PNWD-3266
WTP-RPT-053, Rev 0

Small Column Ion Exchange Testing of SuperLig® 644 for Removal of ^{137}Cs from Hanford Waste Tank 241-AZ-101 (Envelope B)

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April 2003

WTP
Project
Report

Prepared for Bechtel National, Inc.
under Contract No. 24590-101-TSA-W0000-0004

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Test specification: 24590-PTF-TSP-RT-01-002, Rev. 1
Test plan: TP-RPP-WTP-111, Rev. 0
Test exceptions: None
R&T focus area: Pretreatment
Test Scoping Statement(s): B-44

Battelle, Pacific Northwest Division
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COMPLETENESS OF TESTING

This report describes the results of work and testing specified by Test Specification 24590-PTF-TSP-RT-01-002, Rev. 1 and Test Plan TP-RPP-WTP-111, Rev. 0. 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, Manager Date
WTP R&T Support Project

_____ _____

Research and Technology Manager Date

Summary

The U. S. Department of Energy is tasked with the disposition of high-level radioactive waste stored at the Hanford site. The waste is to be vitrified following specific pretreatment processing, separating the waste into a small-volume high-activity waste fraction, and a large-volume low-activity waste fraction. The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline process for ^{137}Cs removal from Hanford high-level tank waste is ion exchange. The current pretreatment flowsheet includes the use of Cs-selective, elutable, organic ion exchange material, SuperLig® 644 (SL-644), for Cs removal from the aqueous waste fraction. This material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. SL-644 has been shown to be effective in removing Cs from a variety of Hanford tank wastes.

Battelle, Pacific Northwest Division (PNWD) was contracted to perform Cs ion exchange studies under contract 24590-101-TSA-W000-0004. The Cs ion exchange activities are defined in Appendix C of the *Research and Technology Plan*[a] under Technical Scoping Statement B-44. The Cs ion exchange studies are to verify design and operating parameters for plant-scale ion exchange systems. Test results will also be used to validate ion exchange models.

Objectives

The Cs ion exchange test objectives were to develop load and elution breakthrough profiles using waste from Hanford tank 241-AZ-101 (AZ-101, Envelope B); produce and characterize the Cs eluate for use in evaporation and vitrification tests; remove ^{137}Cs from the AZ-101 matrix to meet low-activity waste (LAW) vitrification criteria; and develop batch-distribution coefficients for AZ-101. The final effluent was to contain <0.339 μCi ^{137}Cs/mL, based on a 5 wt% waste Na_2O loading in the waste glass. Enough AZ-101 was available to perform two independent ion exchange column tests. All testing objectives were met except for the second process cycle, where the ^{137}Cs effluent concentration exceeded the minimum Cs-removal threshold by a factor of two.

Conduct of Test

This report summarizes testing of the SL-644 in batch-contact studies and in a dual small-column system with AZ-101.[b] Batch contacts were performed with the waste at three Cs concentrations at a phase ratio of 100 mL/g (liquid volume to exchanger mass) with SL-644. Ion exchange processing was conducted with two small columns in series with resin bed volumes (BVs) of nominally 10.3 mL (L/D[c] = 4.2) during the conditioning phase with 0.25 M NaOH, and 9.5 mL (L/D = 3.9) during the AZ-101 loading phase. Proper functioning of the ion exchange apparatus and resin beds had initially been tested with an AW-101 simulant. The resin beds had then been used to process 1.2 L of AP-101 diluted feed (an Envelope A waste feed), 0.75 L of AN-102 (an Envelope C waste feed) combined with wash and leachate solutions of C-104 solids, and 1.07 L of AZ-102 concentrated to 4.6 M Na (an

(a) Research and Technology Plan, 24590-WTP-PL-RT-01-002, Rev. 1, April 2002, S. Barnes, R. Roosa, R. Peterson, Bechtel National, Inc., Richland, WA.
(b) AZ-101 contained 4.85 M Na, and 1645 μCi ^{137}Cs/mL.
(c) L/D, equal to length over diameter, is the resin bed aspect ratio.

Envelope B waste feed). Two tests were conducted with AZ-101. The first test AZ-101 waste volume processed was 1.16 L, corresponding to 122 BVs, at 1.5 BV/h. The second test processed 1.05 L corresponding to 96 BVs at 2.7 BV/h. All ion exchange process steps were tested including resin-bed preparation, loading, feed displacement, water rinse, elution, eluant rinse, and resin regeneration.

Results and Performance Against Objectives

Table S.1 summarizes the batch-contact and column performance data. The batch-contact tests resulted in a predicted Cs distribution coefficient (K_d) of 520 mL/g in the feed condition (Na/Cs mole ratio of 1.08 E+4). Using a bed density of 0.239 g/mL in the 0.25 M NaOH regeneration condition (first test),[(a)] the Cs λ value (column distribution ratio) was predicted to be 124 BVs. The Cs λ value represents a measure of the effective capacity of the SL-644 resin; the higher the λ value, the higher the loading capacity.

Table S.1. Summary of Performance Measures

Flow-rate (BV/h)	Test	Interpolated and Extrapolated 50% Cs breakthrough, BV	Composite DF[(1)]	Maximum DF[(2)]	K_d, mL/g (feed condition)	Predicted Cs λ, BV
1.5	1	122	25,300	2.7E+5	520	124
2.7	2	110	2,240	5.7E+4	520	119[(3)]

(1) The decontamination factor (DF) was calculated by dividing the feed Cs concentration by the composite effluent Cs concentration. The required contract DF is 4840.
(2) The maximum DF was obtained by dividing the feed Cs concentration by the lowest ^{137}Cs concentration sample from the lead column (at nominally 60 BVs).
(3) The Cs λ value was based on the lead column dry resin-bed density in 0.25 M NaOH. The lead column volume was larger for the second test than for the first test, hence the lower λ value.

Two different load flowrates, 1.5 BV/h and 2.7 BV/h, were tested in the dual small-column system. The measured 50% Cs breakthrough for the first test lead column was interpolated at 122 BVs. The second test 50% breakthrough was extrapolated to be 110 BVs. In both tests, no Cs breakthrough from the lag column was observed. The ^{137}Cs decontamination factor (DF) was based on the ^{137}Cs concentration in the feed relative to the ^{137}Cs concentration in the effluent. The LAW vitrified waste form must be no greater than 0.3 Ci/m^3; this limit was converted to ^{137}Cs concentration in the ion exchange effluent (0.339 μCi/mL) and corresponded to a DF of 4840.[(b)] The first test resulted in an effluent that met the Cs DF requirement; the second test effluent failed to meet the minimum DF. The low DF result for the second run was attributed to a combination of ^{137}Cs bleed-off from the lag column as a result of insufficient baseline elution during the previous test and to the higher process flowrate. The maximum DF was a measure of the best performance that can be expected from this column system. It was calculated relative to the sample containing the lowest ^{137}Cs concentration, i.e., the sample at nominally 60-BVs from the *lead* column.

(a) The second test resin bed density was 0.228 g/mL.
(b) The conversion requires the following assumptions: Envelope B vitrified LAW will contain 5 wt% waste Na_2O, the glass density is 2.66 g/mL, and the waste Na concentration is 4.85 M Na.

For each test, only the lead column was eluted with 0.5 M HNO_3. Two different flowrates, 1 BV/h and 1.8 BV/h, were tested. In each case, a C/C_o of 1% was reached in 11 BVs with >99.9% of the ^{137}Cs contained in the eluate. The peak ^{137}Cs C/C_o values were 105 and 88 (based on 1-BV collection increments of nominally 10-mL) from the first and second test, respectively. The eluates from both tests were combined. The ^{137}Cs concentration in the combined composite eluate was 1.30E+4 μCi/mL, corresponding to a C/C_o of 7.9.

QA Requirements

PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the quality assurance project plan (QAPjP) approved by the RPP-WTP Quality Assurance (QA) organization. This work was conducted to the quality requirements of NQA-1-1989 and NQA-2a-1990, Part 2.7 as instituted through PNWD's *Waste Treatment Plant Support Project Quality Assurance Requirements and Description* (WTPSP) Manual, and to the approved Test Plan, TP-RPP-WTP-111.

PNWD addressed verification activities by conducting an "independent technical review" of the final data report in accordance with 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.

Issues

The baseline elution condition ending at $C/C_o = 0.01$ or 15 BVs eluant (whichever is greater) was insufficient. Incompletely eluted ^{137}Cs from previous tank waste processing continued to bleed from the lag column into the effluent product. The bleed-off from the lag column (resulting from insufficient elution or increased process flowrate) during the second AZ-101 test resulted in the failure of the product effluent to meet the Cs decontamination requirement. Additional elution will likely be required to address this issue. Additional elution testing will be needed to determine the relative contribution of flowrate and Cs bleed, and the requirements for sufficient elution.

Elution flowrate at nearly twice the baseline operating design for Envelope B was shown to provide virtually equivalent Cs elution performance. This suggests that the processing time may be reduced or that the elution volume can be increased without sacrifice of processing time.

The load flowrate at nearly twice the baseline operating design for Envelope B was shown to provide similar lead column Cs load performance (similar Cs breakthrough onset and breakthrough slope) as the baseline operating design. This indicates that it may be possible to increase the feed processing flowrate. (Additional testing will be needed to remove the uncertainty attributed to inadequate baseline Cs elution.)

This test resulted in the highest chloride concentration found for the eluate using these ion exchange columns. Approximately 19% of the feed chloride was found in the eluate. Chloride can volatilize during evaporation, leading to corrosion problems in the offgas processing system. High chloride concentration can also lead to salt formation in the melter.

Terms and Abbreviations

AN-102/C-104	AN-102 tank waste mixed with C-104 wash and leachate solutions, then evaporated to 4.8 M Na
AP-101DF	AP-101 tank waste diluted to 4.97 M Na
AZ-102C	AZ-102 tank waste evaporated to 4.6 M Na
ASR	analytical services request
ASTM	American Society for Testing and Materials
AV	apparatus volume
BV	bed volume
C/C_o	analyte concentration in column effluent divided by analyte concentration in feed
CMC	Chemical Measurement Center
DF	decontamination factor
DI	deionized
DOE	U.S. Department of Energy
ED3A	ethylenediaminetriacetic acid
EDTA	ethylenediaminetetraacetic acid
F	furnace
F-factor	ratio of dry resin mass over wet resin mass
FMI	Fluid Metering, Inc., Syosset, NY
GC-FID	gas chromatography/flame ionization detector
GEA	gamma energy analysis
HEDTA	N-(2-hydroxyethyl)ethylenediaminetriacetic acid
HLW	high-level waste
HP	hot persulfate method
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
IDA	iminodiacetic acid
IDL	instrument detection limit
KPA	kinetic phosphorescence analysis
λ	column distribution ratio
LAW	low-activity waste
L/D	length over diameter ratio

meq	milli-equivalents
MRQ	minimum reportable quantity
NA	not applicable
NIDA	nitrosodiacetic acid
NMRQ	no minimum reportable quantity
NPT	National Pipe Thread
NQARD	Nuclear Quality Assurance Requirements and Description
NTA	nitrilotriacetic acid
PNWD	Battelle, Pacific Northwest Division
QARD	Quality Assurance Requirements and Description
ρ	dry bed density
RPP-WTP	River Protection Project-Waste Treatment Plant
RPL	Radiochemical Processing Laboratory (PNWD facility)
SRTC	Savannah River Technology Center
TC	total carbon
TIC	total inorganic carbon
TIMS	thermal ionization mass spectrometry
TOC	total organic carbon

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1.0 Introduction

The U. S. Department of Energy (DOE) plans to vitrify tank wastes at the Hanford Site in preparation for permanent disposal. Before vitrification, tank wastes will be divided into low-activity and high-activity fractions through specific pretreatment processes. The pretreatment flowsheet for the Hanford high-level tank wastes includes the use of SuperLig® 644 (SL-644) material for ^{137}Cs removal from the aqueous waste fraction. The SL-644 is a Cs-selective, elutable, organic ion exchanger and has been shown to be effective in removing Cs from a variety of Hanford tank wastes [1-10].(a) The SL-644 resin has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT (IBC).

This report summarizes batch-contact studies and dual small-column testing of the SL-644 ion exchange material.(b) The matrix for these tests was Envelope B 241-AZ-101 Hanford tank waste (hereafter referred to as AZ-101). Two ion exchange processing tests were conducted varying the feed flowrate and the eluant flowrate. The ion exchange process steps tested include resin-bed preparation, loading, feed displacement, deionized (DI) water rinse, elution, and resin regeneration.

The objectives of this work were to:

- measure distribution coefficients (K_ds) as a function of Na/Cs mole ratios for SL-644 in the AZ-101 matrix
- demonstrate ^{137}Cs decontamination from tank waste sample AZ-101 and provide a Cs-decontaminated sample for downstream process testing (i.e., ^{99}Tc removal, low activity waste [LAW] melter feed testing, and LAW vitrification)
- provide Cs eluate for downstream process testing (i.e., evaporation and high-level waste [HLW]) vitrification
- develop Cs load and elution profiles
- demonstrate the effectiveness of all SL-644 ion exchange process steps, including loading, feed displacement, DI water rinse, elution, and resin regeneration.

(a) Reference callouts have been numbered and enclosed in square brackets.
(b) Testing was conducted according to Test Specification "Tank 241-AZ-101 and 241-AZ-102 Ion Exchange Test Specification," 24590-PTF-TSP-RT-01-002, Rev. 1, James Toth, Bechtel National Inc., October, 2001 and Test Plan "Tank 241-AZ-101 and 241-AZ-102 Waste Sample Ion Exchange Testing," TP-RPP-WTP-111, Rev. 0, D. L. Blanchard, November 5, 2001.

2.0 Experimental

This section describes materials, experimental approach to batch-contact tests and column tests, sample analyses, and calculations. Experimental data were recorded in specific test instructions (as identified) and analytical reports. All raw data are maintained in the Project File 42365 at Battelle, Pacific Northwest Division (PNWD).

2.1 SL-644 Resin

The SL-644 resin was obtained from IBC production batch number 010319SMC-IV-73, prepared at IBC on 3/19/02. This material batch exhibited a black-red appearance peppered with light-brown specks. It was received from the vendor as a dry, granular, free-flowing material in a 1-L polyethylene bottle with an approximately 32% gaseous headspace. There was no indication that this headspace was filled with nitrogen or other inert gas, and no attempt was made to exclude air during storage. The as-received resin form was not identified by the vendor; it was found to contain potassium salts [7]. Before sampling SL-644 from the IBC-supplied plastic bottle, the bulk resin bottle was turned several times to produce a well-mixed material. Sub-samples were removed immediately after mixing. Table 2.1 reproduces the sieve results of the resin batch that was previously reported [7]. The 212- to 425-μm particle-size dry-sieved fraction was used in the ion exchange columns.[(a)] This fraction represented 24 wt% of the as-received material. The average particle size, expanded in 3 M NaOH-2 M $NaNO_3$-0.1 M KNO_3, corresponded to ~540-μm in diameter [7]. As a general rule, the column diameter should be 20 times greater than the resin particle diameter to minimize wall effects [11]. Given the diameter of the column at 1.46 cm, the column diameter was estimated to be 27 times the calculated average diameter of the 212- to 425-μm dry-sieved resin particles expanded in caustic solution.

Table 2.1. Dry Particle-Size Weight-Percent Distribution of As-Received SL-644

Sieve Size[(1)]	Particle Size, (μm)	010319SMC-IV-73, wt %
18	>1000	0.06
30	600 - 1000	37.27
40	425 - 600	38.23
50	300 - 425	18.01
70	212 - 300	6.08
100	150 - 212	0.26
140	106 - 150	0.06
>140	<106	0.03
(1) U. S. standard sieve size corresponding to ASTM E-11 specification.		

Properties of the 212- to 425-μm 010319SMC-IV-73 SL-644 resin have been previously reported [7], and Table 2.2 reproduces selected properties. The F-factor is the ratio of the dry mass of exchanger to the initial mass of the exchanger and was determined at the same time the batch-contact samples and column-resin fractions were weighed. The F-factor was obtained by drying approximately 0.5 g resin, under

(a) This particle size distribution was used successfully in AW-101 simulant testing. The as-received particle size distribution was shown not to meet the Cs decontamination specification [7].

vacuum, at 50°C to constant mass. The F-factor was determined on the H-form and the as-received form of the resin. The F-factor for the Na-form of the resin was performed differently because of stability problems observed in prior tests on the Na-form of resin [12]. Drying to constant mass under vacuum at ambient temperature was considered adequate for removing water from the Na-form resin.(a) The L-factor represents the fractional mass remaining after washing the as-received resin form with 0.5 M HNO_3 and DI water, and correcting for residual water content as described above. The I_{Na} represents the fractional mass gain upon conversion from the H-form to the Na-form, correcting for water content as described above.

Table 2.2. SL-644 Properties

Property	010319SMC-IV-73
Bulk density, g/mL	0.74
F-factor, as-received	0.877
L, conversion to H-form, fractional mass remaining	0.538
F-factor, H-form(1)	0.762
I_{Na}, fractional mass gain from H-form to Na-form	1.25
(1) Stored for 1 year in the H-form, used in batch-contact calculations.	

An aliquot of the sieved resin fraction was washed on 3/24/01 (approximately 5 days after production) by contacting three times with 0.5 M HNO_3 followed by four contacts with DI water. The resin was then air-dried and stored in a polyethylene bottle for nearly one year before sub-sampling for batch-contact testing. The F-factor, 0.762, was determined on the stored H-form of SL-644 at the same time aliquots were taken for the batch-contact test.

2.2 AZ-101 Feed

The AZ-101 sample receipt, mixing, subsampling, analysis, and filtration were reported separately [13 and 14, respectively]. The total volume of AZ-101 available for Cs ion exchange and batch-contact processing was about 2.5 L. Table 2.3 summarizes the AZ-101 feed composition. Most analytical results were reported from Analytical Services Request (ASR) 6306, Radiochemical Processing Laboratory (RPL) sample number 02-0909 taken immediately before ion exchange processing. Other results were taken from ASR 6193, RPL # 01-1844, representing the homogenized supernatant that was previously reported [13]. Analyte results common to both ASRs were in good agreement. The feed ^{90}Sr concentration was taken from ASR 6284, RPL # 02-0822, which was sampled and analyzed after filtration.

Total carbon (TC), total inorganic carbon (TIC or carbonate), and total organic carbon (TOC) are reported here and elsewhere for two different analytical methods: hot persulfate oxidation (HP) and furnace oxidation (F). The differences in the two methods are reflective of the ease with which various organic constituents (and carbonates) oxidize in the given method. Phosphate was reported based on P determination by inductively-coupled plasma-atomic emission spectrometry (ICP-AES) and on PO_4 determination by ion chromatography (IC).

(a) After initial drying at ambient temperature under vacuum to constant mass, the resin was heated to 50°C. The heated product appeared (visual inspection) to have degraded, thus potentially nullifying subsequent mass measurements.

Table 2.3. Composition of AZ-101 (Envelope B)

Cations	M	Anions	M
Na	4.85 E+0	AlO_2^- (2)	2.09 E-1
K	1.15 E-1	F^-	5.79 E-2
Cs (1)	4.48 E-4	Cl^-	2.03 E-3
As	[1.2 E-4]	CrO_4^{-2} (2)	1.21 E-2
Ba	[2.1 E-6]	NO_2^-	1.54 E+0
Ca	<2 E-4	NO_3^-	9.92 E-1
Cd	<4 E-6	OH^-	6.8 E-1
Co	<2 E-5	PO_4^{-3} (2) (ICP-AES)	1.64 E-2
Cu	<1 E-5	PO_4^{-3} (IC)	1.68 E-2
Fe	[4.7 E-5]	SO_4^{-2}	1.75 E-1
Li	[1.3 E-4]	$C_2O_4^{-2}$	1.79 E-2
Mo	9.75 E-4	**Radionuclides**	**(μCi/mL)**
Ni	<2 E-5	^{60}Co	<7 E-2
Pb	[1.5 E-5]	^{90}Sr	[1.19 E+0](4)
Sn	[4.7 E-4]	^{99}Tc	3.27 E-1(3)
U (Uranyl)(2)	9.33 E-5	$^{99}TcO_4$	3.77 E-1(3)
V	[2.9 E-5]	^{134}Cs	7.51 E+0
W	[3.0 E-4]	^{137}Cs	1.64 E+3
Mole ratios		^{154}Eu	<2 E-1
Na/Cs	1.08 E+4	^{238}Pu	3.44 E-4(3)
K/Cs	2.57 E+2	$^{239+240}Pu$	1.81 E-3(3)
Density	**g/mL**	^{241}Am	1.05 E-4(3)
Density	1.224 T = 24°C	$^{243+244}Cm$	4.18 E-5(3)
Carbon	**M**	**Carbon**	**M**
CO_3^{2-} (F)	4.9 E-1(3)	CO_3^{2-} (HP)	8.2 E-1(3)
TOC (F)	1.2 E-1(3)	TOC (HP)	4.2 E-2(3)
TC (F)	6.1 E-1(3)	TC (HP)	8.7 E-1(3)

(1) Cs isotopic analysis resulted in 51.85 wt% 133Cs, 16.9 wt% 135Cs, 31.25 wt% 137Cs.
(2) Al, Cr, and P were determined by ICP-AES. U was measured by KPA. The ionic forms were assumed on the basis of waste chemistry.
(3) Reported in ASR 6193, supernatant characterization results.
(4) Reported in ASR 6284, sample 02-0822, after filtration.
HP = hot persulfate method; F = furnace method
Bracketed results indicate that the analyte concentration uncertainty exceeded ±15%. Less-than (<) results indicate that the analyte concentrations were below the instrument detection limit (IDL); the dilution-corrected IDLs are given.
ASR 6306 RPL # = 02-0909; ASR 6193 RPL # = 01-1844 [13]; ASR 6284 RPL # 02-0822 [14].

2.3 Batch Contacts

Batch contacts were performed using AZ-101 feed at three different Cs concentrations.[(a)] Aliquots of tank waste samples were tested without spiking, and additional aliquots were spiked with 0.5 M $CsNO_3$ to obtain stock solutions of nominally 4 E-3 M and 7 E-3 M Cs. Table 2.4 shows the initial Cs concentrations in the stock contact solutions and the corresponding Na/Cs and K/Cs mole ratios.

Table 2.4. Initial Cs Concentrations Used for the Batch Distribution Tests

Solution	Target initial Cs conc. [M]	Target nominal Na/Cs[(1)] mole ratio	Target nominal K/Cs[(1)] mole ratio
Un-spiked	4.4 E-4	1.1 E+4	2.6 E+2
Cs Spike 1	4.0 E-3	1.2 E+3	2.9 E+1
Cs Spike 2	7.0 E-3	6.9 E+2	1.6 E+1
(1) Na^+ and K^+ are the primary cations that compete with Cs^+ for ion exchange sites with SL-644.			

