. The less-than values (<) indicate that the results were less than the MDL: reported values were the instrument detection limits multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.

Data were derived from previous report (Fiskum et al. 2006b).

Table B.4. AN-102 Radionuclide Load and Fractionation to the Effluent and Eluate

Radionuclide[a]	AN-102 Actual Waste						
				Resin B Eluate		Resin A Eluate	
	Feed	Effluent	Recovery	Eluate	Recovery	Eluate	Recovery
	µCi	µCi	%	µCi	%	µCi	%
^{60}Co	3.92E+1	4.74E+1	121	<3.E+0	<8.7	<2.E-1	<0.5
^{137}Cs	3.33E+5	3.06E+0	9.18E-4	3.76E+5	113[b]	8.08E+3	2.43
^{238}Pu	4.48E-1	3.99E-1	89.1	5.45E-3	1.22	1.81E-3	0.405
$^{239+240}$Pu	1.26E+0	1.14E+0	90.2	1.08E-2	0.858	6.33E-3	0.502
^{241}Am	2.42E+1	2.35E+1	97.0	2.29E-2	0.095	1.39E-2	0.0574
^{242}Cm	8.99E-2	8.78E-2	97.7	<2.E-4	<0.2	<1.1E-4	<0.1
$^{243+244}$Cm	1.15E+0	1.01E+0	87.9	1.34E-3	0.117	3.27E-3	0.286
Total alpha	1.88E+1	1.96E+1	104	<1.E-1	<0.7	<1.E-1	<0.7
Sum of alpha	2.71E+1	2.61E+1	96.1	4.07E-2	0.149	2.54E-2	0.0937

(a) Reference date is 8/15/05.

(b) Includes fraction from AP-101 processing.

Data were derived from previous test report (Fiskum et al. 2006b).

Table B.5. Spent Resin Analyte Recoveries Relative to All Processed Feeds

Analyte(a)	Analyte Recoveries			
	Resin A		Resin B	
	Total mass	Fraction of All Feeds	Total mass	Fraction of All Feeds
	µg	%	µg	%
Ag	[9.1]	[0.52]	[9.8]	[0.56]
As	<19	<0.29	[20]	[>0.31]
Ba	[4.2]	[0.17]	[2.8]	[0.11]
Cd	[1.7]	[0.0027]	[2.0]	[0.0033]
Cr	986	0.192	983	0.192
K	<1008	<0.0012	<998	<0.0012
Mn	[0.85]	[0.063]	[0.96]	[0.071]
Na	[197]	[3.3E-5]	[453]	[7.5E-5]
Ni	76.1	0.019	[28]	[0.0071]
Pb	[28]	[0.017]	[25]	[0.016]
Se	[26]	[0.27]	[28]	[0.29]
Sr	[1.3]	[4.9E-4]	[1.7]	[6.4E-4]
Th	<220	na	<218	na
U KPA	564	0.68	295	0.354
Rb-85	[1.5E-2]	[1.1E-4]	[1.7E-2]	[1.3E-4]
Cs-133	0.0415	1.65E-4	0.345	1.37E-3
Opportunistic Analytes				
Al	[55]	[2.2E-4]	[80]	[3.1E-4]
B	49.6	0.057	64.7	0.074
Be	<0.11	<0.007	<0.11	<0.0067
Bi	[29]	[0.29]	[25]	[0.25]
Ca	205	0.071	93.4	0.032
Co	[21]	[0.40]	[7.4]	[0.14]
Cu	204	0.67	86.4	0.282
Dy	<5.9	<0.26	<5.8	<0.25
Eu	<1.9	<0.17	[1.9]	[0.17]
Fe	[20]	[0.11]	[27]	[0.14]
La	<8.2	<0.16	<8.1	<0.16
Li	<6.0	<0.11	<6.0	<0.11
Mg	<15	na	<15.3	na
Mo	[25]	[0.028]	46.6	0.051
Nd	<38	<0.15	<38	<0.15
P	[35]	[0.0013]	[58]	[0.0021]
Pd	<102	<0.17	<102	<0.17
Rh	<47	<0.16	<47	<0.16
Ru	<12	<0.03	[13]	[0.037]
Sb	[25]	[0.33]	[26]	[0.34]
Si	<31	<0.01	<31	<0.006
Sn	[267]	[0.17]	[200]	[0.13]

Table B.5. (Contd)

Analyte[(a)]	Analyte Recoveries			
	Resin A		Resin B	
	Total recovered	Fraction of All Feeds	Total recovered	Fraction of All Feeds
	μg	%	μg	%
Te	<26	<0.14	[29]	[0.15]
Ti	<1.8	<0.21	<1.8	<0.21
Tl	<17	na	<17	na
V	<3.5	<0.19	<3.4	<0.18
W	[63]	[0.030]	120	0.057
Y	<1.6	<0.20	<1.6	<0.20
Zn	17.4	0.07	[13]	[0.051]
Zr	[31]	[0.41]	[11]	[0.14]
Bracketed results were greater than the MDL but less than the EQL. The less-than values (<) indicate that the results were less than the MDL: reported values were the instrument detection limits multiplied by the sample dilution factor. Overall errors for values greater than the EQL were estimated to be within 15%. Errors for values less than the EQL but greater than the MDL were likely to exceed 15%.				

Table B.6 Spent Resin Radionuclide Recoveries Relative to Actual Waste Feeds

Analyte	Analyte Recoveries			
	Resin A		Resin B	
	Total recovered	Fraction of All Feeds	Total recovered	Fraction of All Feeds
	μCi	%	μCi	%
^{60}Co	2.30E-1	0.525	5.72E-2	0.131
^{137}Cs	1.03	2.01E-4	10.7	2.10E-3
^{238}Pu	1.05E-3	0.203	4.93E-4	0.095
$^{239+240}$Pu	5.02E-3	0.355	1.81E-3	0.128
^{241}Am	7.94E-4	0.00325	7.04E-4	2.88E-3
^{242}Cm	<3.E-5	<3.E-2	<3.E-5	<3.E-2
$^{243+244}$Cm	<6.E-5	<5.E-3	<6.E-5	<5.E-3
Total alpha	5.43E-3	0.0277	2.72E-3	0.0139
Sum of alpha	6.89E-3	0.0250	3.04E-3	0.0110
Reference date is 8/15/05.				

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