The batch-distribution tests were performed in duplicate at a phase ratio of approximately 100 mL/g (liquid volume to exchanger mass). Typically, 0.07 g of exchanger were contacted with 7 mL of feed. The exchanger mass was determined to an accuracy of 0.0002 g. The waste volume was transferred by pipet, and the actual volume was determined by mass difference with an accuracy of 0.0002 g and the solution density. Samples were agitated with an orbital shaker for approximately 48 h. The temperature was not controlled but was generally constant at 23 to 27°C during the two days of contact.

All Cs-distribution measurements were determined by measuring ^{137}Cs on both the stock solution (initial concentration) and the contacted solution (final concentration). Initial ^{133}Cs concentrations were confirmed by inductively-coupled plasma mass spectrometry (ICP-MS).

The batch distribution coefficient, K_d (with units of mL/g), was determined using the following relationship:

$$K_d = \frac{(C_o - C_{eq})}{C_{eq}} * \frac{V}{M * I_{Na} * F} \qquad (2.1)$$

where C_o = the initial ^{137}Cs concentration
C_{eq} = the equilibrium ^{137}Cs concentration
V = the volume of the liquid sample (mL)
M = the mass of the ion exchanger (g) (SL-644 H-form)
I_{Na} = fractional mass gain on conversion from H-form to Na-form (1.25) (this factor was set to 1 when calculating the dry-bed density in the H-form or 0.5 M HNO_3 feed).
F = the dried resin mass divided by the initial resin mass, 0.762.

(a) Batch-contact tests for the AZ-101 actual waste (in parallel with the AZ-102C waste) were conducted according to Test Instruction TI-RPP-WTP-164, Rev. 0 *Batch Contact of AZ-101 and AZ-102 Concentrate Tank Waste with SuperLig 644 (Batch ID 010319SMC-IV-73)*, SK Fiskum, February 2002.

The Cs λ value (column distribution ratio) is a function of the dry-bed density (ρ) and feed-condition equilibrium distribution coefficient and was obtained as shown in Equation 2.2.

$$\lambda = K_d * \rho \tag{2.2}$$

The SL-644 dry-bed resin density was determined from the ion exchange processing test. It was calculated from the column dry-bed resin mass (M_c) and the resin bed volume (BV) according to Equation 2.3.

$$\rho = \frac{M_c}{BV} \tag{2.3}$$

where M_c = the dry Na-form SL-644 mass in the resin bed (discussed in Section 2.4)
BV = resin BV in the feed (discussed in Section 3.2.5).

2.4 Column-Run Experimental Conditions

Figure 2.1 shows a schematic of the ion exchange column system. The system consisted of two small columns containing the SL-644 ion exchange material, a small metering pump, three valves, a pressure gauge, and a pressure relief valve. Valves 1, 2, and 3 were three-way valves that could be turned to the flow position, sample position, or no-flow position. Valve 1 was placed at the outlet of the pump and was used to eliminate air from the system, purge the initial volume of the system, or isolate the columns from the pump. Valves 2 and 3 were primarily used to obtain samples and could also be used to isolate the columns from the rest of the system. The columns were connected in series with the first column referred to as the lead column and the second column referred to as the lag column.

The columns were prepared at the Savannah River Technology Center (SRTC) Glassblowing Laboratory. Each column consisted of a 15-cm glass column with a 24/40 taper ground-glass fitting on top and a threaded fitting on the bottom. A polyethylene bushing was installed in the glass-threaded fitting to accommodate ¼-in. stainless steel National Pipe Thread (NPT) fitting. The inside diameter of each column was 1.46 cm, which corresponded to a volume of 1.67 mL/cm. A stainless steel, 200-mesh screen supported the resin bed. The height of the resin bed (and thus shrinkage and swelling) was measured with a decal millimeter scale affixed to the column. The upper section contained four entry ports and a taper joint with screw cap that securely fitted the column. The lead column assembly used a pressure relief valve (10 psi trigger), pressure gauge, and sample inlet; the remaining port was plugged. The lag column assembly used one port for sample entry, and the other three ports were plugged. In both columns, the inlet sample lines extended through the port opening to the top of the column. The connecting tubing was $^1/_8$-in. OD, $^1/_{16}$-in. ID polyethylene. Valved quick-disconnects (Cole Parmer, Vernon Hills, IL) were installed in-line to allow for ease of column switching. An FMI QVG50 pump (Fluid Metering, Inc., Syosset, NY) equipped with a ceramic and Kynar® coated low-flow piston pump head was used to introduce all fluids. The flowrate was controlled with a remotely operated FMI stroke-rate controller. The pump was set up to deliver flowrates from 0.08- to 16-mL/min. The volume actually pumped was determined using the mass of the fluid and the fluid density. The pressure indicated on the pressure gauge remained below 5 psi during all runs. The holdup volume of the entire Cs ion exchange

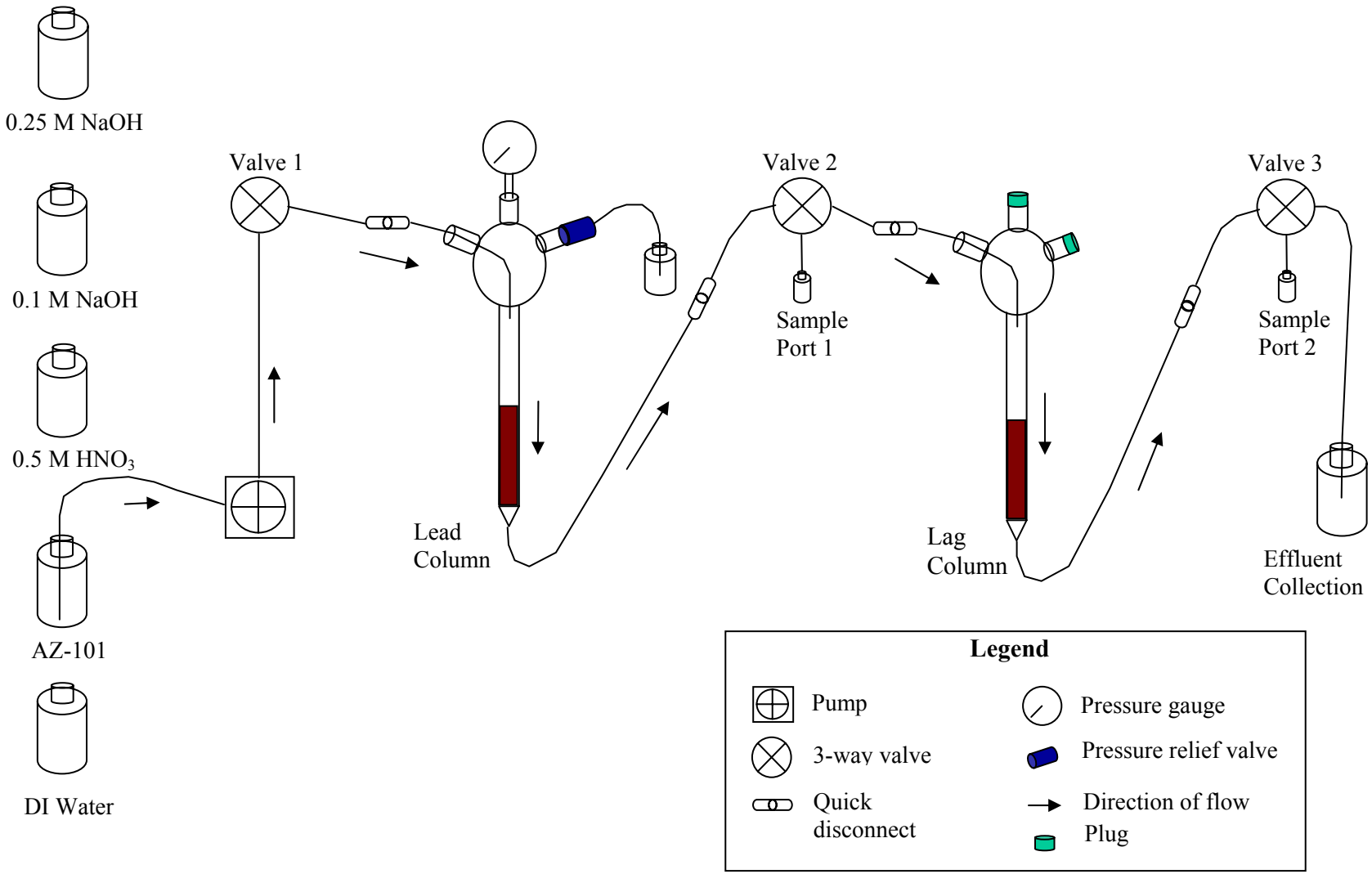


Figure 2.1. Cesium Ion Exchange Column System

system was the summed volume of all fluid-filled parts and was estimated to be 42 mL; the holdup volume for the lead-column section (feed line through the lead-column sample line) was similarly estimated to be 23 mL.

Before installing the system into the hot cell, both of the resin beds were individually cycled through the H-form and back to the Na-form. After the resin cycling, the mass of the Na-form SL-644 (M_c) was calculated to be 2.4 g, on a dry-weight basis, in each column, according to Equation 2.4.

$$M_c = M * L * F * I_{Na} \tag{2.4}$$

where M_c = dry Na-form SL-644 mass in the resin bed
M = SL-644 as-received resin mass
L = fractional mass remaining after washing (0.538)
F = water-loss factor, as-received form (0.877)
I_{Na} = fractional mass gain on conversion from H-form to Na-form (1.25) (this factor was set to 1 when calculating the dry bed density in the H-form or 0.5 M HNO_3 feed).

The entire ion exchange system was used for a full shakedown experiment with AW-101 simulant [7]. Both columns were individually eluted, rinsed, and regenerated. The ion exchange system was then transferred to the hot cell and used to process AP-101DF tank waste [8]. Only the lead column was eluted to a ^{137}Cs concentration C/C_o of 4.2 E-3, which was equivalent to a ^{137}Cs concentration of 0.53 μCi/mL and 1.5E-7 M total Cs. The lead column was then rinsed with DI water, regenerated with 0.25 M NaOH, and again rinsed with DI water. The lag column contained an estimated 35 μCi ^{137}Cs calculated from integrating the lead-column Cs breakthrough. After an 8-week storage period (resin as Na form in DI water), the lead and lag column positions were switched, and 0.75 L of AN-102/C-104 were processed [9]. Again, only the lead column was eluted to a ^{137}Cs concentration C/C_o of 6.3 E-3, which was equivalent to a ^{137}Cs concentration of 1.0 μCi/mL and 3.8E-7 M total Cs. The lead column was then rinsed with DI water, regenerated with 0.25 M NaOH, and again rinsed with DI water. The lag column contained an estimated 0.22 μCi ^{137}Cs calculated from integrating the lead column Cs breakthrough. After a 17-week storage period (Na-form resin in DI water), the lead and lag column positions were switched, and 1.07 L of AZ-102 concentrated to 4.6 M Na were processed [10]. The lead column was eluted to a ^{137}Cs concentration C/C_o of 6.6E-4, which was equivalent to a ^{137}Cs concentration of 1.3 μCi/mL and 3.4E-7 M total Cs. The lead column was regenerated, rinsed, and re-eluted to a C/C_o of 1.83E-4, which was equivalent to 3.7E-1 μCi/mL ^{137}Cs and 9.5E-8 M total Cs. The lag column contained an estimated 1.2E+5 μCi ^{137}Cs calculated from the average of the lead-column Cs-breakthrough integration, integrated Cs recovery in the eluate samples, and Cs recovery in the composite eluate. A summary of the column process cycles is given in Table 2.5.

Table 2.5. Resin History Summary

Tank Waste	Cycle	Load volume, mL (BV)	Lead column	Cs breakthrough % C/C_o	Lag column	Elution	Eluate volume, BV	Final eluate sample ^{137}Cs concentration C/C_o (µCi/mL)	Reference
AW-101 simulant	1	1576 (143)	Column 1	0.99	Column 2	Both columns	12.5	5.52E-3 (trace)	[7]
AP-101	2	118 (112)	Column 1	0.19	Column 2	Lead column only	19.3	4.16E-3 (0.53)	[8]
AN-102/C-104	3	753 (72)	Column 2	1.3E-4 (flat)	Column 1	Lead column only	18.9	6.27E-3 (1.0)	[9]
AZ-102C	4	1072 (105)	Column 1	78	Column 2	Lead column only	14.8	6.65E-4 (1.3)	[10]
		Regenerate then re-elute	Column 1	NA	NA	Lead column only	6.5	1.83E-4 (0.37)	[10]
NA = not applicable									

2.4.1 First AZ-101 Ion Exchange Column Test[a]

The ion exchange assembly was stored for 20 days since the end of the AZ-102C column test. The lead and lag column positions were switched, and the apparatus volume (AV) of DI water was displaced with 0.25 M NaOH before introducing AZ-101 feed. All subsequent processing was performed in the hot cells at a nominal temperature of 24°C. Table 2.6 shows the experimental conditions for each process step. One BV is the lead resin BV in 0.25 M NaOH (10.3 mL) just before loading AZ-101. The bed conditioning, AZ-101 loading, feed displacement, and DI water-rinse steps were conducted by passing these solutions through both resin beds connected in series. The AZ-101 effluent was collected in 13 effluent bottles. The first bottle collected 41 mL, nominally one AV, and consisted primarily of the displaced regeneration solution. The remaining effluent was collected in nominally 10-BV fractions. After ascertaining that the ^{137}Cs concentration met product specifications, the individual effluent fractions were combined.[b] Sampling of the feed displacement and DI water rinse solutions began immediately after switching the feed line into the respective feed solution. The elution was conducted on the lead column only, continuing until 14.3 BVs had been processed through the column. The resin bed was then rinsed, regenerated, and rinsed again. The Cs eluate samples were composited[c] and sampled for analysis.

Table 2.6. Experimental Conditions for AZ-101 Ion Exchange First Test

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[1]	AV[2]	mL	BV/h	mL/min	h	
Two Columns in Series								
DI water displacement	0.25 M NaOH	8.2	2.0	84	2.2	0.39	3.6	24
Loading lead column[3]	AZ-101 Feed	122	NA	1261	1.46	0.25	83.3	24
Loading lag column[4]	AZ-101 Feed	117	NA	1204	1.46	0.25	83.3	24
Feed displacement	0.1 M NaOH	8.2	2.0	84.9	2.8	0.49	2.8	24
Rinse	DI water	8.9	2.2	91	2.9	0.50	3.0	24
Lead Column Only								
Elution	0.5 M HNO_3	14.3	NA	147	1.0	0.18	15.0	24
Rinse	DI water	5.1	2.3	53	3.0	0.52	1.75	24
Regeneration	0.25 M NaOH	4.5	2.0	46	0.88	0.15	4.6	24
Rinse	DI water	4.2	1.9	43	2.6	0.45	1.6	24

(1) BV = bed volume (10.3 mL in 0.25 M NaOH regeneration condition)
(2) AV = apparatus volume (42 mL for columns in series; 23 mL for lead column and 21 mL for lag column)
(3) Start date = 12/3/01
(4) The feed volume through the lag column was reduced because of sampling from the lead column.
NA = not applicable

(a) The first ion exchange processing of the AZ-101 actual waste was conducted according to Test Instruction TI-PNNL-WTP-131, Rev. 0, *Separation of Cesium from Hanford Tank Waste 241-AZ-101 Using the Dual Small-Column SuperLig® 644 Cesium Ion Exchange System*, SK Fiskum, November 2001.
(b) The effluent was composited according to Test Instruction TI-RPP-WTP-150, Rev. 0, *Compositing AZ-101 Cesium Ion Exchange Effluent and Subsequent Sub-Sampling for Analysis*, SK Fiskum, December 2001.
(c) The Cs eluate samples were composited according to Test Instruction TI-RPP-WTP-167, Rev. 0, *Preparing a Composite Solution of the Acid Eluant Samples from AZ-101 Cs Ion Exchange Lead Column: Two IX Processing Runs,* SK Fiskum, March 2002.

2.4.2 Second AZ-101 Ion Exchange Column Test[a]

The AZ-101 load and 0.5 M HNO_3 eluant flowrates were nominally doubled for the second ion exchange test relative to the first AZ-101 test. The assembly was stored for 49 days since the end of the initial AZ-101 column run. The lead and lag column positions were switched, and the AV of DI water was displaced with 0.25 M NaOH before introducing AZ-101 feed. All subsequent processing was performed in the hot cells at a nominal temperature of 26°C. Table 2.7 shows the experimental conditions for each process step where one BV is the lead resin BV in 0.25 M NaOH, 10.9 mL, just before loading AZ-101. The bed conditioning, AZ-101 loading, feed displacement, and DI water rinse steps were conducted by passing these solutions through both resin beds connected in series. The AZ-101 effluent was collected in three effluent bottles. The first bottle collected 41 mL, nominally one AV, and consisted primarily of the displaced regeneration solution. The remaining effluent was collected in one 723-mL fraction (78-BVs, through the lead column) and a 173-mL fraction (additional 17.6 BVs, through the lead column). Sampling of the feed displacement and DI water rinse solutions began immediately after switching the feed line into the respective feed solution. The elution was conducted on the lead column only, continuing until 15.9 BVs had been processed through the column. The resin bed was then rinsed, regenerated, and rinsed again. The Cs eluate samples were composited[b] and sampled for analysis. After subsampling the composite, the first and second test Cs eluates were combined and again sampled for a large suite of analytes supporting follow-on vitrification studies.

Table 2.7. Experimental Conditions for AZ-101 Ion Exchange Second Test

Process step	Solution	Total Volume			Flowrate		Time	T, °C
		BV[1]	AV[2]	mL	BV/h	mL/min	h	
Two Columns in Series								
DI water displacement	0.25 M NaOH	7.4	1.9	80	2.5	0.46	2.9	26
Loading Lead column[3]	AZ-101 Feed	95.9	NA	1046	2.66	0.48	36.7	25 - 27
Loading Lag column[4]	AZ-101 Feed	91.7	NA	999	2.66	0.48	36.7	25 - 27
Feed displacement	0.1 M NaOH	10	2.6	110	3.0	0.55	3.5	26
Rinse	DI water	9.0	2.3	98	2.6	0.48	3.4	26
Lead Column Only								
Elution	0.5 M HNO_3	15.9	NA	173	1.8	0.33	7.5	26
Rinse	DI water	6.4	3.0	70	2.8	0.51	1.7	26
Regeneration	0.25 M NaOH	4.4	2.1	48	0.93	0.17	4.4	27
Rinse	DI water	4.7	2.2	51	2.2	0.41	1.8	27

(1) BV = bed volume (10.9 mL in 0.25 M NaOH regeneration condition)
(2) AV = apparatus volume (42 mL for columns in series; 23 mL for lead column and 21 mL for lag column)
(3) Start date = 1/28/02
(4) The feed volume through the lag column was reduced because of sampling from the lead column.
NA = not applicable

(a) The second ion exchange processing of the AZ-101 actual waste was conducted according to Test Instruction TI-PNNL-WTP-152, Rev. 0, *Second Separation of Cesium from Hanford Tank Waste 241-AZ-101 Using the Dual Small-Column SuperLig® 644 Cesium Ion Exchange System,* SK Fiskum, January 2002.

(b) The Cs eluate samples were composited according to Test Instruction TI-RPP-WTP-167, Rev. 0, *Preparing a Composite Solution of the Acid Eluant Samples from AZ-101 Cs Ion Exchange Lead Column: Two IX Processing Runs,* SK Fiskum, March 2002.

2.5 Column Sampling and Analysis

Table 2.8 shows the sampling and analysis protocol. During the loading phase, small samples (about 2 mL) were collected from the lead and lag columns at nominal 5-BV increments. The feed displacement, DI water rinse, elution, and elution rinse samples were taken at 1-BV increments.

Table 2.8. Sampling Interval and Analyses

Process Step	Frequency		Approximate Sample Size (mL)	Analysis
	Lead Column	Lag Column		
Loading	Every 5 BV	Every 5 BV	2	GEA
Feed displacement	NA	Every 1 BV	10	GEA
Rinse	NA	Every 1 BV	10	GEA
Elution	Every 1 BV	NA	10	GEA
Rinse	Every 1 BV	NA	10	GEA
Composite Samples		**Analyses**		
Effluent composite(1)		GEA, ICP-AES, IC, U, OH^-, Tc^{+7}, density		
Regeneration composite(1)		GEA, ICP-AES, OH^-		
Eluate(1)		GEA, ICP-AES, TOC, IC, U, Pu, Am, Cm, density		
Eluate combined first and second test		GEA, ICP-AES, ICP-MS, TIMS, IC, U, TOC, Hg, NH_3, ^3H, ^{14}C, ^{63}Ni, ^{79}Se, ^{90}Sr, ^{151}Sm, Pu, Am, Cm, total alpha, total beta, wt% solids, wt% oxides, organic anions, chelators, density		

GEA = gamma energy analysis
ICP-AES = inductively-coupled plasma-atomic emission spectrometry
IC = ion chromatography
ICP-MS = inductively-coupled plasma-mass spectrometry
NA = not applicable
OH^- = hydroxide
TOC = total organic carbon
TIMS = thermal ionization mass spectrometry
(1) Analyses were conducted on the first ion exchange test solutions. Only ^{137}Cs was measured on the second test solutions.

The ^{137}Cs concentrations were determined comparatively using a bench-top GEA spectrometer, allowing for rapid sample analysis. Selected sample results were later confirmed by GEA analysis in the Chemical Measurements Center (CMC) analytical laboratory. The effluent composite sample was submitted to the CMC (ASR 6306, # 02-0935) for various analyses: GEA, ICP-AES, IC, U and hydroxide. The eluate samples required dilution before removal from the hot cell to reduce the dose rate from ^{137}Cs. The extent of dilution was determined by mass difference. Once the GEA results were confirmed for the eluate samples, the eluate samples were composited, and a sample of the composite was submitted to the CMC (ASR 6401, # 02-2238) for various analyses: GEA, ICP-AES, TOC, IC, U, Pu, and Am. The eluates from the two different runs were combined, and a large suite of analyses was performed in support of vitrification activities (ASR 6403, # 02-2246). Only the first-test regeneration solution was sampled for ICP-AES, GEA, and hydroxide determination.

The Na and other metal concentrations were determined with ICP-AES. The OH^- concentration was determined by potentiometric titration with standardized HCl. The OH^- concentration was based on the first inflection point of the titration curve. Uranium was determined using kinetic phosphorescence. The pertechnetate concentration was determined using radiochemical separations specific for pertechnetate followed by beta counting. Anions were determined using IC. TOC was determined by silver-catalyzed hot-persulfate oxidation and furnace-oxidation methods. Pu, Am, and Cm were determined, after separations, by alpha spectrometry. Following element-specific separations, ^{3}H, ^{14}C, ^{63}Ni, ^{79}Se, and ^{151}Sm were determined by liquid scintillation counting. The organic anions (oxalate, citrate, formate, gluconate, and glycolate) were determined by two organic IC methods. An experimental derivatization method was used to determine chelators and degradation products: ethylenediaminetetraacetic acid (EDTA), ethylenediaminetriacetic acid (ED3A), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), iminodiacetic acid (IDA), nitrosodiacetic acid (NIDA), and nitrilotriacetic acid (NTA). Derivatization was performed with BF_3/methanol, and the products were analyzed by gas chromatography/flame ionization detector (GC-FID). Additional analytical details are provided in Appendix D.

3.0 Results and Discussion

This section describes the results from batch-contact and column testing of AZ-101 with SL-644.

3.1 Batch-Contact Results

The equilibrium K_d values were calculated according to Equation 2.1 and were based on ^{137}Cs concentrations measured by GEA. The equilibrium Cs concentrations were based on the ^{137}Cs concentrations and the ratio of ^{137}Cs:total Cs determined for the unspiked and spiked solutions. The Na/Cs mole ratios were calculated based on the measured Na and total Cs concentrations in the uncontacted AZ-101. The Na concentration was assumed to be constant for the batch contacts. Since the quantity of H^+ added with the resin was small relative to the moles of Na^+ and OH^- in the contact solution (phase ratio of 100 mL of solution:gram of exchanger), this was a reasonable assumption. In these experiments, the waste solutions were estimated to contain 4.8 meq of OH- (i.e., 7 mL × 0.68 M) and 34 meq of Na^+ (i.e., 7 mL × 4.85 M), while the resin contained 0.15 meq of H^+ (i.e., 0.07 g × 2.2 meq/g).[(a)]

The calculated ^{137}Cs K_ds for AZ-101 are summarized in Table 3.1 and plotted as a function of the equilibrium Na:total Cs mole ratio in Figure 3.1. The best-fit logarithmic regression is shown in equation 3.1.

$$K_d = 221.9 * \ln\left(\frac{[Na]}{[Cs]}\right) - 1541, \; r^2 = 0.988 \qquad (3.1)$$

where: ln = natural log
[Na] = Na molarity (moles/liter)
[Cs] = Cs molarity.

At the nominal feed condition of 1.08 E+4 Na:Cs mole ratio, the SL-644 K_d value is 520 mL/g.

Table 3.1. Cs Equilibrium Distribution Values

Equilibrium Na:Cs mole ratio	K_d, mL/g	λ, BVs (where bed density = 0.239 g/mL)	λ, BVs (where bed density = 0.228 g/mL)
1.30E+5	1120	268	255
1.20E+5	1026	245	234
2.76E+3	159	37.9	36.1
2.70E+3	154	36.8	35.1
1.26E+3	91	21.8	20.8
1.22E+3	85	20.3	19.4

(a) The SL-644 contains 2.2 meq H^+ per gram of H-form resin [15].

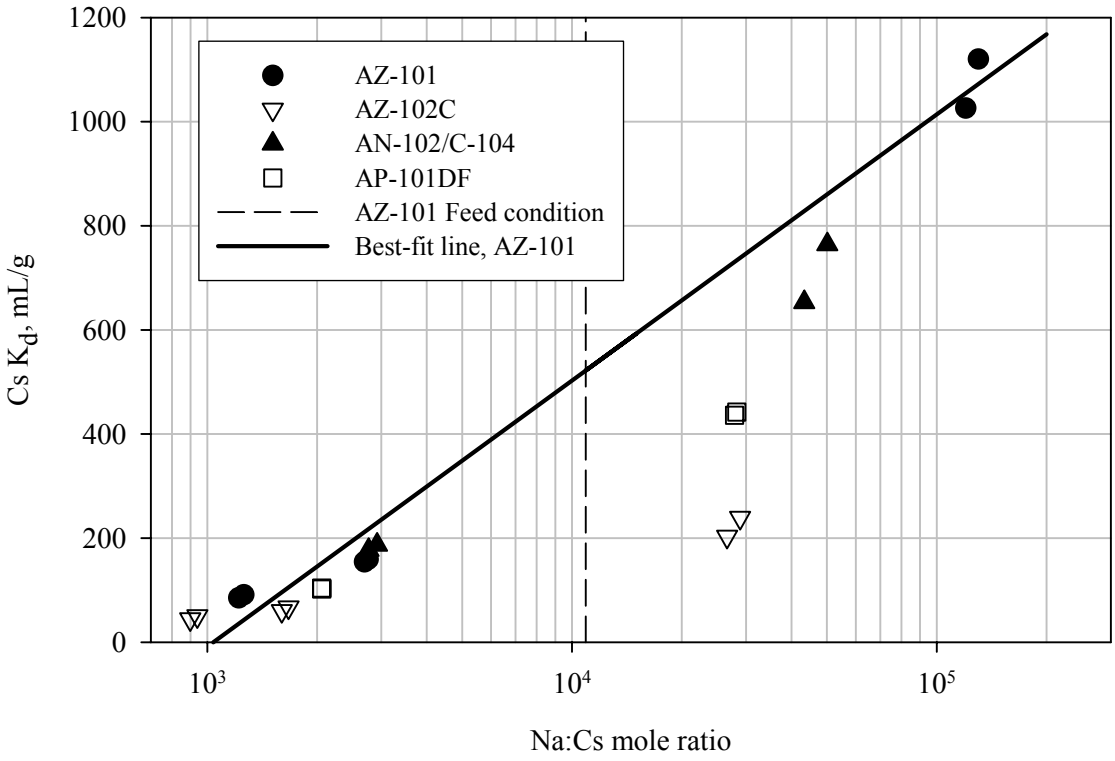


Figure 3.1. ^{137}Cs Distribution Coefficients (K_d) for SL-644 (AZ-101) (T = 24 °C)

For comparison, the K_d values obtained at the lower Na:Cs mole ratio range for AP-101DF [8] and AN-102/C-104 [9], and AZ-102C [10] are also shown. The AZ-101 feed-condition K_d values were clearly higher than similar Na:Cs mole ratios for AP-101DF (260 mL/g) and AZ-102C (170 mL/g).[a]

The calculated dry-bed densities were determined according to Equation 2.3 and are summarized in Table 3.2. The dry-bed density variation between the lead and lag columns was greater than that observed in prior waste-processing tests. The previous test conducted with AZ-102C feed [10] resulted in a resin-bed expansion part way through the load cycle. The BV expansion resulted in a lowering of the bed density. The low-bed-density trend was evidenced in the lag column for the first AZ-101 test and the lead column for the second AZ-101 test.

The Cs λ value was calculated according to Equation 2.2 to be 124 BVs (first test, 0.25 M NaOH regeneration condition) and 119 BVs (second test). These values were the approximate points at which the Cs breakthrough curves were predicted to pass through C/C_o = 50%. Figure 3.2 shows the Cs λ value as a function of the Na:Cs mole ratio for the SL-644 (calculated bed density in 0.25 M NaOH of 0.239 g/mL first test and 0.228 g/mL second test). The coordinate values for these data points are presented in Table 3.1.

(a) The AZ-102C batch contacts were performed in parallel to the AZ-101 batch contacts. The poor performance for the AZ-102C was noted [10]. It may be in part attributed to some of the resin floating in the AZ-102C matrix.

Table 3.2. Dry Bed Density

Property	010319SMC-IV-73, Lead Column	010319SMC-IV-73, Lag Column
First AZ-101 Ion Exchange Test		
Column resin mass, g, as-received	4.17	4.20
Corrected resin mass, M_c, in column, g (Na-form)	2.46	2.48
Corrected resin mass, M_c, in column, g (H-form)	1.97	1.98
Bed volume, 0.25 M NaOH, mL	10.3	10.9
Bed volume, AZ-101, mL	9.5	10.2
Bed volume, 0.5 M HNO_3, mL	7.7	not performed
Dry Bed Density, ρ, in Feed		
0.25 M NaOH, g/mL	0.239	0.228
AZ-101, g/mL	0.259	0.243
0.5 M HNO_3, g/mL	0.256	not performed
Second AZ-101 Ion Exchange Test(1)		
Column resin mass, g, as-received	4.20	4.17
Corrected resin mass, M_c, in column, g (Na-form)	2.48	2.46
Corrected resin mass, M_c, in column, g (H-form)	1.98	1.97
Bed volume, 0.25 M NaOH, mL	10.9	10.5
Bed volume, AZ-101, mL	10.2	9.7
Bed volume, 0.5 M HNO_3, mL	8.4	not performed
Dry Bed Density, ρ_b, in Feed		
0.25 M NaOH, g/mL	0.228	0.234
AZ-101, g/mL	0.243	0.254
0.5 M HNO_3, g/mL	0.236	not performed
(1) Note that the lead and lag columns were switched in position.		

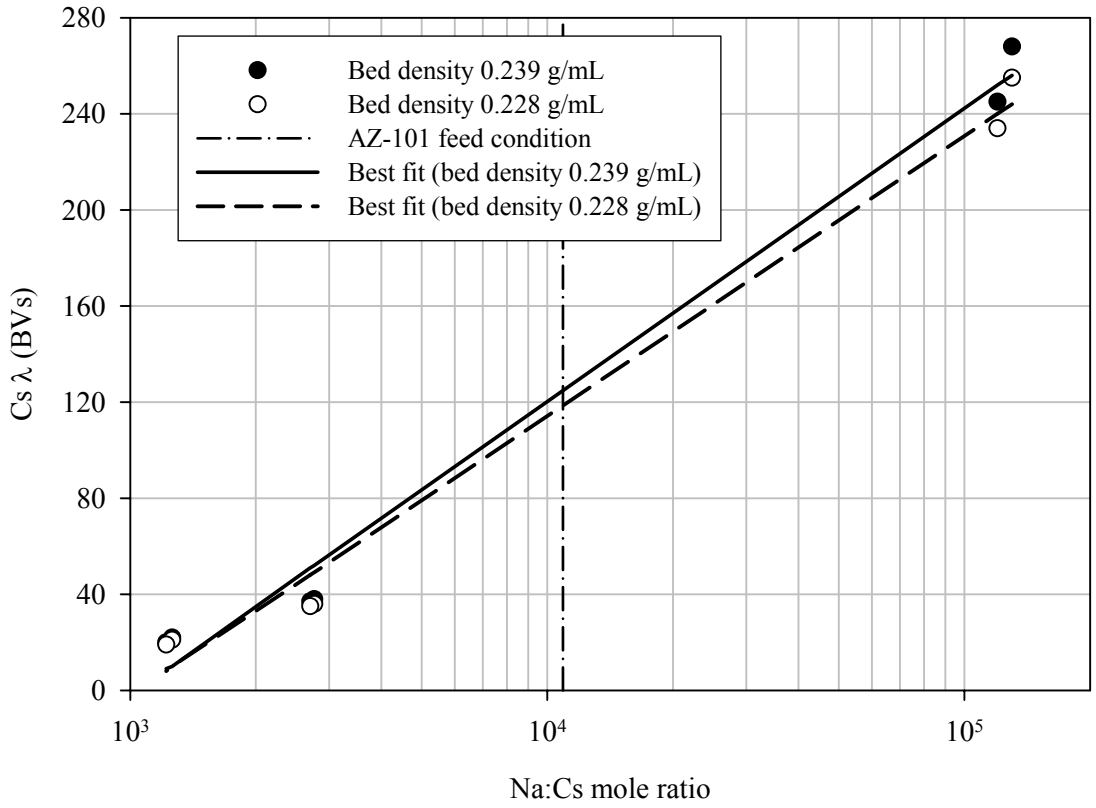


Figure 3.2. Calculated ^{137}Cs λ Values for SL-644 (AZ-101) (T = 25 °C)

3.2 Column Test

The column system used for AZ-101 processing was the same system used for processing the AW-101 simulant, AP-101DF actual waste, AN-102/C-104 actual waste, and AZ-102C actual tank waste. The processing reported herein represents the 5th and 6th processing cycles for these resin beds.

3.2.1 Loading, Feed Displacement, and Rinse

The loading phase was initiated with AZ-101. Approximately 1.0 AV of effluent was initially collected in a separate collection bottle. Most of this effluent was 0.25 M NaOH mixed with some AZ-101 in the ion exchange apparatus. This solution was maintained separately and not mixed with the final Cs-decontaminated AZ-101 effluent composite; thus, most of the apparatus 0.25 M NaOH solution was prevented from mixing with the AZ-101 effluent. The resin beds shrank 6.4% to 7.9% as they converted from the 0.25 M NaOH matrix to the AZ-101 feed.

3.2.1.1 First Ion Exchange Column Test

The lead column for this test was the lag column for AZ-102C processing. It was partially loaded with Cs (0.12 Ci ^{137}Cs). The lag column for this test was the lead AZ-102C column that had been eluted (14.8 BVs), regenerated, and eluted (6.5 BVs) again. The re-elution minimized residual Cs on the AZ-101 lag column.

Table 3.3 and Figure 3.3 summarize the ^{137}Cs effluent concentrations from the lead and lag columns as % C/C_o vs. the BVs of feed processed through each column. The Figure 3.3 abscissa reflects BVs as a function of the resin in the expanded regeneration condition. The C_o value for ^{137}Cs was determined to be 1645 μCi/mL. The C/C_o is plotted on a probability scale. A probability scale is the inverse of the Gaussian cumulative distribution function (characteristic of ideal ion exchange theory) such that a graph of the sigmoidally-shaped Gaussian cumulative distribution function appears as a straight line [16]. The probability scale has a couple of advantages including making low C/C_o data easily readable such that the initial load performance is discernable, and extrapolation to 50% breakthrough can be easily estimated in the sigmoidal region. Figure 3.4 also shows the lead-column load profile on a log-probability scale (again the probability scale results in a straight line if the resin performs according to ideal ion exchange theory). The C/C_o values, determined using the bench-top GEA spectrometer, were generally in good agreement with selected samples independently analyzed by the CMC analytical laboratory. The final two lead-column CMC sample results, however, were ~22 % low relative to the bench-top analysis. Because the CMC values dropped away from the linear nature of the load profile, the bench-top results were given more credibility. A dilution error may have caused the CMC divergence. Processing results and calculations are given in Appendix C, and analytical results are located in Appendix D.

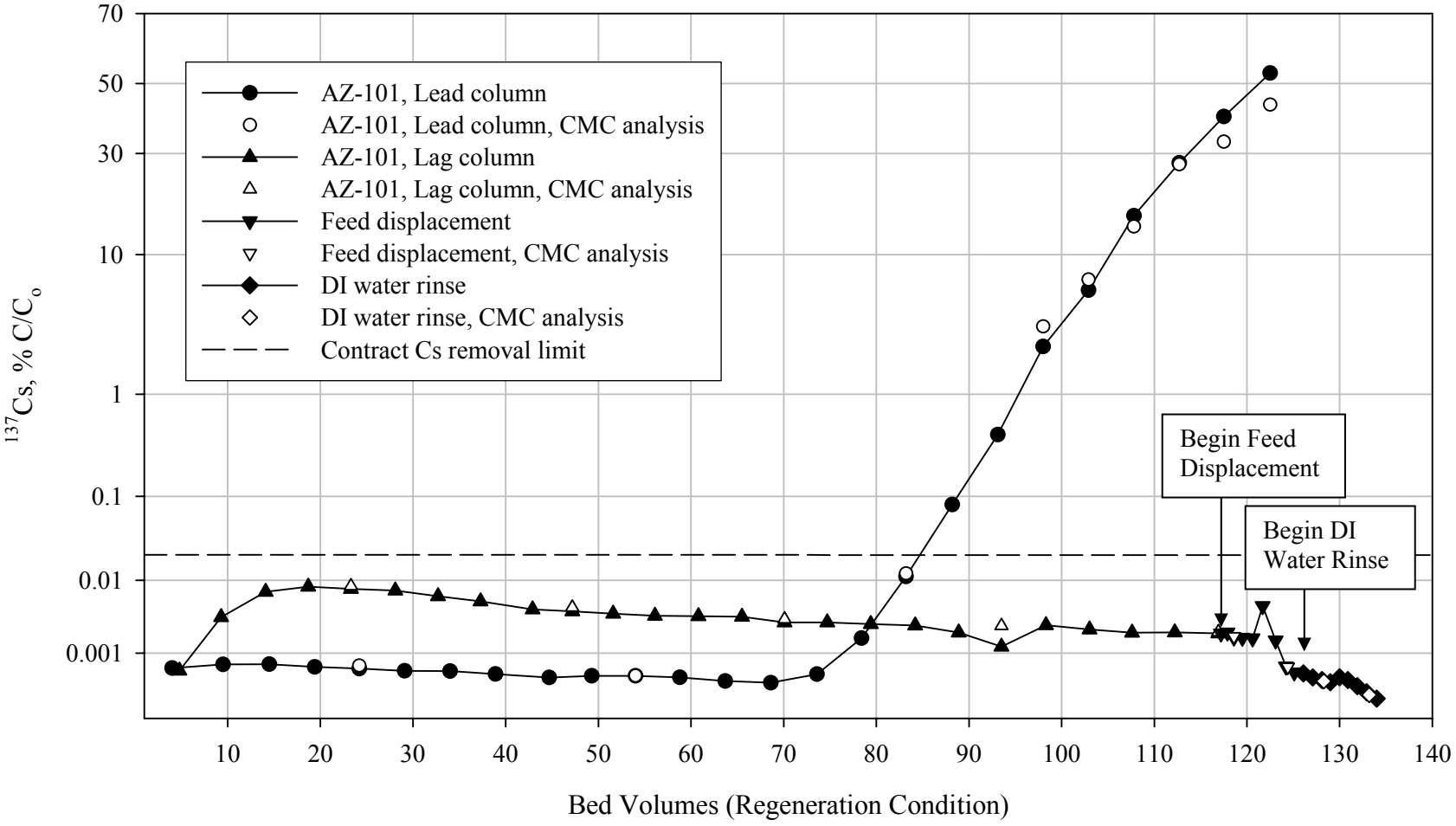
The lead-column load profile resulted in excellent removal of Cs where breakthrough began between 74 and 78 BVs. The breakthrough was linear on a log-probability scale, culminating in 50% C/C_o at approximately 122 BVs (Figure 3.4). This value was essentially equivalent to the Cs λ value predicted from batch-contact studies (124 BVs). The steep load characteristic of this waste (relative to the AP-101 breakthrough curve [8]) was consistent with modeling predictions where steeper breakthrough curves are expected as Cs concentrations increase [17].

The ^{137}Cs concentrations in the lag-column samples were about an order of magnitude higher in ^{137}Cs concentration than those obtained from the lead column through the first 75 BVs processed. This observation was similar to that observed with the AN-102/C-104 processing where ^{137}Cs concentrations in the lag-column samples were 50 times higher than the lead-column samples, a result of ^{137}Cs bleed-off from previous waste processing [9]. The lag column ^{137}Cs bleed-off concentration tapered slowly from 0.136 μCi/mL (C/C_o = 0.0083 %) to 0.032 μCi/mL (C/C_o = 0.0019 %). The bleed-off was fairly constant up to 4.8 BVs of the feed displacement. A slight rise in bleed-off occurred at 4.8 BVs (1.5 AVs) of feed displacement, followed by ^{137}Cs tailing. Introducing water did not significantly change the effluent Cs concentration. The slight rise in % C/C_o during feed displacement may have been caused by the decrease in ionic strength and/or the decrease of hydroxide concentration of the processing solution. The subsequent drop in Cs bleed-off was most likely related to the drop in Na concentration in the rinse solution, where Na acted as a competitor to Cs on the ion exchanger.

Table 3.3. AZ-101 First Test ^{137}Cs Concentration, % C/C_o, as a Function of Processed Bed Volume

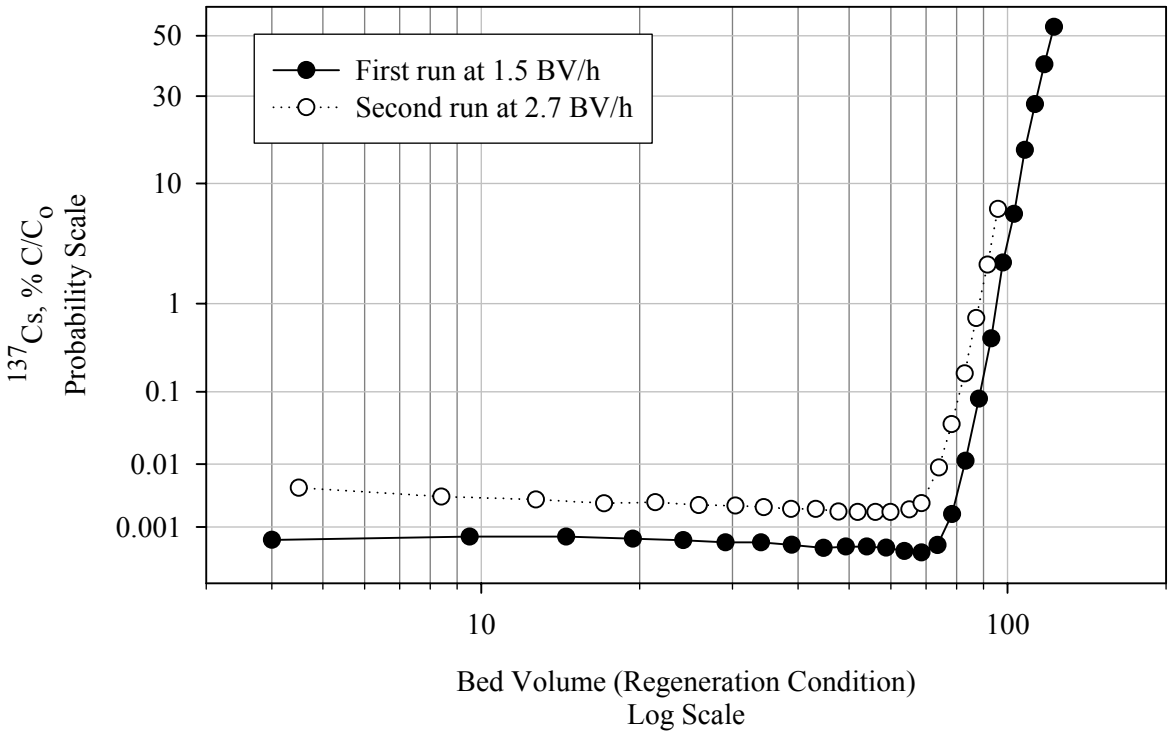
Column Feed	BV	^{137}Cs, % C/C_o	^{137}Cs, % C/C_o CMC Analysis	BV	^{137}Cs, % C/C_o	^{137}Cs, % C/C_o CMC Analysis
	Lead Column			*Lag Column*		
AZ-101	4.8	6.02E-4	--	4.8	5.54E-4	--
	9.5	6.83E-4	--	9.3	3.23E-3	--
	14.5	6.86E-4	--	14.1	7.07E-3	--
	19.4	6.28E-4	--	18.7	8.27E-3	--
	24.2	5.91E-4	6.57E-4	23.3	7.70E-3	8.39E-3
	29.1	5.45E-4	--	28.1	7.33E-3	--
	34.0	5.44E-4	--	32.7	6.17E-3	--
	38.9	4.91E-4	--	37.3	5.28E-3	--
	44.7	4.35E-4	--	42.9	4.11E-3	--
	49.3	4.59E-4	--	47.2	3.88E-3	4.38E-3
	54.0	4.60E-4	4.63E-4	51.6	3.59E-3	--
	58.8	4.37E-4	--	56.1	3.38E-3	--
	63.7	3.84E-4	--	60.8	3.32E-3	--
	68.6	3.62E-4	--	65.5	3.27E-3	--
	73.6	4.88E-4	--	70.1	2.74E-3	3.03E-3
	78.4	1.64E-3	--	74.7	2.73E-3	--
	83.2	1.12E-2	1.22E-2	79.4	2.59E-3	--
	88.2	8.09E-2	--	84.2	2.47E-3	--
	93.1	4.29E-1	--	88.9	1.97E-3	--
	98.0	2.45E+0	3.45E+0	93.5	1.24E-3	2.45E-3
	102.9	6.09E+0	7.11E+0	98.3	2.50E-3	--
	107.8	1.61E+1	1.42E+1	103.0	2.17E-3	--
	112.7	2.76E+1	2.72E+1	107.6	1.96E-3	--
	117.5	4.02E+1	3.31E+1	112.2	1.99E-3	--
	122.5	5.31E+1	4.37E+1	116.9	1.92E-3	2.01E-3
0.1 M NaOH	No data taken from lead column			117.2	1.91E-3	--
				117.9	1.97E-3	--
				118.6	1.68E-3	1.73E-3
				119.5	1.66E-3	--
				120.6	1.63E-3	--
				121.7	4.56E-3	--
				123.1	1.56E-3	--
				124.2	6.40E-4	6.26E-4
				125.1	5.19E-4	--
DI Water	No data taken from lead column			126.1	5.02E-4	--
				127.1	4.37E-4	--
				128.1	3.97E-4	3.81E-4
				129.0	3.68E-4	--
				130.0	4.40E-4	--
				130.9	3.97E-4	--
				131.9	3.20E-4	--
				132.9	2.59E-4	2.36E-4
				134.0	2.05E-4	--

"--" indicates no data were taken.



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425-µm dry particle size
BV = 10.3 mL
^{137}Cs C$_o$ = 1645 µCi/mL
Process temperature = 24°C
Flowrate = 1.46 BV/h
Na concentration = 4.85 M

Figure 3.3. ^{137}Cs Breakthrough Curves for First Test of AZ-101 Tank Waste, Probability Plot



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425-μm dry particle size
Process temperature = 25 to 27°C
BV 1st test = 10.2 mL BV 2nd test = 10.9 mL
^{137}Cs C_o = 1645 μCi/mL Na concentration = 4.85 M.

Figure 3.4. Lead-Column Cs Breakthrough Profile Comparison of AZ-101 Tank Waste

The total quantity of AZ-101 ^{137}Cs processed through the ion exchange system was 2.08 Ci (23.9 mg ^{137}Cs, 76.4 mg total Cs). The ^{137}Cs breakthrough from the lead column was integrated. The lag-column ^{137}Cs loading was assumed to be 100% of the lead-column breakthrough and was calculated at 9.94E+4 μCi (1.1 mg ^{137}Cs, 3.7 mg total Cs), corresponding to 4.8% breakthrough from the lead column. The calculated lead column ^{137}Cs loading was 2.10 Ci, calculated from the total AZ-101 feed ^{137}Cs (2.08 Ci), subtracting the ^{137}Cs breakthrough onto the lag column (0.099 Ci), and adding the Cs remaining on the column from the previous processing with AZ-102C (0.12 Ci) (i.e., 2.08 Ci – 0.099 Ci + 0.12 Ci).

Figure 3.3 also shows the minimum contract ^{137}Cs removal limit. The C/C_o value of 0.0207% (0.339 μCi/mL) corresponds to the contract limit of 0.3 Ci/m^3 for ^{137}Cs in the LAW glass. The C/C_o value corresponding to this limit was determined using the Na concentration of 4.85 M in the AZ-101, the ^{137}Cs feed concentration of 1645 μCi/mL, a 5 wt% waste Na_2O loading and a glass-product density of 2.66 g/mL (see calculations in Appendix A). The lag-column effluent remained below the contract limit; at the highest concentration (20 BVs), the lag column effluent was 40% of the contract limit (C/C_o = 8.27E-3%). The composite lag-column effluent contained 0.0649 μCi/mL, corresponding to 0.0036% (75 μCi ^{137}Cs, 2.75 μg total Cs) of the total Cs processed through the columns.

The decontamination factors (DFs) were calculated on selected effluent samples, and the composite effluent and are summarized in Table 3.4. These may be compared to the contract limit of $C/C_o = 0.0207\%$ (DF = 4840). Sufficient Cs was removed that the ^{137}Cs contract limit was met.

Table 3.4. Selected Decontamination Factors for ^{137}Cs from AZ-101

Sample	Volume, BV	Volume, mL	^{137}Cs Concentration, μCi/mL[(1)]	C/C_o, %	DF
Fifth lead column sample	24.2	249	1.08E-2	6.57E-4	1.52E+5
Final lead column sample	122.5	1261	8.73E+2[(2)]	5.3E+1[(2)]	1.88E+0
Fifth lag column sample	23.3	240	1.38E-1	8.39E-3	1.19E+4
Final lag column sample	116.9	1204	3.30E-2	2.01E-3	4.98E+4
Composite effluent	111.9	1152	6.49E-2	3.95E-3	2.53E+4

(1) The CMC ^{137}Cs uncertainty ranges from 3% to 4% relative error, 1-σ.
(2) The CMC analysis-based ^{137}Cs concentration was lower at 718 μCi/mL and the C/C_o (44%) was lower than the bench-top comparative analysis C/C_o.

The Cs-decontaminated effluent was characterized, and Table 3.5 summarizes the results. Most analyte concentrations were equivalent to the feed concentration within the experimental error of the method (typically ±15%). The Fe and Cl^- concentrations dropped 38% and 65%, respectively, in the effluent relative to the feed. The loss of U was consistent with previous test results.

3.2.1.2 Second Ion Exchange Column Test

The lead- and lag-column positions were switched for the second Cs ion exchange processing. The lead column for this test was the lag column for the first test AZ-101 processing. It was partially loaded with Cs (0.099 Ci ^{137}Cs). The lag column for this test was the first test lead AZ-101 column that had been eluted according to baseline conditions (14.3 BVs) and regenerated.

Table 3.6 and Figure 3.5 summarize the lead- and lag-column ^{137}Cs effluent concentrations as % C/C_o vs. the BVs of feed processed through each column. The BV was based on the lead-column BV in 0.25 M NaOH just before AZ-101 loading (10.9 mL). All processing conditions were equivalent to the previous test processing conditions, except the AZ-101 load flowrate was nearly doubled from 1.46 BV/h to 2.66 BV/h. The process volume was reduced because limited feed volume was available. The % C/C_o values, determined using the bench-top GEA spectrometer, were generally in good agreement with selected samples independently analyzed by the CMC analytical laboratory. Processing results and calculations are given in Appendix C, and analytical results are located in Appendix D.

The lead-column load profile resulted in good removal of Cs from the feed where breakthrough began between 68 and 75 BVs. This breakthrough was slightly earlier than the previous test (74 to 78 BVs). The Cs breakthrough after 78 BVs loading was nearly linear on the probability plot and was extrapolated to 50% C/C_o at approximately 110 BVs based on the line defined by the last six lead-column samples. This value was essentially equivalent to the Cs λ value predicted from batch-contact studies (119 BVs).

Table 3.5. AZ-101 Cs-Decontaminated Product

Cations	M	% Change	Radionuclides	μCi/mL	% Change
Na	4.87 E+0	+0.4	^{60}Co	<4 E-5	NA
K	1.11 E-1	-2.9	^{99}Tc	3.75E-1(2)	+15(2)
Cs	1.77 E-8	>-99.9	$^{99}TcO_4^-$	3.78E-1(2)	+0.3(2)
As	[1.1 E-4]	[-11.2]	^{134}Cs	<2 E-5	-99.994
Ba	< 2 E-6	[> -12]	^{137}Cs	6.49 E-2	-99.996
Ca	<2 E-4	NA	^{154}Eu	<6 E-5	NA
Cd	<4 E-6	NA	^{238}Pu	<7E-5	-80
Cu	<1 E-5	NA	$^{239+240}Pu$	<5E-5	>-97
Fe	[2.9 E-5]	-38	**Anions**	**M**	**% Change**
Li	<1.1 E-4	[>-12]	AlO_2^- (1)	2.06 E-01	-1.2
Mo	9.67 E-4	-0.7	Cl^-	<7 E-4	>-65
Ni	<2 E-5	NA	CrO_4^{-2} (1)	1.18 E-2	-2.3
Pb	<1.3 E-5	[>-17]	F^-	7.36 E-2	+27
Sn	[4.0 E-4]	[-14]	NO_2^-	1.40 E+0	-9
U (Uranyl)(1)	9.41 E-9	>-99.9	NO_3^-	8.66 E-1	+2
V	[2.7 E-5]	[-6.7]	OH^-	6.3 E-1 (2)	-7(2)
W	[3.1 E-4]	[+2.7]	PO_4^{-3} (1) ICP	1.60 E-2	-2.4
Solution Density	**g/mL**	**% Change**	PO_4^{-3} IC	1.47 E-2	-13
Density	1.224 (23°C)	0	SO_4^{-2}	1.76 E-1	+1
			Oxalate	1.71 E-2	-4

ASR 6306 RPL # = 02-0935
NA = not applicable, analyte was not detected in the feed.
The IC and ICP-AES result uncertainties are expected to be ±15% (2-σ). Bracketed results indicate that the analyte concentration uncertainties exceeded 15%. Less-than (<) results indicate that the analyte concentrations were below the instrument detection limits (IDL); the dilution-corrected IDLs are given.
Greater-than (>) values indicate the analyte recoveries were greater than shown and could not be calculated; calculation was based on less-than (<) values of the product analytes.
(1) Al, Cr, and P determined by ICP-AES. U measured by KPA. The ionic form was assumed on the basis of waste chemistry.
(2) The pertechnetate and hydroxide results are reported based on the Tc ion exchange feed analyses (ASR 6481, RPL # 02-2787) and represents the combined first and second process test AZ-101 effluents [18].

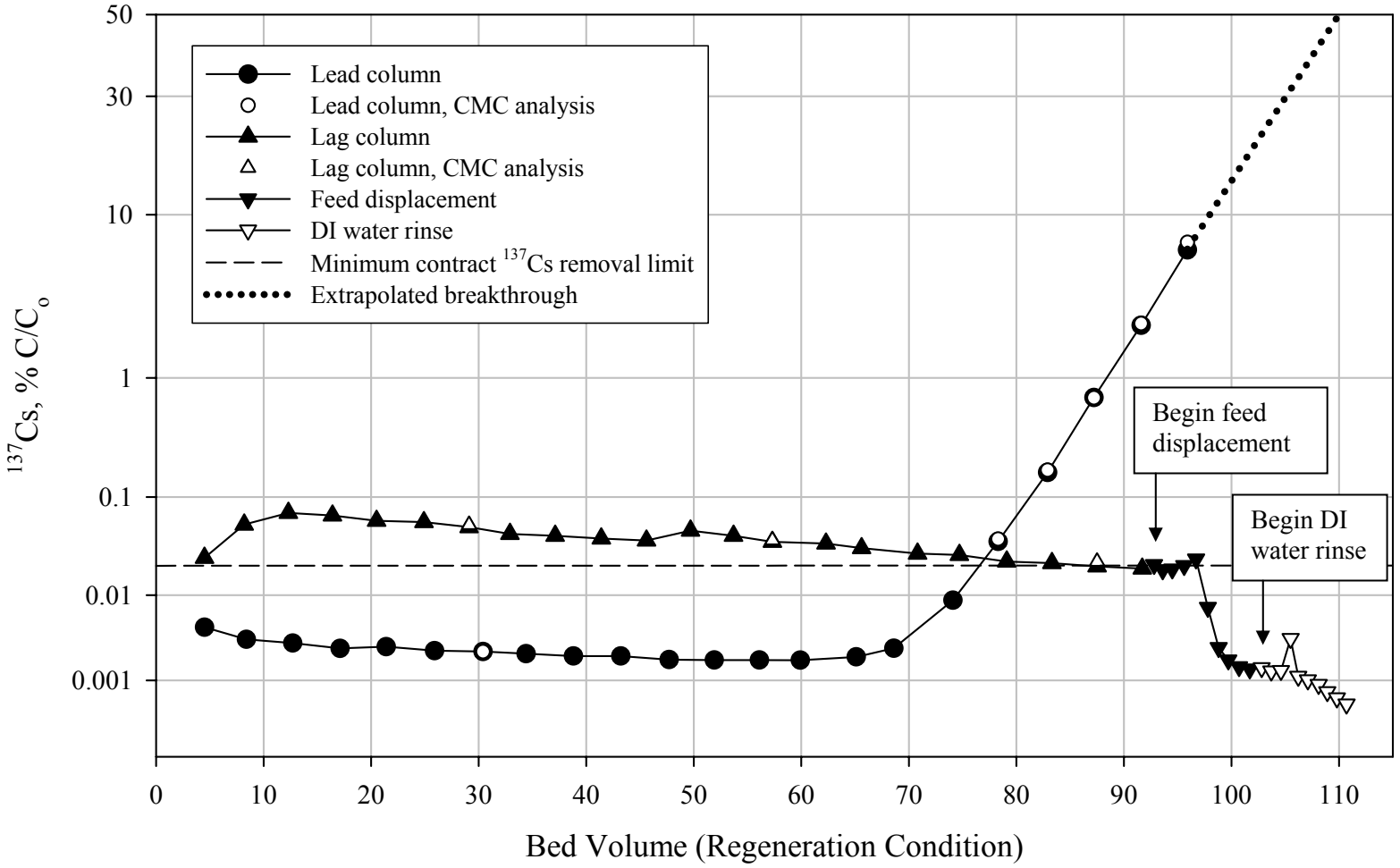
The ^{137}Cs concentrations in the lag-column effluent samples were nominally a factor of 20 higher than the lead-column samples through the first 62 BVs processed. Again, this observation was consistent with that observed during AN-102/C-104 and the first AZ-101 processing tests. In this case, however, the lag column samples **exceeded** the contract Cs-removal limit. The composite effluent, sampled after processing 78-BVs, contained 0.813 μCi ^{137}Cs/mL.[(a)] This was 2.4 times greater than the minimum Cs-removal limit for Envelope B waste. The lag column ^{137}Cs bleed-off concentration tapered slowly from 1.2 μCi/mL (C/C_o = 0.071%) to 0.34 μCi/mL (C/C_o = 0.019%). The bleed-off continued through 5 BVs (1.3 AVs) of the feed displacement, and then the ^{137}Cs concentration tailed. Introducing water did not significantly change the effluent Cs concentration. The slight rise in % C/C_o during the water rinse

(a) The baseline processing schedule identifies a normal process volume of 74 BVs through Cs ion exchange.

Table 3.6. AZ-101 Second Test ^{137}Cs Concentration, % C/C_o, as a Function of Processed Bed Volume

Column Feed	BV	^{137}Cs, % C/C_o	^{137}Cs, % C/C_o CMC Analysis	BV	^{137}Cs, % C/C_o	^{137}Cs, % C/C_o CMC Analysis
	Lead Column			*Lag Column*		
AZ-101	4.5	4.35E-3	--	4.5	2.51E-2	--
	8.4	3.14E-3	--	8.2	5.45E-2	--
	12.7	2.84E-3	--	12.3	7.06E-2	--
	17.1	2.45E-3	--	16.4	6.67E-2	--
	21.4	2.57E-3	--	20.5	5.93E-2	--
	25.9	2.30E-3	--	24.9	5.75E-2	--
	30.4	2.25E-3	2.24E-3	29.1	5.13E-2	5.41E-2
	34.4	2.12E-3	--	32.9	4.40E-2	--
	38.8	1.99E-3	--	37.1	4.21E-2	--
	43.2	1.99E-3	--	41.4	3.96E-2	--
	47.7	1.80E-3	--	45.6	3.77E-2	--
	51.9	1.78E-3	--	49.7	4.75E-2	--
	56.1	1.78E-3	--	53.7	4.22E-2	--
	59.9	1.77E-3	--	57.3	3.67E-2	3.80E-2
	65.1	1.94E-3	--	62.3	3.52E-2	--
	68.6	2.46E-3	--	65.6	3.16E-2	--
	74.1	8.81E-3	--	70.8	2.78E-2	--
	78.3	3.69E-2	3.91E-2	74.7	2.69E-2	--
	82.9	1.68E-1	1.76E-1	79.1	2.30E-2	--
	87.2	7.09E-1	7.05E-1	83.3	2.21E-2	--
	91.6	2.33E+0	2.39E+0	87.5	2.04E-2	2.29E-2
	95.9	6.59E+0	7.23E+0	91.7	1.94E-2	--
0.1 M NaOH	No data taken from lead column			92.8	2.13E-2	--
				93.6	1.88E-2	--
				94.5	1.91E-2	--
				95.6	2.08E-2	--
				96.7	2.44E-2	--
				97.8	6.21E-3	--
				98.8	2.50E-3	--
				99.7	1.78E-3	--
				100.7	1.47E-3	--
				101.7	1.38E-3	--
DI Water	No data taken from lead column			102.8	1.43E-3	--
				103.7	1.30E-3	--
				104.6	1.32E-3	--
				105.5	3.21E-3	--
				106.2	1.12E-3	--
				107.1	1.01E-3	--
				108.1	8.86E-4	--
				108.9	7.18E-4	--
				109.8	5.98E-4	--
				110.7	4.99E-4	--

"--" indicates no data were taken.



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425-µm dry particle size
BV = 10.9 mL
^{137}Cs C_o = 1645 µCi/mL
Process temperature = 25 to 27°C
Flowrate = 2.66 BV/h
Na concentration = 4.85 M

Figure 3.5. ^{137}Cs Breakthrough Curves for Second Test of AZ-101 Tank Waste, Probability Plot

that was observed at 105 BVs may have been caused by the decrease in ionic strength and/or the decrease of hydroxide concentration of the processing solution. (A similar spike in Cs concentration occurred during the first process-run feed displacement.) The subsequent drop in Cs bleed-off was most likely related to the drop in feed Na concentration, which acted as a competitor to Cs on the ion exchanger.

The ^{137}Cs bleed-off into the lag-column effluent was due primarily to insufficient elution[(a)] of the lead column during the previous AZ-101 test. The final eluate sample, taken from the preceding run, contained 3.67 μCi/mL ^{137}Cs. This concentration was well below the 1% C/C_o target for elution completion. Conversion of the aqueous phase from acid to DI water slowed the elution of Cs. However, adding caustic Na slightly increased Cs displacement. Better elution of Cs from the resin is needed. This can be approached in one or both of two ways. A larger elution volume could be processed, especially for the AZ tanks with high Cs concentrations. However, because of the tailing effect, this will have diminishing returns with increasing volumes. After elution, the resin could be expanded by regeneration then re-eluted. This scenario was utilized during previous testing with AZ-102C [10]. In this case, the re-elution peak C/C_o was higher than the final baseline elution sample.

The total quantity of ^{137}Cs processed through the ion exchange system was 1.72 Ci (19.8 mg ^{137}Cs, 63.2 mg total Cs). Integrating the ^{137}Cs breakthrough from the lead column resulted in a calculated lag-column ^{137}Cs loading of 5.2 E+3 μCi (0.059 mg ^{137}Cs, 0.19 mg total Cs), corresponding to 0.3 % of the total loaded Cs. The lead-column load was 1.81 Ci calculated from the total AZ-101 feed ^{137}Cs (1.72 Ci), subtracting the ^{137}Cs breakthrough onto the lag column (0.005 Ci), and adding the Cs remaining on the column from the previous processing with AZ-101 (0.099 Ci). The total lag-column effluent contained 0.70 μCi ^{137}Cs /mL (0.040% of the total Cs processed through the columns), corresponding to a DF of 2350.

The AZ-101 feed was loaded at nearly twice the flowrate of the first test (2.7 BV/h as opposed to 1.5 BV/h). The Cs breakthrough for the faster flowrate began nominally 5-BV sooner than that of the slower flowrate. The breakthrough offset may be attributable, in part, to the faster flowrate and the slightly lower resin bed density (0.228 g/mL vs. 0.239 g/mL, see Table 3.2), uncertainty in the BV measurement, or chemical degradation of the resin. The breakthrough curves had virtually identical slopes. This indicated that the kinetics for Cs loading was similar at the two flowrates tested, and thus particle diffusion was the controlling parameter. The C/C_o values for the second test lead column were offset from the first test by nominally a factor of 4. This was attributed to a combination of Cs buildup in the columns and associated bleed-off and the effect of the faster flowrate. Figure 3.4 shows the comparison of the lead column AZ-101 ^{137}Cs breakthrough profiles. The lag-column effluent was also higher in ^{137}Cs concentration in the second test than in the first test. Again, this was attributed to a combination of the residual Cs loading on the resin beds and the increased flowrate.

(a) Baseline elution conditions were applied, where elution proceeds through 15 BVs eluate and to a C/C_o of 1%.

3.2.2 Elution and Eluant Rinse

The lead column elution was tested at two flowrates, 1.0 BV/h and 1.8 BV/h, for the first and second processing runs, respectively. These are described in detail.

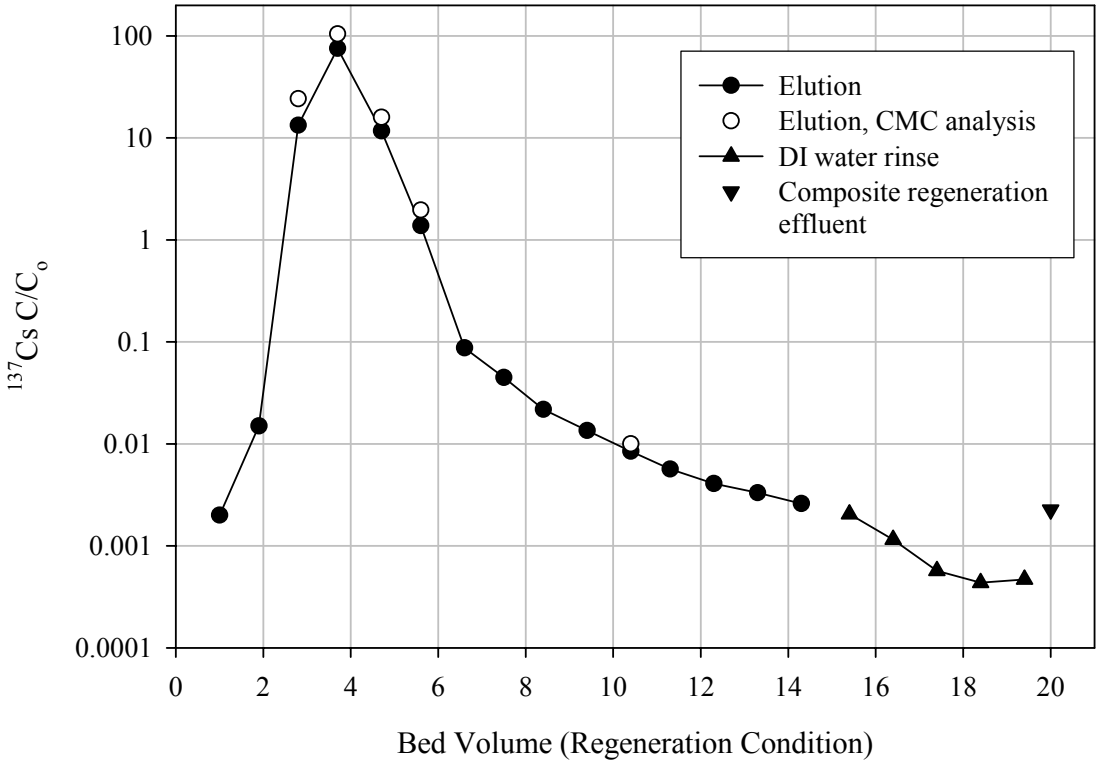
3.2.2.1 First Ion Exchange Processing

Table 3.7 and Figure 3.6 show the lead-column elution and water-rinse results and profiles. The Figure 3.6 ordinate shows the ^{137}Cs C/C_o[(a)] values on a logarithmic scale to clearly show the large range of C/C_o values obtained. The abscissa is given in BVs relative to the regeneration condition just before the AZ-101 load. Most of the ^{137}Cs was contained in elution BVs 3 through 6. The peak value of C/C_o was found to be 105 (1.72E+5 μCi/mL). Integrating the elution peak resulted in 108% ^{137}Cs recovery in the eluate. The elution cutoff of $C/C_o = 0.01$ was reached at 10 BVs. Because of the lag between sample collection and the determination of the Cs concentration, elution was continued beyond 1% C/C_o to 14.3 BVs. Analysis of the composite eluate resulted in 112% ^{137}Cs recovery and 87% ^{134}Cs recovery, averaging 99.5% Cs recovery. The final eluate sample contained 3.7 μCi/mL ^{137}Cs, $C/C_o = 0.0022$. The water rinse C/C_o significantly tailed off, indicating that the water matrix reduced Cs elution.

Table 3.7. First AZ-101 Test ^{137}Cs Elution from Lead Column

Column Feed	BV	^{137}Cs, C/C_o	^{137}Cs, μCi/mL	^{137}Cs, C/C_o CMC analysis	^{137}Cs, μCi/mL
0.5 M HNO_3	1.0	2.00E-3	3.29 E+0	--	--
	1.9	1.50E-2	2.47 E+1	--	--
	2.8	1.33E+1	2.18 E+4	2.42E+1	3.98E+4
	3.7	7.55E+1	1.24 E+5	1.05E+2	1.72E+5
	4.7	1.17E+1	1.92 E+4	1.59E+1	2.62E+4
	5.6	1.38E+0	2.27 E+3	1.96E+0	3.23E+3
	6.6	8.74E-2	1.44 E+2	--	--
	7.5	4.48E-2	7.36 E+1	--	--
	8.4	2.18E-2	3.59 E+1	--	--
	9.4	1.35E-2	2.21 E+1	--	--
	10.4	8.47E-3	1.39 E+1	8.94E-3	1.47E+1
	11.3	5.66E-3	9.31 E+0	--	--
	12.3	4.08E-3	6.72 E+0	--	--
	13.3	3.32E-3	5.46 E+0	--	--
	14.3	2.60E-3	4.28 E+0	2.23E-3	3.67E+0
DI Water Rinse	15.4	2.05E-3	3.38 E+0	--	--
	16.5	1.15E-3	1.90 E+0	--	--
	17.5	5.69E-4	9.37 E-1	--	--
	18.4	4.35E-4	7.16 E-1	--	--
	19.4	4.69E-4	7.72 E-1	--	--
Regeneration effluent composite		2.25E-3	3.71E+0	2.43E-3	3.99E+0
"--" no data taken					

(a) The C_o refers to the ^{137}Cs concentration in the AZ-101 feed sample. For elution, the C/C_o value was an indication of the extent to which ^{137}Cs is concentrated relative to the feed. It was an indirect measure of the extent to which the resin is actually eluted.



Conditions: SL-644 batch # 010319SMC-IV-73 212- to 425-µm dry-particle size
BV = 10.3 mL Eluant = 0.5 M HNO_3
Flowrate = 1.0 BV/h Process temperature = 24°C
^{137}Cs C_o = 1645 µCi/mL

Figure 3.6. ^{137}Cs Elution and Eluant Rinse of the Lead Column First Ion Exchange Processing

The eluate samples from the first ion exchange processing were composited, and a sub-sample was taken for various analyses. Table 3.8 summarizes the analytical results. As found in previous waste testing, Na was the dominant metal detected using ICP-AES with some Cd, Cr, Cu, and Fe found as well. Uranium analysis by KPA was more sensitive than ICP-AES, and the KPA results were used in recovery calculations. The U KPA analysis demonstrated quantitative recovery of U in the eluate. The Fe and U % recoveries found in the eluate were generally supported by the under-recoveries in the AZ-101 effluent. The Pu results varied between 51% ^{238}Pu and 92% $^{239+240}Pu$ recovery. This discrepancy did not make physical sense and indicated that either the feed or elute concentration of one or both of the Pu isotopes was incorrect. The 92% Pu recovery in the eluate was supported by the ^{238}Pu and $^{239+240}Pu$ losses in the effluent (>80% and >97%, respectively). About half of the feed Am was also found in the eluate. The major anion detected was nitrate, which was not surprising since the eluant was 0.5 M HNO_3. Approximately 19% of the Cl^- was recovered in the eluate, which was consistent with the low Cl^- recovery in the AZ-101 effluent. Other detected anions, SO_4^{2-} and $C_2O_4^{2-}$, may be related to precipitates from AZ-102C processing.[(a)] Large dilutions were required for the TOC measurements. The hot persulfate TOC result was within three times the MDL and should be considered semi-quantitative.

(a) The AZ-102 was concentrated by evaporation before Cs ion exchange processing. A precipitate comprised mostly of Na_3FSO_4 and $Na_2C_2O_4$ formed during evaporation. Although the feed was filtered before processing, some residual solids may have further formed and filtered on the column.

Table 3.8. Analyte Concentrations and Recoveries in the Lead-Column Eluate Composite, First Test

Analyte	Method	Cs eluate[(1)] µg/mL	Recovery % of feed[(2)]	Analyte	Method	Cs eluate[(1)] µg/mL	Recovery % of feed
Al	ICP-AES	[20]	[0.04]	U	ICP-AES	[305]	[160]
Ba	ICP-AES	[3.7][(5)]	[(5)]	U	KPA	204	107
Ca	ICP-AES	[32]	[>57]	Zn	ICP-AES	[7.1][(5)]	[(5)]
Cd	ICP-AES	[2.8]	[>83]	TOC	Hot Pers.	[5,100]	[116][(6)]
Co	ICP-AES	<5	ND	TOC	Furnace	<2,700	ND
Cr	ICP-AES	51.9	0.96	Cl^-	IC	120	19
Cs[(3)]	GEA/TIMS	589	112[(4)]	F^-	IC	<13	ND
Cu	ICP-AES	[3.0]	[>58]	NO_3^-	IC	33,500	NA
Fe	ICP-AES	[8.8]	[39]	NO_2^-	IC	<26	ND
K	ICP-AES	[220]	[0.57]	PO_4^{-3}	IC	68	0.49
La	ICP-AES	<5	ND	SO_4^{-2}	IC	230	0.16
Mg	ICP-AES	<10	ND	Oxalate	IC	150	1.74
Mn	ICP-AES	<5	ND	**Radioisotope**	**Method**	**µCi/mL**	**Recovery % of feed**
Mo	ICP-AES	<5	ND	^{134}Cs	GEA	5.78	87
Na	ICP-AES	1,040	0.11	^{137}Cs	GEA	1.60E+4	112
Ni	ICP-AES	<3	ND	^{238}Pu	AEA	1.50E-3	51
Pb	ICP-AES	<10	ND	$^{239+240}Pu$	AEA	1.43E-2	92
Si	ICP-AES	<52	ND	^{241}Am	AEA	4.37E-4	48
Sn	ICP-AES	<52	ND	$^{243+244}Cm$	AEA	2.57E-5	7.6
Sr	ICP-AES	<2	ND	Alpha sum	AEA	1.63E-2	88
Ti	ICP-AES	<3	ND	**Density, g/mL**		1.014	NA

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(1) The overall error was estimated to be within ± 15%. Values in brackets were within 10-times the detection limit, and errors were likely to exceed ± 15%. Less-than (<) values indicate that the analytes were not detected by the instrument, and the reported values represent the IDLs multiplied by the sample dilution factors.

(2) Greater-than (>) values indicate the analyte recoveries were greater than shown and could not be calculated; calculation was based on less-than (<) values of the AZ-101 feed analytes.

(3) The total Cs concentration was calculated based on the ^{137}Cs concentration and the isotopic distribution ratio.

(4) When combined with the pre-existing Cs on the column from AZ-102C processing, the Cs recovery was calculated to be 107%.

(5) The diluent blank concentration equaled the sample concentration.

(6) The TOC result was within 3 times the MDL, and uncertainty is large.

NA = not applicable; ND = not detected in the eluate

Table 3.9 presents a condensed summary of the analyte masses and millimoles recovered in the eluate. The millimoles recovered in the eluate may be related to the active sites available on the resin. Milli-equivalents were not estimated because the ionic form exchanging onto the resin was not always known. The major cation was Na. The total millimoles recovered were consistent with previous testing.

Table 3.9. Select Analyte Recoveries in Eluate

Analyte	AZ-101 feed, total mg[(1)]	Amount Recovered in Eluate[(1)]		
		mg	mmoles	%[(2)]
U	28.1	30.0	0.126	107
Ca	<8	[4.6]	[0.10]	[>57]
Cd	<0.5	[0.40]	[0.0032]	[>83]
Cr	793	7.6	0.15	0.96
Cs	77.2[(3)]	86.4	0.642	112
Cu	<0.8	[0.44]	[0.0061]	[>55]
Fe	[3.28]	[1.29]	[0.023]	[39]
Na	1.41E+5	153	6.65	0.11
Ni	<1	<1	NA	NA
Pb	[4.0]	<10	NA	NA
Sum	--	--	7.06	--

(1) Values in brackets were based on results reported within 10-times the detection limit, and errors were likely to exceed ±15%. Less-than (<) values indicate the analytes were not detected by the instrument, and the reported values represents the IDLs multiplied by the sample dilution factors.
(2) Greater-than (>) values indicate the analyte recoveries were greater than shown and could not be calculated; calculation was based on less-than (<) values of the AZ-101 feed analytes.
(3) A total estimate of 72.8 mg Cs was loaded on the lead column in addition to 4.4 mg from AZ-102C processing.
"--" indicates no data or not applicable.

3.2.2.2 Second Ion Exchange Processing

A total of 1.81 Ci ^{137}Cs was calculated to be loaded on the lead column. Table 3.10 summarizes the eluate Cs concentration results as a function of BV processed. Figure 3.7 shows the lead-column elution profile (in comparison to the 1.0 BV/h elution profile where 2.10 Ci ^{137}Cs were loaded). The peak value of C/C_o was found to be 88 (1.45E+5 μCi/mL). Integrating the elution peak resulted in 108% ^{137}Cs recovery in the eluate. The elution cutoff of $C/C_o = 0.01$ was reached at 11 BVs. Because of the lag between sample collection and the determination of the Cs concentration, elution was continued beyond 1% C/C_o to 15.9 BVs. The eluate samples from the second ion exchange processing were composited and only analyzed by GEA, resulting in 1.20E+4 μCi ^{137}Cs/mL and 4.09E+0 μCi ^{134}Cs/mL. Analysis of the composite eluate resulted in 115% recovery.

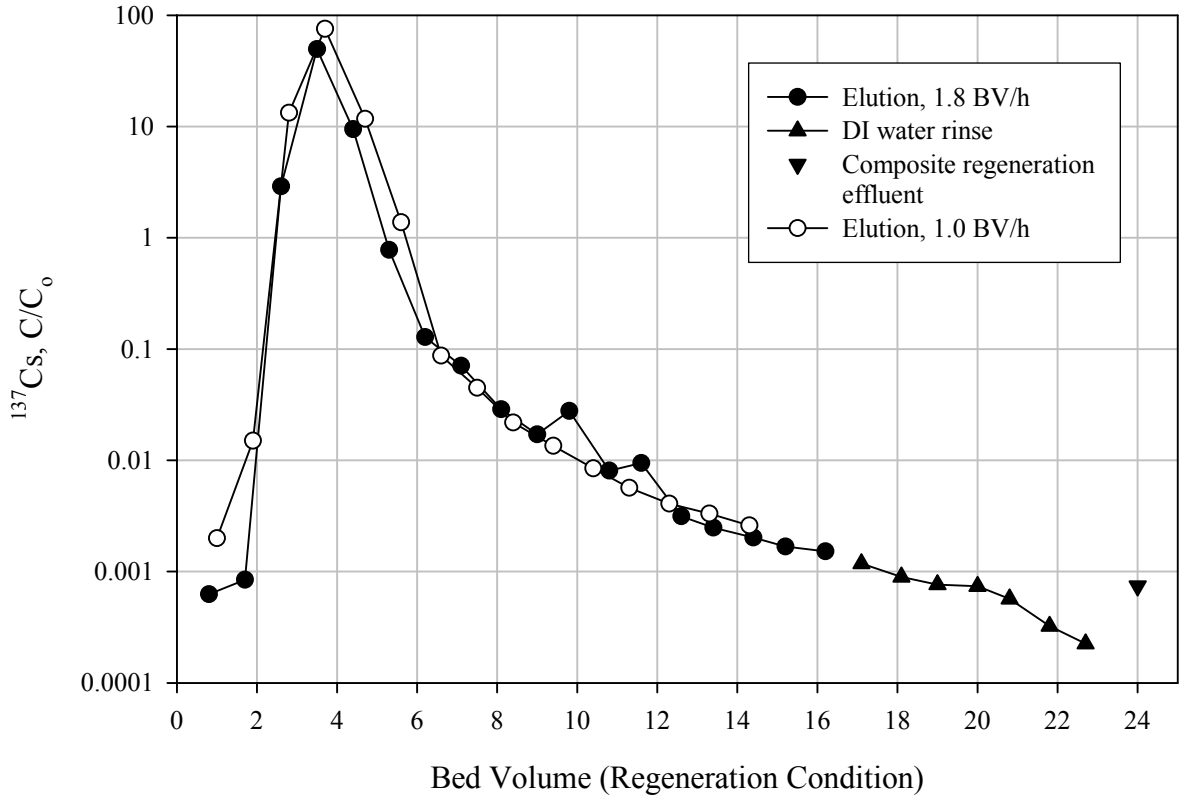
Table 3.10. Second AZ-101 Test ^{137}Cs Elution from Lead Column

Column Feed	BV	^{137}Cs, C/C_o	^{137}Cs, µCi/mL	^{137}Cs, C/C_o CMC analysis	^{137}Cs, µCi/mL
0.5 M HNO_3	0.8	6.25 E-4	1.03 E+0	--	--
	1.7	8.43 E-4	1.39 E+0	--	--
	2.5	2.89 E+0	4.75 E+3	7.17 E+0	1.18 E+4
	3.4	4.97 E+1	8.17 E+4	8.75 E+1	1.44 E+5
	4.3	9.46 E+0	1.56 E+4	2.54 E+1	4.17 E+4
	5.2	7.78 E-1	1.28 E+3	1.73 E+0	2.84 E+3
	6.1	1.28 E-1	2.10 E+2	3.16 E-1	5.20 E+2
	7.0	7.09 E-2	1.17 E+2	--	--
	7.9	2.88 E-2	4.74 E+1	--	--
	8.9	1.71 E-2	2.81 E+1	1.61 E-2	2.65 E+1
	9.6	2.78 E-2	4.57 E+1	--	--
	10.6	8.06 E-3	1.33 E+1	--	--
	11.4	9.44 E-3	1.55 E+1	--	--
	12.4	3.14 E-3	5.16 E+0	3.41 E-3	5.61 E+0
	13.2	2.48 E-3	4.08 E+0	--	--
	14.1	2.02 E-3	3.33 E+0	--	--
	15.0	1.68 E-3	2.76 E+0	--	--
	15.9	1.52 E-3	2.50 E+0	--	--
DI Water Rinse	16.8	1.18 E-3	1.94 E+0	--	--
	17.7	8.97 E-4	1.48 E+0	--	--
	18.7	7.64 E-4	1.26 E+0	--	--
	19.6	7.37 E-4	1.21 E+0	--	--
	20.4	5.69 E-4	9.36 E-1	--	--
	21.4	3.24 E-4	5.34 E-1	--	--
	22.3	2.25 E-4	3.69 E-1	--	--
Regeneration effluent composite		7.40 E-4	1.22 E+0	--	--

"--" no data taken

The elution profile, generated at 1.8 BV/h, was virtually identical to the elution profile generated at 1.0 BV/h. The peak maximum, width, and tailing through 14 BVs were virtually identical. Both elutions recovered >99.9% of the Cs loaded on the lead column. Thus, nearly doubling the flowrate from 1.0 to 1.8 BV/h was well accommodated. Larger elution volumes can thus be processed with no penalty in processing time if higher elution volumetric flowrates can be provided.

The eluates from both ion exchange processing runs were composited and analyzed. Table 3.11 and Table 3.12 summarize the analytical results. The specified minimum reportable quantities (MRQ) and the % mass recovered relative to the mass in the feed are also provided. In cases where a result was below the IDL, then the dilution-corrected IDL was provided as a "less-than" value.



Conditions: SL-644 batch # 010319SMC-IV-73 212- to 425-µm dry particle size
Process temperature = 26°C BV = 10.9 mL
Flowrate = 1.8 BV/h ^{137}Cs C_o = 1645 µCi/mL
Eluant = 0.5 M HNO_3

Figure 3.7. ^{137}Cs Elution and Eluant Rinse of the Lead Column 1.8 BV/h in Comparison to First Processing at 1.0 BV/h

Table 3.11. Inorganic and Organic Analytes in the Combined Lead Column Eluate Composites

Analyte	Method	MRQ[1] µg/mL	Cs eluate µg/mL	% recovered	Analyte	Method	MRQ[1] µg/mL	Cs eluate µg/mL	% recovered
Ag	ICP-AES	NMRQ	<2.6	ND	Sr	ICP-AES	NMRQ	<1.5	ND
Al	ICP-AES	75	<6.2	<0.02	Ta	ICP-MS	NMRQ	<5E-2[7]	ND
As	ICP-AES	NMRQ	<26	<40	Te	ICP-AES	NMRQ	<160	ND
B	ICP-AES	NMRQ	[9.5]	[1.7]	Th	ICP-AES	NMRQ	<110	ND
Ba	ICP-AES	2.3	[2.5]	[115]	Ti	ICP-AES	17	< 2.6	ND
Be	ICP-AES	NMRQ	<1.0	ND	U	ICP-AES	600	<210	ND
Bi	ICP-AES	NMRQ	<10	ND	U	KPA	600	159	99
Ca	ICP-AES	150	[27]	>58	U	ICP-MS	NMRQ	168	105
Cd	ICP-AES	8	[2.0]	>71	V	ICP-AES	NMRQ	<5.2	ND
Ce	ICP-AES	NMRQ	<21	ND	Y	ICP-AES	NMRQ	<5.2	ND
Co	ICP-AES	30	<5.2	ND	Zn	ICP-AES	17	[6.9]	HB[3]
Cr	ICP-AES	15	32.7	0.72	Zr	ICP-AES	NMRQ	<5.2	ND
Cs[2]	GEA/TIMS	1.5	478	109	Br^-	IC	NMRQ	<130	ND
Cu	ICP-AES	17	<2.6	ND	Cl^-	IC	3	180	35
Dy	ICP-AES	NMRQ	<5.2	ND	F^-	IC	150	<13	< 0.2
Eu	ICP-AES	NMRQ	<10	ND	NO_2^-	IC	3000	<26	ND
Fe	ICP-AES	150	[6.8]	[36]	NO_3^-	IC	3000	31,200	NA
Hg	CVAA	NMRQ	<0.003	ND	PO_4^{-3}	IC	2500	<26	ND
K	ICP-AES	75	<210	< 0.6	SO_4^{-2}	IC	2300	300	0.25
La	ICP-AES	35	<5.2	ND	Oxalate	IC	NMRQ	170	2.3
Li	ICP-AES	NMRQ	<3.1	ND	TOC	Hot Pers.	1500	20,500	556
Mg	ICP-AES	300	<10	ND	TOC	Furnace	1500	[3,300][4]	[30]
Mn	ICP-AES	150	<5.2	ND	EDTA	GC/FID	1500	<5	ND
Mo	ICP-AES	150	<5.2	ND	HEDTA	GC/FID	1500	<9	ND
Na	ICP-AES	75	803	0.10	ED3A	GC/FID	1500	<5	ND
Nd	ICP-AES	NMRQ	<10	ND	NTA	GC/FID	1500	<6	ND
Ni	ICP-AES	30	<3.1	ND	NIDA/IDA	GC/FID	1500	<11	ND
P	ICP-AES	600	<10	ND	Citric Acid	GC/FID	1500	<6	ND
Pb	ICP-AES	300	<10	ND	Glycolate	Organic IC	1500	<140	ND
Pd	ICP-MS	NMRQ	<8E-2	ND	Acetate	Organic IC	1500	<110	ND
Pr	ICP-MS	NMRQ	<5E-2	ND	Formate	Organic IC	1500	<190	ND
Pt	ICP-MS	NMRQ	<2E-1	ND	Oxalate	Organic IC	1500	<250	ND
Rh	ICP-MS	NMRQ	<8E-2	NM	Citrate	Organic IC	1500	<480	ND
Ru[6]	ICP-MS	NMRQ	2.0E-2	NM	OH^-	Titration	17	NM	NA
Sb	ICP-AES	NMRQ	<52	ND	NH_3	ISE	NMRQ	[1.8]	[11][5]

Table 3.11 (Contd)

Analyte	Method	MRQ(1) µg/mL	Cs eluate µg/mL	% recovered	Analyte	Method	MRQ(1) µg/mL	Cs eluate µg/mL	% recovered
Se	ICP-AES	NMRQ	<26	ND	Wt% dried solids		0.1	0.48%	NA
Si	ICP-AES	170	<52	ND	Wt% oxides		NMRQ	0.19%	NA
Sn	ICP-AES	1500	<160	ND	Density, g/mL		NMRQ	1.012	NA

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Notes:

The overall error was estimated to be within ±15%. Values in brackets were within 10-times the detection limit, and errors were likely to exceed ±15%. Less-than (<) values indicate that the analytes were not detected by the instrument, and the reported values represent the IDLs multiplied by the sample dilution factors.

NA = not applicable; ND = analyte not detected in the eluate; NM = analyte not measured in the feed; HB = high preparative blank concentration—recovery cannot be calculated.

(1) MRQ is minimum reportable quantity. NMRQ indicates no minimum reportable quantity was requested.
(2) Calculated based on ^{137}Cs concentration in the eluate and applied eluate Cs isotopic distribution ratio (^{133}Cs 51.85 wt%, ^{135}Cs 16.9 wt%, ^{137}Cs 31.25 wt%).
(3) The preparation blank resulted in [6.5] µg/mL Zn, indicating that the reported result is probably spurious.
(4) Within the analytical uncertainty, the sample result was equivalent to the TOC diluent blank of 3100 µg/mL and the MDL of 2700 µg/mL.
(5) The NH_3 feed concentration was estimated at 2.3 µg/mL and was less than 10-times the detection limit. [13]
(6) Total Ru based on ^{235}U fission yield.
(7) The Ta BS and MS recovered low (33% and 40%, respectively).

The chloride concentration was slightly higher in the combined eluate than that found in the first AZ-101 test eluate. The eluate Cl^- concentration may be increasing with operational cycles or may be a function of the AZ-101 waste type. The eluates obtained after AW-101 simulant, AP-101DF, and AN-102/C-104 processing each contained <60 µg/mL Cl^-. The AZ-102C test resulted in 75 µg/mL Cl^-. The AZ-101 first test resulted in 120 µg/mL Cl^-, and the combined eluate was higher yet at 180 µg/mL Cl^-, indicating that the second AZ-101 eluate Cl^- concentration exceeded 180 µg/mL Cl^-. Chloride in the eluate may affect downstream processing. Chloride can volatilize in the evaporator [19], leading to potential corrosion problems in the off-gas treatment equipment. Appropriate system equipment material selection can accommodate the chloride and mitigate or eliminate corrosion problems. High chloride concentration can also lead to salt formation in the melter [20].

The Fe, Cr, U, Ca, Cd, oxalate, and sulfate concentration results from the combined composite were similar to those found for the first AZ-101 processing eluate. The Pu and Am recoveries were much lower (factor of 2) when calculated from the combined composite eluates than from the first-run eluate composite (Table 3.7). This indicated that the second AZ-101 test lead column did not exchange Pu and Am, and the reported recoveries reflect the dilution factor associated with combining the second eluate with the first eluate. This scenario, however, seems unlikely and would point to an analytical problem. The feed ^{90}Sr concentration was close to the method detection limit and had high uncertainty. Thus, the 38% ^{90}Sr recovery in the eluate also had a high uncertainty.

Table 3.12. Radionuclides in the Combined Lead Column Eluate Composites

Analyte	Method	MRQ[(1)] µCi/mL	Cs eluate µCi/mL	Error % (1-σ)	% recovered	Analyte	Method	MRQ[(1)] µCi/mL	Cs eluate µCi/mL	Error % (1–σ)	% recovered
^{3}H	Radchem	NMRQ	<3E-3	–	ND	^{155}Eu	GEA	9.00 E-2	<4 E+0	–	ND
^{14}C	Radchem	NMRQ	<3E-3	–	ND	^{232}Th	GEA	NMRQ	<3 E+0	–	ND
^{51}Cr	GEA	NMRQ	<2 E+1	–	ND	^{233}U	ICP-MS	NMRQ	7.91 E-5	8	NM[(3)]
^{59}Fe	GEA	NMRQ	<2 E-1	–	ND	^{234}U	ICP-MS	NMRQ	7.25 E-5	5	NM[(3)]
^{60}Co	GEA	NMRQ	<7 E-2	–	ND	^{235}U	ICP-MS	NMRQ	2.68 E-6	0.2	NM[(3)]
^{63}Ni	Radchem	NMRQ	4.1 E-3[(2)]	20	NM	^{236}U	ICP-MS	NMRQ	6.20 E-6	1	NM[(3)]
^{79}Se	Radchem	NMRQ	<2 E-4	–	ND	^{238}U	ICP-MS	NMRQ	5.61 E-5	0.3	NM[(3)]
^{88}Y	GEA	NMRQ	<2 E-1	–	ND	^{236}Pu	Radchem	NMRQ	<9 E-6	–	ND
^{90}Sr	Radchem	1.50 E-1	3.30 E+0	6	38	^{237}Np	ICP-MS	NMRQ	<4 E-5	0.5	ND
^{95}Nb	GEA	NMRQ	<2 E-1	–	ND	^{238}Pu	Radchem	NMRQ	8.26 E-4	5	33
^{99}Tc	ICP-MS	3.00 E-3	<3 E-4	–	<0.006	$^{239+240}Pu$	Radchem	NMRQ	7.94 E-3	3	61
^{103}Ru	GEA	NMRQ	<2 E+0	–	ND	^{239}Pu	ICP-MS	NMRQ	6.42 E-3	0.7	44
^{106}Ru	GEA	NMRQ	<9 E+0	–	ND	^{240}Pu	ICP-MS	NMRQ	1.77 E-3	5	>12
^{113}Sn	GEA	NMRQ	<3 E+0	–	ND	^{241}Pu	Radchem	NMRQ	3.90 E-2	7	NM
^{125}Sb	GEA	NMRQ	<6 E+0	–	ND	^{241}Am	Radchem	7.20E-04	2.28 E-4	5	30
$^{126}Sn/Sb$	GEA	NMRQ	<3 E+0	–	ND	^{241}Pu, ^{241}Am	ICP-MS	NMRQ	5.8 E-4 µg/mL[(4)]	7	NM
^{129}I	ICP-MS	NMRQ	<3.1E-4	–	ND	^{242m}Am	Radchem	NMRQ	<4 E-6	–	ND
^{134}Cs	GEA	NMRQ	4.39 E+0	3	78	^{242}Cm	Radchem	NMRQ	<3 E-6	–	ND
^{137}Cs	GEA	5.00 E-2	1.30 E+4	3	109	^{242}Pu	ICP-MS	NMRQ	<4 E-4	–	ND
^{144}Ce	GEA	NMRQ	<8 E+0	–	ND	$^{243+244}Cm$	Radchem	NMRQ	2.00 E-5	15	6.6
^{151}Sm	Radchem	NMRQ	6.12 E-3	7	NM	Alpha sum[(5)]	Radchem	NMRQ	9.01 E-3	3	55
^{152}Eu	GEA	NMRQ	<3 E-1	–	ND	Total alpha	Radchem	2.30E-01	7.24 E-3	22	NM
^{154}Eu	GEA	2.00 E-3	<2 E-1	–	ND	Total beta	Radchem	NMRQ	1.40 E+4	4	NM

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NM = not measured in the feed; ND = not detected in feed or eluate; "—" not applicable

Less-than (<) values indicate the analytes were not detected by the instrument, and the reported values represent the IDLs multiplied by the sample dilution factors.

(1) MRQ is minimum reportable quantity. NMRQ indicates no minimum reportable quantity requested.

(2) The ^{63}Ni duplicate sample was less than the MDA of 3E-3 µCi/mL. The beta spectrum did not clearly show a ^{63}Ni peak.

(3) The total eluate U recovery was measured by ICP-MS at 105%.

(4) The AMU-241 is reported in units of µg/mL. Applying the specific activity of ^{241}Pu, the sample activity is 5.97E-2 µCi/mL; applying the specific activity of ^{241}Am, the sample activity is 1.99E-3 µCi/mL.

(5) The alpha sum (total of individually-measured Pu, Am, and Cm alpha emitters) provides the best estimate of the total alpha activity in the sample.

The TOC results for the two methods, hot persulfate and furnace oxidation, were very different. The furnace method TOC result was equivalent to the diluent blank result of 3,100 μg C/mL, within analytical uncertainty. The hot-persulfate TOC analysis result (20,500 μg/mL) was a factor of 6 times higher than that of the furnace method. The oxalate contributed a small portion to the TOC. The remaining TOC was potentially from resin degradation product(s) leaching into the eluate.

Most of the specified MRQ levels were met with some exceptions. The large dilutions required to reduce radiation dose and safely handle the sample at the ICP-AES workstation prevented the K MRQ level from being met. The large amount of ^{137}Cs prevented the detection limits for ^{154}Eu and ^{155}Eu from meeting the MRQ levels. Relatively high ^{137}Cs activity increased the gamma background level in the detectors due to Compton scattering, thereby making it difficult to detect lower concentrations of other lower energy gamma emitters. The TIC analysis was not completed because carbonate is known to evolve as CO_2 in acidic solutions. Cyanide analysis was also not completed because CN was not expected to be present in acid solution as it should evolve as HCN gas.

3.2.3 Regeneration

The composite regeneration solution C/C_o values are also shown on the elution profiles (Figure 3.6 and Figure 3.7). In both cases, the ^{137}Cs concentrations in the composite regeneration solutions were higher than those of the last DI water-rinse solutions and nearly equivalent to the last elution sample. The Cs K_d values increase with increasing hydroxide concentration [15]. As 0.25 M NaOH was added to the H-form resin bed, the residual acid in the system neutralized the hydroxide. The Na then more effectively competed for Cs on the resin. By this mechanism, the regeneration solution was expected to result in higher C/C_o values than obtained with the last DI water sample.

Table 3.13 shows the composition of the regeneration solution for the first ion exchange processing of AZ-101. Sodium and a small amount of Cr and Al were measured by ICP-AES. The regeneration solution was slightly acidic (pH = 6.2); therefore, OH^- was not present in significant amounts in the regeneration solution effluent. The amount of 0.25 M NaOH processed was just sufficient to neutralize the apparatus and resin bed. Because the resin bed reached maximum volume, and the apparatus was rinsed with DI water and then again with 0.25 M NaOH, the resin bed was considered fully converted to the Na form before processing the next AZ-101 sample.

The total mmoles of Na exchanged onto the resin was calculated to be 5.5 mmoles per 2.48 g dry Na-form resin according to Equation 3.2. This result was lower than the calculated 6.6 mmoles Na obtained during elution of the ion exchange column (see Table 3.9). This indicated the resin was ~83% (5.5/6.6) converted to the Na form.

$$\left(V - V_H\right) * C - \left(C_E * V_E\right) \tag{3.2}$$

where: V = volume of 0.25 M NaOH processed, 46 mL
V_H = holdup volume (volume of 0.25 M NaOH remaining in system), 23 mL
C = NaOH concentration, 0.25 M
C_E = Na concentration in regeneration effluent, [5.2E-3]M
V_E = regeneration effluent volume, 46 mL.

Table 3.13. Composition of Regeneration Solution First Ion Exchange Processing

Analyte	Concentration, µg/mL	Concentration, M
Na^+	120	5.2E-3
K^+	< 20	< 5E-4
Al	[0.70]	[2.6E-5]
Cr	[0.46]	[8.8E-6]
OH^-	NM	NM
^{137}Cs	3.99 µCi/mL	3.35E-7
Total Cs	0.15	1.1E-6
NM = not measured; the solution was slightly acidic, pH = 6.2. The overall error was estimated to be within ±15%. Values in brackets were within 10-times the detection limit, and errors likely exceeded ±15%. Less-than (<) values indicate that the analytes were not detected by the instrument, and the reported values represent the IDLs multiplied by the sample dilution factors.		

The current design basis utilizes spent regeneration solution as feed displacement for follow-on processing. For this to be effective, the spent regeneration solution must be caustic. The slight acidic nature of the composite regeneration solution precludes this follow-on use. The acidity may be a result of mixing of rinse solutions in the system apparatus, reducing the efficiency of the rinse process. In this column system, the largest mixing areas were both above and below the resin beds. Regeneration effluent acidity may be reduced by minimizing all mixing areas in the column apparatus, utilizing a larger DI water wash volume after elution, and/or using a larger regeneration solution volume.

3.2.4 Activity Balance for ^{137}Cs

An activity balance for ^{137}Cs was completed for both tests to compare the ^{137}Cs recovered in various process streams to the ^{137}Cs present in the feed sample (Table 3.14 and Table 3.15). As expected, virtually all ^{137}Cs loaded on the lead column was found in the eluate stream, recovering >99% of the ^{137}Cs present in the initial AZ-101 feed. The eluates resulted in greater than 100% Cs recovery. This was attributed to analytical error and was most likely closer to 100%.

Table 3.14. Activity Balance for ^{137}Cs, First AZ-101 Ion Exchange Processing

Solution	^{137}Cs, µCi	^{137}Cs Relative to total 2.20 Ci, %[(1)]
Feed Sample (AZ-101)	2.08 E+6	9.45 E+1
Initial AZ-102C lead column loading	1.2 E+5	5.5 E+0
Combined feed + AZ-102C	2.20 E+6	1.00 E+2
Effluent	7.1 E+1	3.2 E-3
Load samples (lead and lag columns)	4.8 E+3	2.2 E-1
Feed displacement	2.5 E+0	1.1 E-4
DI water rinse	5.5 E-1	2.5 E-5
Lead column eluate	2.35 E+6	1.07 E+2
Lead column DI water rinse	8.5 E+1	3.9 E-3
Lead column regeneration	1.7 E+2	7.7 E-3
Lag column Cs loading	9.9 E+4	4.5 E+0[(2)]
Total ^{137}Cs Recovery	2.45 E+6	1.12 E+2
(1) 2.08 Ci in feed plus 0.12 Ci remaining on column from previous AZ-102C test. (2) Based on integration of the lead-column sample breakthrough.		

Table 3.15. Activity Balance for ^{137}Cs, Second AZ-101 Ion Exchange Processing

Solution	^{137}Cs, µCi	^{137}Cs Relative to total 1.82 Ci, %[1]
Feed Sample (AZ-101)	1.72 E+6	9.45 E+1
Initial AZ-101 lead column loading	9.9 E+4	5.4 E+0
Combined feed + AZ-101	1.82 E+6	1.00 E+2
Effluent	6.6 E+2	3.6 E-2
Load samples (lead and lag columns)	3.6 E+2	2.0 E-2
Feed displacement	2.1 E+1	1.2 E-3
DI water rinse	2.0 E+0	1.1 E-4
Lead column Eluate	2.07 E+6	1.14 E+2
Lead column DI water rinse	7.7 E+1	4.2 E-3
Lead column regeneration	5.8 E+1	3.2 E-3
Lag column Cs loading	5.2 E+3	2.9 E-1[2]
Total ^{137}Cs Recovery	2.08 E+6	1.14 E+2
(1) 1.72 Ci in feed plus 0.099 Ci remaining on column from previous AZ-101 test. (2) Based on integration of the lead-column sample breakthrough.		

3.2.5 SL-644 Resin Volume Changes

The SL-644 resin is known to change in volume as a function of the solution pH and ionic strength [21]. Table 3.16 shows the resin BV change history. The ion exchange columns are labeled 1 and 2. Column 1 was the lead column for the AW-101 simulant test and the AP-101DF test; Column 2 was the lag column for these tests. Results from both tests have been previously reported [7, 8]. These columns were switched for the AN-102/C-104 ion exchange test. Thus Column 1 was placed in the lag position, and Column 2 was placed in the lead position [9]. The column positions were switched again for the AZ-102C ion exchange test, where Column 1 was placed back into the lead position and Column 2 back to the lag position [10]. Switching continued for the AZ-101 ion exchange processing.

Table 3.16. SL-644 Bed Volumes

Feed	Symbol	Process Step	010319SMC-IV-73 212-425 µm particle size	
			Column 1[1]	Column 2[2]
Initial Column Positions			**Lead Column, mL**	**Lag Column, mL**
Initial packing	P	1	10.9	10.9
0.5 M HNO_3	E	2	9.2	8.9
DI water	W	3	9.2	8.9
0.25 M NaOH	R	4	11.2	10.8
AW-101 simulant	F	5	10.2	10.0
0.1 M NaOH	FD	6	10.9	10.7
DI water	W	7	10.9	10.9
0.5 M HNO_3	E	8	8.9	8.9
DI water	W	9	8.9	8.7
Re-fluidize bed	RP	10	7.5	7.5
Place Columns in Hot Cell				
0.25 M NaOH	R	11	11.0	10.9
DI water	W	12	10.5	11.0
0.5 M HNO_3	E	13	7.9	7.7
DI water	W	14	7.9	7.7
0.25 M NaOH	R	15	10.7	10.5

Table 3.16 (Contd)

Feed	Symbol	Process Step	010319SMC-IV-73 212-425 μm particle size	
			Column 1[(1)]	Column 2[(2)]
AP-101DF	F	16	9.7	9.7
0.1 M NaOH	FD	17	10.5	10.4
DI water	W	18	10.4	10.5
0.5 M HNO_3	E	19	7.7	—
DI water	W	20	7.7	—
0.25 M NaOH	R	21	10.5	—
DI water	W	22	10.2	—
Switch Column Positions			**Lag Column, mL**	**Lead Column, mL**
0.25 M NaOH	R	23	10.5	10.4
AN-102/C-104	F	24	9.9	9.9
0.1 M NaOH	FD	25	10.7	10.4
DI water	W	26	10.7	10.4
0.5 M HNO_3	E	27	—	7.7
DI water	W	28	—	7.5
0.25 M NaOH	R	29	—	10.2
DI water	W	30	—	10.4
Switch Column Positions			**Lead Column, mL**	**Lag Column, mL**
0.25 M NaOH	R	31	10.2	10.0
AZ-102C	F	32	9.5 (jumped to 10.4)	9.4
0.1 M NaOH	FD	33	10.7	10.0
DI water	W	34	10.9	10.3
0.5 M HNO_3	E	35	8.1	—
DI water	W	36	8.2	—
0.25 M NaOH	R	37	10.4	—
DI water	W	38	10.7	—
0.5 M HNO_3	E	39	7.9	—
DI water	W	40	7.8	—
0.25 M NaOH	R	41	10.5	—
DI water	W	42	10.7	—
Switch Column Positions			**Lag Column, mL**	**Lead Column, mL**
0.25 M NaOH	R	43	10.9	10.3
AZ-101, first test	F	44	10.2	9.5
0.1 M NaOH	FD	45	10.9	10.5
DI water	W	46	10.9	10.7
0.5 M HNO_3	E	47	—	7.7
DI water	W	48	—	7.9
0.25 M NaOH	R	49	—	10.4
DI water	W	50	—	10.9
Switch Column Positions			**Lead Column, mL**	**Lag Column, mL**
0.25 M NaOH	R	51	10.9	10.5
AZ-101, second test	F	52	10.2	9.7
0.1 M NaOH	FD	53	10.9	10.5
DI water	W	54	10.9	10.5
0.5 M HNO_3	E	55	8.4	—
DI water	W	56	8.5	—
0.25 M NaOH	R	57	11.0	—
DI water	W	58	11.0	—

(1) Calculated resin mass after washing, dry Na-form: 2.46 g.
(2) Calculated resin mass after washing, dry Na-form: 2.48 g.
"—" not applicable

Figure 3.8 shows the variation in BV as a function of the process step for both columns and all process testing. Each process step is denoted with a number corresponding to the number in Table 3.11. The volume contraction after the initial 0.5 M HNO_3 conditioning step and AW-101 processing became more pronounced with cycling as evidenced by comparing the AW-101 simulant run with the AP-101DF run. After this initial cycling, the resin beds appeared to remain fairly consistent with respect to swelling and shrinking. An anomalous behavior was observed during the load phase of AZ-102C (Step 32, Column 1). The lead column resin bed expanded after loading 508 mL AZ-102C in a 3-h time frame from 9.5 mL to 10.4 mL (+9%). The corresponding BV increase of Column 1 was reflected in subsequent processing steps.

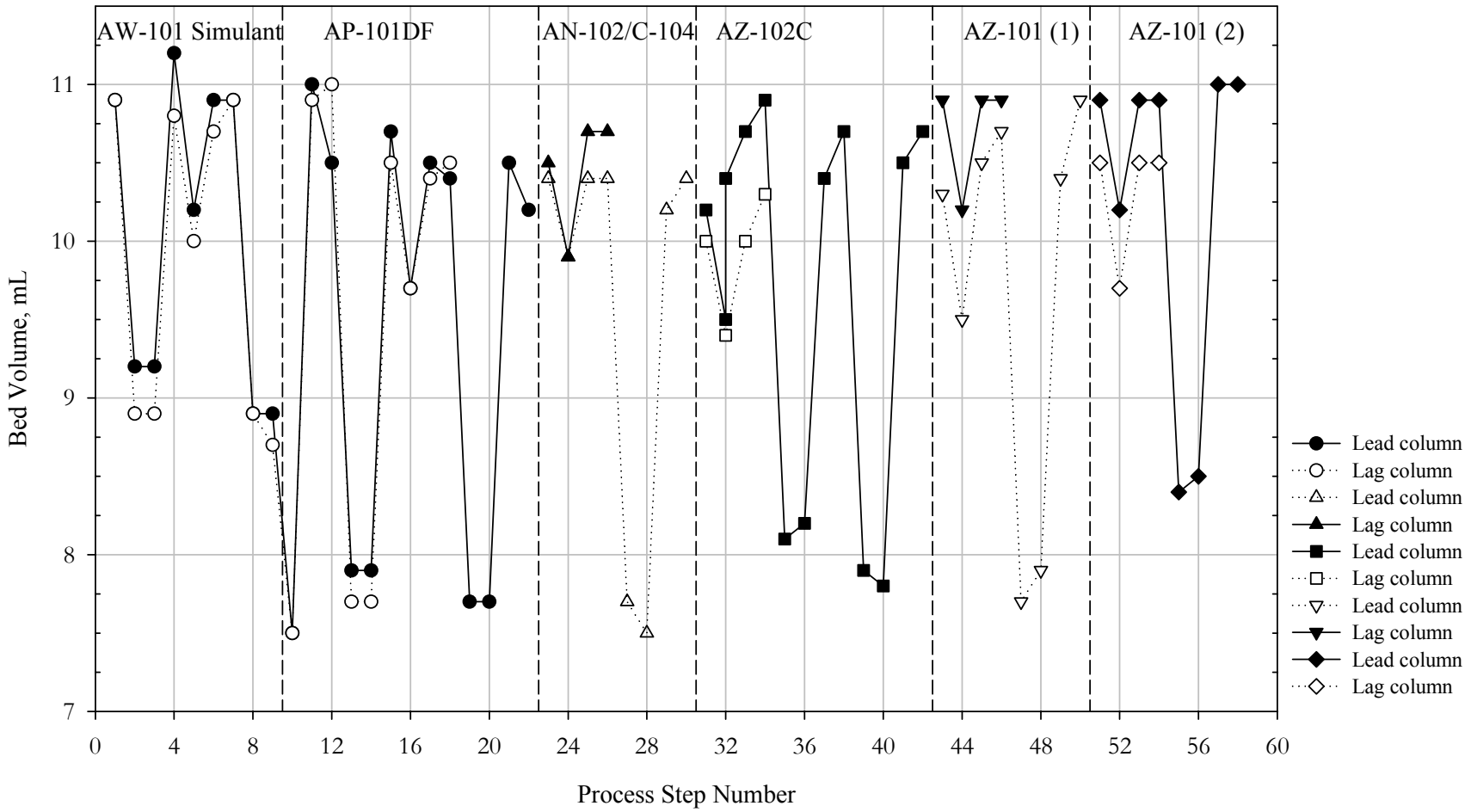
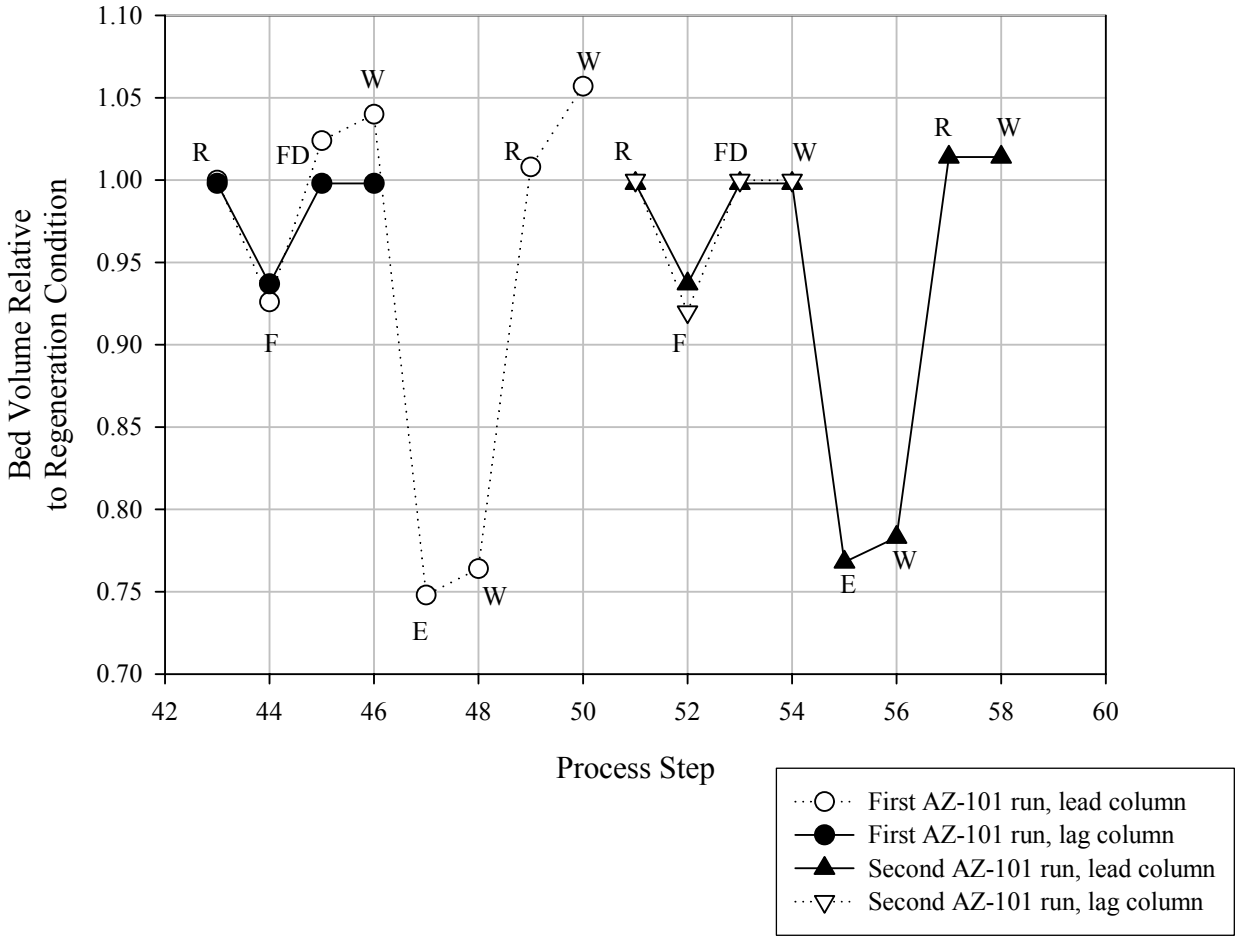


Figure 3.8. Comparison of BVs of the Lead and Lag Columns for all Test Cycles (SL-644 Batch 010319SMC-IV-73, 212-to 425-µm; Solid Points Indicate Column 1, Hollow Points Indicate Column 2)

In Table 3.17 and Figure 3.9, the BVs are normalized to the volume in the 0.25 M NaOH regeneration condition just before AZ-101 loading. Again the process-step number is referenced to the process step in Table 3.11. The relative BV changes remained fairly consistent for the two resin beds.

Table 3.17. Relative Bed Volume for AZ-101 Processing

Feed	Symbol	Process step	BV Relative to Regeneration Condition	
			Column 1	Column 2
Column Position			**Lag**	**Lead**
0.25 M NaOH	R	43	1.00	1.00
AZ-101, first test	F	44	0.94	0.93
0.1 M NaOH	FD	45	1.00	1.02
DI water	W	46	1.00	1.04
0.5 M HNO_3	E	47	—	0.75
DI water	W	48	—	0.76
0.25 M NaOH	R	49	—	1.01
DI water	W	50	—	1.06
Switch Column Positions			**Lead**	**Lag**
0.25 M NaOH	R	51	1.00	1.00
AZ-101, second test	F	52	0.94	0.92
0.1 M NaOH	FD	53	1.00	1.00
DI water	W	54	1.00	1.00
0.5 M HNO_3	E	55	0.77	—
DI water	W	56	0.78	—
0.25 M NaOH	R	57	1.02	—
DI water	W	58	1.02	—
"—" indicates no data				



Regeneration condition BVs:

First run, lead column 10.3 mL	Second run, lead column 10.9 mL
First run, lag column 10.9 mL	Second run, lag column 10.5 mL

Figure 3.9. Relative Bed Volumes For AZ-101 Processing

4.0 Conclusions and Recommendations

Batch-distribution coefficients were determined as a function of Na/Cs mole ratios for SL-644 in AZ-101.

- The SL-644 equilibrium data resulted in a K_d value of 520 mL/g, corresponding to a predicted Cs λ of 124 BVs (0.25 M NaOH condition and bed density of 0.239 g/mL), at a Na/Cs mole ratio of 1.09 E+4, 23 to 27°C

Cs decontamination from AZ-101 (Envelope B) was successfully demonstrated for the first processing run, but failed the second processing run.

- An overall DF of 2.5 E+4 was obtained for the first test after processing 123 BVs, providing a Cs-decontaminated effluent with a ^{137}Cs concentration of 6.5 E-2 μCi/mL. This represented 19% of the contract limit of 3.39 E-1 μCi/mL in the treated effluent (based on the minimum 5 wt% waste Na_2O loading).
- An overall DF of 2.3 E+3 was obtained for the second test after processing 96 BVs, providing a Cs-decontaminated effluent with a ^{137}Cs concentration of 7.0 E-1 μCi/mL. This represented 200% of the contract limit. The low DF was mostly attributed to Cs bleed-off from the lag column, a result of inadequate baseline elution from the previous waste-processing test and the higher flowrate.

Cs load and elution breakthrough profiles were developed.

- The first test 50% Cs breakthrough for the lead ion exchange column was interpolated at 122 BVs (0.25 M NaOH regeneration condition) at a flowrate of 1.5 BV/h. This was nearly equivalent to the predicted 124 BVs based on batch-contact studies.
- The second test, which estimated 50% Cs breakthrough for the lead ion exchange column, was extrapolated from 6% C/C_o to 50% C/C_o at 110 BVs (0.25 M NaOH regeneration condition) at a flowrate of 2.7 BV/h. This indicated that it may be possible to increase the processing rate.
- The second Cs load profile resulted in higher C/C_o values relative to the first load profile; the profile slopes were similar. The Cs load profile offset was attributed to two factors: buildup of residual Cs and its associated bleed-off from the column and the faster feed flowrate.
- The Cs-loaded lead column was efficiently eluted with 0.5 M HNO_3. The peak C/C_o values for ^{137}Cs were 105 and 88 for the lead column, the first and second test, respectively. Over 99% of the ^{137}Cs was eluted from the column in 2.5 BVs of eluate. A total of 11 BVs eluate were required to reach the elution end point of $C/C_o = 0.01$.
- The elution profiles for the two flowrates tested (1.0 BV/h and 1.8 BV/h) were virtually identical.

The effectiveness of all SL-644 ion exchange process steps was demonstrated.

- The regeneration solution effluent was slightly acidic; the calculated Na uptake by the resin bed appeared to be 17% low.
- Activity balances indicated 112% and 114% of the ^{137}Cs present in the feed sample plus carryover from previous testing were accounted for in the process streams (mostly in the eluate), which is indicative of good experimental integrity.

An additional elution study and alternate elution evaluation are recommended.

- Cs elution needs to be better optimized. Larger elution volumes at faster flowrates are recommended.
- Evaluation of Cs elution should be moved away from the 1% C/C_o criterion. The final Cs concentration eluting from the column will probably be a better indicator of follow-on performance.

5.0 References

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Appendix A

General Calculations

Appendix A: General Calculations

^{137}Cs Contractual Limit and Design Basis Limit in AZ-101 Envelope B Vitrification Feed

Assumptions-Minimum Waste Loading

1) Concentration of Na_2O in Env. B glass = 5% (= 5 g Na_2O / 100 g glass)

2) For maximum ^{137}Cs concentration in glass, assume all Na comes from the feed. If some Na is added to Vit feed, multiply the maximum ^{137}Cs value determined below by ratio of total Na:feed Na.

3) Glass density = 2.66 MT/m^3 (=2.66 g/mL)

4) Maximum Cs-137 in glass = 0.3 Ci/m^3 (=3 Ci / 1E+6 mL = 3E-7 Ci/mL)

5) AZ-101 (C = concentrate) actual waste Na concentration = 4.85 M

6) AZ-101 actual waste ^{137}Cs concentration = 1.64 E3 μCi / mL / 4.85 M Na

Na Loading in Glass

5 g Na_2O / 100g glass * 1 mole Na_2O / 62 g Na_2O) * (2 mole Na/ mole Na_2O)* (23 g Na / mole Na) * (2.66 g glass / mL glass) = 0.0987 g Na / mL glass

Maximum ^{137}Cs:Na in glass

(3.0E-7 Ci ^{137}Cs / mL glass) / (0.0987 g Na / mL glass) = 3.04 E-6 Ci ^{137}Cs / g Na

(3.04 E-6 Ci ^{137}Cs / g Na) * (23 g Na / mole) = 6.99E-5 Ci ^{137}Cs / mole Na

Maximum ^{137}Cs:Na in feed

(6.99E-5 Ci ^{137}Cs / mole Na) * (4.85 mole Na / L feed) = 3.39 E-4 Ci ^{137}Cs / L
= 3.39 E+2 μCi ^{137}Cs / L
= 0.339 μCi ^{137}Cs / mL

AZ-101 actual waste Cs fraction remaining (C/C_o) Contractual Limit

(0.339 μCi ^{137}Cs/ mL) / (1.64E3 μCi ^{137}Cs / mL) = 2.07 E-4 C/C_o
= 0.0207 % C/C_o

Decontamination factor

(1.64E+3 μCi ^{137}Cs/ mL) / (3.39E-1 μCi ^{137}Cs/ mL) = 4840

^{137}Cs Contractual Limit and Design Basis Limit in AZ-101 Envelope B Vitrification Feed

Assumptions-Maximum Waste Loading

1) Concentration of Na_2O in Env. B glass = 5.5% (= 5.5 g Na_2O / 100 g glass)

2) For maximum ^{137}Cs concentration in glass, assume all Na comes from the feed. If some Na is added to Vit feed, multiply the maximum ^{137}Cs value determined below by ratio of total Na:feed Na.

3) Glass density = 2.66 MT/m^3 (=2.66 g/mL)

4) Maximum Cs-137 in glass = 0.3 Ci/m^3 (=3 Ci / 1E+6 mL = 3E-7 Ci/mL)

5) AZ-101 (C = concentrate) actual waste Na concentration = 4.85 M

6) AZ-101 actual waste ^{137}Cs concentration = 1.64 E3 μCi / mL / 4.85 M Na

Na Loading in Glass

5.5 g Na_2O / 100g glass * 1 mole Na_2O / 62 g Na_2O) * (2 mole Na/ mole Na_2O)* (23 g Na / mole Na) * (2.66 g glass / mL glass) = 0.109 g Na / mL glass

Maximum ^{137}Cs:Na in glass

(3.0E-7 Ci ^{137}Cs / mL glass) / (0.109 g Na / mL glass) = 2.75 E-6 Ci ^{137}Cs / g Na

(2.75 E-6 Ci ^{137}Cs / g Na) * (23 g Na / mole) = 6.33E-5 Ci ^{137}Cs / mole Na

Maximum ^{137}Cs:Na in feed

(6.33E-5 Ci ^{137}Cs / mole Na) * (4.85 mole Na / L feed) = 3.07 E-4 Ci ^{137}Cs / L
= 3.07 E+2 μCi ^{137}Cs / L
= 0.307 μCi ^{137}Cs / mL

AZ-101 actual waste Cs fraction remaining (C/C_o) Contractual Limit

(0.307 μCi ^{137}Cs/ mL) / (1.64E3 μCi ^{137}Cs / mL) = 1.87 E-4 C/C_o
= 0.0187 % C/C_o

Decontamination factor

(1.64E+3 μCi ^{137}Cs/ mL) / (3.07E-1 μCi ^{137}Cs/ mL) = 5340

Appendix B

Batch Contact Calculations

Appendix B: Batch Contact Calculations

Table B.1. Batch Distribution Test and Results

		m	**F**	**I_{Na}**			**V**	**C_o**				**C_{eq}**	
CMC ID	Sample ID	H-form SL-644 Resin, g	F-factor	I_{Na} factor	corrected resin mass, g	AZ-101, g	AZ-101, mL	Starting [Cs], M	[Na], M	Net Cs-137 µCi/mL	Fraction of original Cs-137	Equilibrium [Cs], M	Equilibrium Na:Cs mole ratio
02--2288	AZ101-TI-164-S0	0	1	1	0	3.6911	3.0161	4.48 E-4	4.85	1890	1	4.48 E-4	1.08 E+4
02--2289	AZ101-TI-164-S0-D	0	1	1	0	3.6951	3.0194	4.48 E-4	4.85	2030	1	4.48 E-4	1.08 E+4
02--2290	AZ101-TI-164-S0-73	0.0733	0.762	1.25	0.0698	8.6767	7.0900	4.48 E-4	4.85	163	0.0832	3.73 E-5	1.30 E+5
02--2291	AZ101-TI-164-S0-73D	0.0732	0.762	1.25	0.0697	8.6868	7.0982	4.48 E-4	4.85	177	0.0903	4.05 E-5	1.20 E+5
02--2292	AZ101-TI-164-S1	0	1	1	0	3.6560	2.9874	4.50 E-3	4.85	1980	1	4.50 E-3	1.08 E+3
02--2293	AZ101-TI-164-S1-D	0	1	1	0	3.4286	2.8016	4.50 E-3	4.85	762	not used	not used	not used
02--2294	AZ101-TI-164-S1-73	0.0732	0.762	1.25	0.0697	8.6619	7.0779	4.50 E-3	4.85	773	0.390	1.76 E-3	2.76 E+3
02--2295	AZ101-TI-164-S1-73D	0.0731	0.762	1.25	0.0696	8.7230	7.1278	4.50 E-3	4.85	791	0.399	1.80 E-3	2.70 E+3
02--2296	AZ101-TI-164-S2	0	1	1	0	3.6433	2.9770	7.27 E-3	4.85	1820	1	7.27 E-3	6.67 E+2
02--2297	AZ101-TI-164-S2-D	0	1	1	0	3.5930	2.9359	7.27 E-3	4.85	1880	1	7.27 E-3	6.67 E+2
02--2298	AZ101-TI-164-S2-73	0.0723	0.762	1.25	0.0689	8.6588	7.0753	7.27 E-3	4.85	979	0.529	3.85 E-3	1.26 E+3
02--2299	AZ101-TI-164-S2-73D	0.0728	0.762	1.25	0.0693	8.6771	7.0903	7.27 E-3	4.85	1010	0.546	3.97 E-3	1.22 E+3

		K_d	**ρ**	**λ**	**ρ**	**λ**
CMC ID	Sample ID	K_d, mL/g	Lead column dry bed density, g/mL	Lead column λ, BVs	Lag column dry bed density, g/mL	Lag column λ, BVs
02--2290	AZ101-TI-164-S0-73	1120	0.239	268	0.228	255
02--2291	AZ101-TI-164-S0-73D	1026	0.239	245	0.228	234
02--2294	AZ101-TI-164-S1-73	159	0.239	37.9	0.228	36.1
02--2295	AZ101-TI-164-S1-73D	154	0.239	36.8	0.228	35.1
02--2298	AZ101-TI-164-S2-73	91	0.239	21.8	0.228	20.8
02--2299	AZ101-TI-164-S2-73D	85	0.239	20.3	0.228	19.4

Where: $K_d = (C_o - C_{eq})/C_{eq} \times V/(m*F*I_{Na})$, and

$\lambda = K_d * \rho$

Appendix C

Column Testing Calculations

Appendix C: Column Testing Calculations

AZ-101 First Ion Exchange Test Calculations

Table C1. First AZ-101 Cs Ion Exchange Column Test: Solution Flows Through Columns

AZ-101 Test Run
SL-644 batch 010310SMC-IV-73 212-425 um particle size

Bed volume in AZ-101 Feed =	9.5	mL
Bed volume in 0.25M NaOH =	10.3	mL
Apparatus volume =	42	mL
Column 1 apparatus volume =	23	mL

Solution	Density	
HNO3	1.012	g/mL
0.1M NaOH	0.999	g/mL
0.25M NaOH	1.006	g/mL
AZ-101	1.2238	g/mL
DI water	0.997	g/mL

Notes:
start = start time of flow through column
end = end time of flow through column
Δt = time interval
BV = bed volume
cumul. Vol = cumulative volume collected
BV (RGN) = bed volumes relative to regeneration condition
BV (feed) = bed volumes relative to feed condition of columns

DI water displacement 12/3/01
Lead column + Lag column

start	end	Δt	mass, g	volume mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	volume, AV
0.25M NaOH wash								
12/3/2001 8:53	12/3/2001 12:32	3:39	84.9	84.4	0.39	2.24	8.2	2.01

AZ-101 actual waste loading starting on 12/3/01

Initial effluent collection (1 apparatus volume of feed)

start	end	Δt	mass, g	cumul. vol., mL	flow rate mL/min	flowrate, BV/hr	volume, AV
12/3/2001 12:40	12/3/2001 15:30	2:50	42.4	42.1	0.248	1.44	1.0
Δt, total	2:50						

Feed flow								**Lead column sampling**						Running
start	end	Δt	mass g	cumul. vol., mL	flowrate mL/min	flowrate BV/hr	cumulat. BV	Sample ID	mass g	volume mL	total vol. mL	**BV (RGN)**	**BV (feed)**	total sample vol. mL
12/3/2001 15:30	12/3/2001 15:58	0:28	6.8	47.7	0.198	1.16	4.6	AZ101L-F1	2.7501	2.247	49.951	**4.8**	**5.3**	2.2472
16:17	19:23	3:06	53.2	91.2	0.234	1.36	8.9	AZ101L-F2	3.1482	2.572	98.123	**9.5**	**10.3**	4.8197
19:43	22:40	2:57	57.0	137.8	0.263	1.53	13.4	AZ101L-F3	2.8293	2.312	149.743	**14.5**	**15.8**	7.1316
12/3/2001 23:00	12/4/2001 1:59	2:59	56.6	184.0	0.258	1.51	17.9	AZ101L-F4	2.3050	1.883	200.180	**19.4**	**21.1**	9.0150
2:17	5:19	3:02	55.8	229.6	0.251	1.46	22.3	AZ101L-F5	2.3547	1.924	249.410	**24.2**	**26.3**	10.9391
5:33	8:40	3:07	57.0	276.2	0.249	1.45	26.8	AZ101L-F6	2.9041	2.373	299.833	**29.1**	**31.6**	13.3121
9:00	12:00	3:00	55.6	321.6	0.252	1.47	31.2	AZ101L-F7	2.8986	2.369	350.316	**34.0**	**36.9**	15.6807
12:18	15:20	3:02	56.2	367.5	0.252	1.47	35.7	AZ101L-F8	2.4763	2.023	400.373	**38.9**	**42.1**	17.7041
15:35	18:38	3:03	67.5	422.7	0.301	1.76	41.0	AZ101L-F9	3.5837	2.928	460.162	**44.7**	**48.4**	20.6325
18:59	21:51	2:52	51.8	465.0	0.246	1.43	45.1	AZ101L-F10	4.2025	3.434	508.218	**49.3**	**53.5**	24.0664
12/4/2001 22:11	12/5/2001 1:20	3:09	52.5	507.9	0.227	1.32	49.3	AZ101L-F11	3.5117	2.870	555.837	**54.0**	**58.5**	26.9359
1:43	4:40	2:57	55.0	552.9	0.254	1.48	53.7	AZ101L-F12	3.4399	2.811	606.077	**58.8**	**63.8**	29.7468
4:48	8:01	3:13	55.8	598.4	0.236	1.38	58.1	AZ101L-F13	3.3000	2.697	656.343	**63.7**	**69.1**	32.4433
8:17	11:20	3:03	56.7	644.8	0.253	1.47	62.6	AZ101L-F14	2.7125	2.216	706.965	**68.6**	**74.4**	34.6597
11:34	14:40	3:06	56.8	691.2	0.250	1.45	67.1	AZ101L-F15	2.8166	2.302	757.569	**73.6**	**79.7**	36.9613
14:55	17:59	3:04	55.9	736.9	0.248	1.45	71.5	AZ101L-F16	2.7899	2.280	807.415	**78.4**	**85.0**	39.2410
18:13	21:19	3:06	56.5	783.0	0.248	1.45	76.0	AZ101L-F17	2.5755	2.105	857.396	**83.2**	**90.3**	41.3455
12/5/2001 21:34	12/6/2001 0:39	3:05	57.9	830.3	0.256	1.49	80.6	AZ101L-F18	2.7378	2.237	908.890	**88.2**	**95.7**	43.5826
0:54	3:58	3:04	56.2	876.3	0.250	1.45	85.1	AZ101L-F19	2.6281	2.147	958.956	**93.1**	**100.9**	45.7301
4:15	7:20	3:05	57.0	922.8	0.252	1.47	89.6	AZ101L-F20	2.6353	2.153	1009.633	**98.0**	**106.3**	47.8835
7:35	10:42	3:07	57.0	969.4	0.249	1.45	94.1	AZ101L-F21	2.5520	2.085	1060.011	**102.9**	**111.6**	49.9688
10:58	14:00	3:02	56.4	1015.5	0.253	1.48	98.6	AZ101L-F22	3.0035	2.454	1110.594	**107.8**	**116.9**	52.4230
14:20	17:20	3:00	55.9	1061.2	0.254	1.48	103.0	AZ101L-F23	2.8459	2.325	1161.037	**112.7**	**122.2**	54.7485
17:36	20:40	3:04	54.5	1105.7	0.242	1.41	107.4	AZ101L-F24	2.6919	2.200	1210.003	**117.5**	**127.4**	56.9481
12/6/2001 20:55	12/7/2001 0:00	3:05	57.2	1152.5	0.253	1.47	111.9	AZ101L-F25	2.5708	2.101	1261.388	**122.5**	**132.8**	59.0488
Δt, total:	83:20:00			**Average**	**0.25**	**1.46**								

AZ-101 Feed total:

1263 mL through the **lead** column

1.263 L

1204 mL through the **lag** column

1.204 L

Lag column sampling

Date	Sample ID	sample mass, g	volume, mL	total vol., mL	BV (RGN)	BV (feed)	Running total sample
12/3/01	AZ-101P-F1	2.6050	2.129	49.83	**4.8**	**5.2**	2.129
12/3/01	AZ-101P-F2	2.7478	2.731	96.03	**9.3**	**10.1**	4.860
12/3/01	AZ-101P-F3	2.8207	2.305	144.92	**14.1**	**15.3**	7.165
12/4/01	AZ-101P-F4	2.0928	1.710	192.88	**18.7**	**20.3**	8.875
12/4/01	AZ-101P-F5	1.8035	1.474	239.94	**23.3**	**25.3**	10.349
12/4/01	AZ-101P-F6	3.2823	2.682	289.20	**28.1**	**30.4**	13.031
12/4/01	AZ-101P-F7	2.5831	2.111	336.75	**32.7**	**35.4**	15.141
12/4/01	AZ-101P-F8	2.0865	1.705	384.37	**37.3**	**40.5**	16.846
12/4/01	AZ-101P-F9	2.8088	2.295	441.82	**42.9**	**46.5**	19.142
12/4/01	AZ-101P-F10	2.2646	1.850	486.00	**47.2**	**51.2**	20.992
12/5/01	AZ-101P-F11	3.0435	2.487	531.39	**51.6**	**55.9**	23.479
12/5/01	AZ-101P-F12	2.4150	1.973	578.30	**56.1**	**60.9**	25.452
12/5/01	AZ-101P-F13	2.5392	2.075	625.97	**60.8**	**65.9**	27.527
12/5/01	AZ-101P-F14	2.3124	1.890	674.19	**65.5**	**71.0**	29.417
12/5/01	AZ-101P-F15	2.3111	1.888	722.50	**70.1**	**76.1**	31.305
12/5/01	AZ-101P-F16	2.0918	1.709	769.88	**74.7**	**81.0**	33.014
12/5/01	AZ-101P-F17	2.3802	1.945	818.00	**79.4**	**86.1**	34.959
12/6/01	AZ-101P-F18	2.4435	1.997	867.30	**84.2**	**91.3**	36.956
12/6/01	AZ-101P-F19	2.3827	1.947	915.17	**88.9**	**96.3**	38.903
12/6/01	AZ-101P-F20	2.1003	1.716	963.47	**93.5**	**101.4**	40.619
12/6/01	AZ-101P-F21	2.5000	2.043	1012.08	**98.3**	**106.5**	42.662
12/6/01	AZ-101P-F22	2.9860	2.440	1060.61	**103.0**	**111.6**	45.102
12/6/01	AZ-101P-F23	2.7332	2.233	1108.52	**107.6**	**116.7**	47.335
12/7/01	AZ-101P-F24	3.1143	2.545	1155.60	**112.2**	**121.6**	49.880
12/7/01	AZ-101P-F25	2.1790	1.781	1204.12	**116.9**	**126.7**	51.661

0.1M NaOH feed displacement 12/7/2001

Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	flowrate mL/min	flowrate BV/hr	volume, BV	cumul. BV	volume, AV	Cumul. AV	Applied density, g/mL
AZ101-FD-1	0:24	0:34	0:10	3.75	3.09	0.31	1.80	0.30	0.30	0.07	0.07	1.213
AZ101-FD-2	0:34	0:54	0:20	8.66	7.11	0.36	2.07	0.69	0.99	0.17	0.24	1.217
AZ101-FD-3	0:54	1:14	0:20	9.41	7.75	0.39	2.26	0.75	1.74	0.18	0.43	1.215
AZ101-FD-4	1:14	1:34	0:20	11.28	9.29	0.46	2.70	0.90	2.64	0.22	0.65	1.215
AZ101-FD-5	1:34	1:54	0:20	13.31	10.94	0.55	3.19	1.06	3.71	0.26	0.91	1.216
AZ101-FD-6	1:54	2:14	0:20	13.75	11.68	0.58	3.40	1.13	4.84	0.28	1.19	1.178
AZ101-FD-7	2:14	2:34	0:20	14.68	14.28	0.71	4.16	1.39	6.23	0.34	1.53	1.028
AZ101-FD-8	2:34	2:54	0:20	10.86	10.80	0.54	3.14	1.05	7.27	0.26	1.78	1.006
AZ101-FD-9	2:54	3:14	0:20	9.92	9.94	0.50	2.90	0.97	8.24	0.24	2.02	0.998
	Δt, total:	2:50			**Average:**	**0.49**	**2.85**					
				Sum:	**84.9 mL**							

(Density determined from analytical sub-sample.)

DI water rinse	12/7/2001										
Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	flowrate mL/min	flowrate BV/hr	volume, BV	cumul. BV	volume, AV	Cumul. AV
AZ101-Fdi-1	3:21	3:41	0:20	9.569	9.569	0.48	2.79	0.93	0.93	0.23	0.23
AZ101-Fdi-2	3:41	4:01	0:20	10.071	10.071	0.50	2.93	0.98	1.91	0.24	0.47
AZ101-Fdi-3	4:01	4:21	0:20	10.346	10.346	0.52	3.01	1.00	2.91	0.25	0.71
AZ101-Fdi-4	4:21	4:41	0:20	9.903	9.903	0.50	2.88	0.96	3.87	0.24	0.95
AZ101-Fdi-5	4:41	5:01	0:20	9.873	9.873	0.49	2.88	0.96	4.83	0.24	1.18
AZ101-Fdi-6	5:01	5:21	0:20	9.892	9.892	0.49	2.88	0.96	5.79	0.24	1.42
AZ101-Fdi-7	5:21	5:41	0:20	10.114	10.114	0.51	2.95	0.98	6.77	0.24	1.66
AZ101-Fdi-8	5:41	6:02	0:21	10.614	10.614	0.51	2.94	1.03	7.80	0.25	1.91
AZ101-Fdi-9	6:02	6:24	0:22	10.966	10.966	0.50	2.90	1.06	8.87	0.26	2.17
	Δt, total:	3:03			**Average:**	**0.50**	**2.91**				
				Sum:	**91.3**						

Cs elution from lead column, Separate columns 12/7/01	Sample ID	start	end	Δt	mass collected, g	volume, mL	flowrate, mL/min	flowrate, BV/hr	cumul. BV	
	HNO3 wash--lead column									
	AZ101L-E1	6:30	7:32	1:02	10.4547	10.33	0.17	0.97	1.00	
	AZ101L-E2	7:32	8:29	0:57	8.8291	8.72	0.15	0.89	1.85	
	AZ101L-E3	8:30	9:31	1:01	9.7991	9.68	0.16	0.92	2.79	
	AZ101L-E4	9:32	10:29	0:57	9.1501	9.04	0.16	0.92	3.67	
	AZ101L-E5	10:58	11:30	0:32	10.2866	10.16	0.32	1.85	4.65	Flow was impeded for 30 min. then resumed
	AZ101L-E6	11:31	12:30	0:59	9.8248	9.71	0.16	0.96	5.60	
	AZ101L-E7	12:31	13:30	0:59	9.9926	9.87	0.17	0.97	6.56	
	AZ101L-E8	13:31	14:30	0:59	10.0228	9.90	0.17	0.98	7.52	
	AZ101L-E9	14:32	15:30	0:58	9.6482	9.53	0.16	0.96	8.44	
	AZ101L-E10	15:31	16:30	0:59	10.0266	9.91	0.17	0.98	9.41	
	AZ101L-E11	16:33	17:30	0:57	9.9282	9.81	0.17	1.00	10.36	
	AZ101L-E12	17:31	18:30	0:59	10.1534	10.03	0.17	0.99	11.33	
	AZ101L-E13	18:31	19:29	0:58	10.0085	9.89	0.17	0.99	12.29	
	AZ101L-E14	19:30	20:30	1:00	10.3235	10.20	0.17	0.99	13.28	
	AZ101L-E15	20:31	21:30	0:59	10.1550	10.03	0.17	0.99	14.26	
	Δt, total:	15:00		**Sum:**	**148.60**	**146.84**				
						Average:	**0.18**	**1.03**		

DI water rinse start 12/7/01											
Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	Cumul. BV	volume, AV	Cumul. AV
AZ101-Edi-1	21:39	22:00	0:21	11.822	11.857	0.56	3.29	1.2	1.2	0.52	0.52
AZ101-Edi-3	22:00	22:22	0:22	10.706	10.738	0.49	2.84	1.0	2.2	0.47	0.98
AZ101-Edi-4	22:23	22:43	0:20	10.359	10.390	0.52	3.03	1.0	3.2	0.45	1.43
AZ101-Edi-5	22:44	23:03	0:19	9.714	9.743	0.51	2.99	0.9	4.1	0.42	1.86
AZ101-Fdi-10	23:04	23:24	0:20	10.090	10.120	0.51	2.95	1.0	5.1	0.44	2.30
	Δt, total:	1:45		**Sum:**	**52.8**						
					Average:	**0.52**	**3.02**				

Regeneration with 0.25M NaOH start 12/10/01												
Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	Cumul. BV	volume, AV	Cumul. AV	
AZ101L-RGN	8:52	10:14	1:22	14.9	14.8	0.18	1.05	1.4	1.4	0.64	0.64	
	10:27	11:46	1:19	13.0	12.9	0.16	0.95	1.3	2.7	0.56	1.21	note: time corrected for down-time
	11:48	13:30	1:42	13.4	13.3	0.13	0.76	1.3	4.0	0.58	1.78	while lowering column
	13:32	14:12	0:40	5.3	5.3	0.13	0.77	0.5	4.5	0.23	2.01	
	Δt, total:	4:38		**Sum:**	**46.3**							
					Average:	**0.15**	**0.88**					

Rinse with DI water start 12/10/01											
Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	Cumul. BV	volume, AV	Cumul. AV
AZ101-DIRinse-Final	14:17	14:48	0:31	13.4	13.3	0.43	2.50	1.3	1.3	0.58	0.58
	14:49	15:22	0:33	15.2	15.2	0.46	2.68	1.5	2.8	0.66	1.24
	15:23	15:55	0:32	14.2	14.2	0.44	2.58	1.4	4.1	0.62	1.86
	Δt, total:	1:38		**Sum:**	**42.7**						
					Average:	**0.44**	**2.59**				

Table C.2. First AZ-101 Cs Ion Exchange Column Test: Counting Data

Counting Data for TI-RPP-WTP-131, Rev. 0

Background Counts

Date	Net counts	Uncertainty	Time (sec)	cpm	uncertainty %
12/4/01 11:29	319	21	300	63.80	7%
12/4/01 15:00	3074	70	3000	61.48	2%
12/5/01 9:44	3191	71	3000	63.82	2%
12/6/01 13:13	3228	70	3000	64.56	2%
12/6/01 23:55	3992	76	3000	79.84	2%
12/7/01 7:30	3991	78	3000	79.82	2%
12/7/01 0:00	48218	269	36000	80.36	1%
12/10/01 0:00	3844	77	3000	76.88	2%
12/12/01 8:10	3979	78	3000	79.58	2%
			average	**72.24** cpm	

Notes: AZ-101 density = **1.2238** g/mL
cpm = counts per minute
DF = decontamination factor
position = indicates relative position sample is relative to detector

AZ101 Comparitor Standards

F0DD-A: 0.1174 g of AZ-101 into 10.1049g (total), then 0.0944g of this into 10.0489g (total), then 2 mL geometry (1.9310 g, diluent is 0.01M NaOH).

2.11E-04 g AZ-102 — F0DD-A Position 1 on counter

Date	Net counts	Error	Time (sec)	cpm	net cpm	mass, g	net cpm/g	mL	cpm/mL
12/4/01 11:22	23129	168	300	4626	4554	2.11E-4	2.16E+7	1.72E-4	2.64E+7
12/4/01 19:05	22259	167	300	4452	4380	2.11E-4	2.08E+7	1.72E-4	2.54E+7
12/4/01 20:16	21277	162	300	4255	4183	2.11E-4	1.98E+7	1.72E-4	2.43E+7
12/5/01 12:48	22509	169	300	4502	4430	2.11E-4	2.10E+7	1.72E-4	2.57E+7
12/6/01 5:32	21626	163	300	4325	4253	2.11E-4	2.02E+7	1.72E-4	2.47E+7
12/7/01 2:32	21673	166	300	4335	4262	2.11E-4	2.02E+7	1.72E-4	2.48E+7
12/7/01 14:44	22389	171	300	4478	4406	2.11E-4	2.09E+7	1.72E-4	2.56E+7
12/7/01 21:02	22414	168	300	4483	4411	2.11E-4	2.09E+7	1.72E-4	2.56E+7
12/10/01 14:27	21785	163	300	4357	4285	2.11E-4	2.03E+7	1.72E-4	2.49E+7
12/11/01 9:31	23013	166	300	4603	4530	2.11E-4	2.15E+7	1.72E-4	2.63E+7
12/12/01 8:00	22059	164	300	4412	4340	2.11E-4	2.06E+7	1.72E-4	2.52E+7
						Average	**2.07E+7**		**2.54E+7**
						standard deviation	**5.66E+5**		**6.93E+5**
						% standard deviation	**2.7**		**2.7**

F0D-A: 0.1174 g of AZ-101 brought to 10.1049g total, then 2 g of this were placed into a 2-mL geometry (diluent is 0.01M NaOH).

0.022525222 g AZ-101 — F0D-A, Position 6 on counter

Date	Net counts	Error	Time (sec)	cpm	net cpm	mass	net cpm/g	mL	cpm/mL
12/6/2001	88701	334	300	17740	17668	0.0225	7.84E+5	0.0184	9.60E+5
12/7/2001	87245	335	300	17449	17377	0.0225	7.71E+5	0.0184	9.44E+5
12/10/2001	89077	336	300	17815	17743	0.0225	7.88E+5	0.0184	9.64E+5
						Average	**7.81E+5**		**9.56E+5**
						standard deviation	**8.59E+3**		**1.05E+4**
						% standard deviation	**1.1**		**1.1**

AZ-101 run starting 12/3/01			**Lead column, loading phase**												
Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	% C/Co	DF	Position	^{137}Cs µCi/mL	^{137}Cs µCi
AZ101L-F1	12/4/01	1979	300	323.56	2.5939	2.1195	125	153	6.02E-6	**4.0**	6.02E-4	1.7 E+5	1	9.90 E-3	2.10 E-2
AZ101L-F2	12/4/01	2431	300	413.96	2.9265	2.3913	141	173	6.83E-6	**9.5**	6.83E-4	1.5 E+5	1	1.12 E-2	2.69 E-2
AZ101L-F3	12/4/01	2263	300	380.36	2.6758	2.1865	142	174	6.86E-6	**14.5**	6.86E-4	1.5 E+5	1	1.13 E-2	2.47 E-2
AZ101L-F4	12/4/01	1752	300	278.16	2.1379	1.7469	130	159	6.28E-6	**19.4**	6.28E-4	1.6 E+5	1	1.03 E-2	1.80 E-2
AZ101L-F5	12/4/01	1736	300	274.96	2.2453	1.8347	122	150	5.91E-6	**24.2**	5.91E-4	1.7 E+5	1	9.72 E-3	1.78 E-2
AZ101L-F6	12/4/01	1947	300	317.16	2.8087	2.2951	113	138	5.45E-6	**29.1**	5.45E-4	1.8 E+5	1	8.97 E-3	2.06 E-2
AZ101L-F7	12/5/01	1694	300	266.56	2.3643	1.9319	113	138	5.44E-6	**34.0**	5.44E-4	1.8 E+5	1	8.95 E-3	1.73 E-2
AZ101L-F8	12/5/01	1560	300	239.76	2.3566	1.9256	102	125	4.91E-6	**38.9**	4.91E-4	2.0 E+5	1	8.08 E-3	1.56 E-2
AZ101L-F9	12/5/01	1415	300	210.76	2.3397	1.9118	90	110	4.35E-6	**44.7**	4.35E-4	2.3 E+5	1	7.15 E-3	1.37 E-2
AZ101L-F10	12/5/01	1480	300	223.76	2.352	1.9219	95	116	4.59E-6	**49.3**	4.59E-4	2.2 E+5	1	7.55 E-3	1.45 E-2
AZ101L-F11	12/5/01	1480	300	223.76	2.347	1.9178	95	117	4.60E-6	**54.0**	4.60E-4	2.2 E+5	1	7.57 E-3	1.45 E-2
AZ101L-F12	12/5/01	1427	300	213.16	2.3527	1.9225	91	111	4.37E-6	**58.8**	4.37E-4	2.3 E+5	1	7.19 E-3	1.38 E-2
AZ101L-F13	12/5/01	1300	300	187.76	2.3615	1.9296	80	97	3.84E-6	**63.7**	3.84E-4	2.6 E+5	1	6.31 E-3	1.22 E-2
AZ101L-F14	12/6/01	1335	300	194.76	2.4915	2.0359	78	96	3.62E-6	**68.6**	3.62E-4	2.7 E+5	1	5.95 E-3	1.21 E-2
AZ101L-F15	12/6/01	1710	300	269.76	2.5596	2.0915	105	129	4.88E-6	**73.6**	4.88E-4	2.0 E+5	1	8.02 E-3	1.68 E-2
AZ101L-F16	12/6/01	4754	300	878.56	2.4729	2.0207	355	435	1.64E-5	**78.4**	1.64E-3	58318	1	2.70 E-2	5.47 E-2
AZ101L-F17	12/7/01	26149	300	5157.56	2.1334	1.7433	2418	2959	1.12E-4	**83.2**	1.12E-2	8570	1	1.84 E-1	3.21 E-1
AZ101L-F18	12/6/01	2380	300	404	0.0231	0.0189	17486	21400	8.09E-4	**88.2**	8.09E-2	1185	1	1.33 E+0	2.51 E-2
AZ101L-F19	12/6/01	12536	300	2435	0.0263	0.0215	9.28 E+4	1.14 E+5	4.29E-3	**93.1**	4.29E-1	223	1	7.06 E+0	1.51 E-1
AZ101L-F20	12/6/01	61682	300	12264	0.0232	0.0190	5.29 E+5	6.47 E+5	2.45E-2	**98.0**	2.45E+0	39	1	4.02 E+1	7.63 E-1
AZ101L-F21	12/6/01	152358	300	30399	0.0231	0.0189	1.32 E+6	1.61 E+6	6.09E-2	**102.9**	6.09E+0	16	1	1.00 E+2	1.89 E+0
AZ101L-F22	12/6/01	14805	300	2889	0.0230	0.0188	1.26 E+5	1.54 E+5	1.61E-1	**107.8**	1.61E+1	6.2	6	2.64 E+2	4.97 E+0
AZ101L-F23	12/6/01	25341	300	4996	0.0232	0.0189	2.16 E+5	2.64 E+5	2.76E-1	**112.7**	2.76E+1	3.6	6	4.54 E+2	8.60 E+0
AZ101L-F24	12/7/01	37336	300	7395	0.0235	0.0192	3.14 E+5	3.85 E+5	4.02E-1	**117.5**	4.02E+1	2.5	6	6.62 E+2	1.27 E+1
AZ101L-F25	12/7/01	49965	300	9921	0.0239	0.0195	4.15 E+5	5.08 E+5	5.31E-1	**122.5**	5.31E+1	1.9	6	8.73 E+2	1.71 E+1
														sum	**4.68 E+1**

AZ-101 run starting 12/3/01			**Lag column, loading phase**												
Sample ID	Date	Net counts	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF	Position	^{137}Cs µCi/mL	^{137}Cs µCi
AZ101P-F1	12/4/01	1794	300	287	2.4945	2.0383	114.88	140.59	5.54E-6	**4.8**	5.54E-4	1.80E+5	1	9.12 E-3	1.86 E-2
AZ101P-F2	12/4/01	8889	300	1706	2.5492	2.0830	669.06	818.79	3.23E-5	**9.3**	3.23E-3	3.10E+4	1	5.31 E-2	1.11 E-1
AZ101P-F3	12/4/01	19876	300	3903	2.6652	2.1778	1464.42	1792.15	7.07E-5	**14.1**	7.07E-3	1.41E+4	1	1.16 E-1	2.53 E-1
AZ101P-F4	12/4/01	17260	300	3380	1.9726	1.6119	1713.35	2096.80	8.27E-5	**18.7**	8.27E-3	1.21E+4	1	1.36 E-1	2.19 E-1
AZ101P-F5	12/4/01	13846	300	2697	1.6905	1.3814	1595.36	1952.41	7.70E-5	**23.3**	7.70E-3	1.30E+4	1	1.27 E-1	1.75 E-1
AZ101P-F6	12/4/01	24337	300	4795	3.1555	2.5784	1519.62	1859.71	7.33E-5	**28.1**	7.33E-3	1.36E+4	1	1.21 E-1	3.11 E-1
AZ101P-F7	12/5/01	13657	300	2659	2.0797	1.6994	1278.63	1564.78	6.17E-5	**32.7**	6.17E-3	1.62E+4	1	1.02 E-1	1.73 E-1
AZ101P-F8	12/5/01	10394	300	2007	1.8333	1.4980	1094.5	1339.46	5.28E-5	**37.3**	5.28E-3	1.89E+4	1	8.69 E-2	1.30 E-1
AZ101P-F9	12/5/01	10435	300	2015	2.3667	1.9339	851.30	1041.82	4.11E-5	**42.9**	4.11E-3	2.43E+4	1	6.76 E-2	1.31 E-1
AZ101P-F10	12/5/01	8922	300	1712	2.1286	1.7393	804.36	984.38	3.88E-5	**47.2**	3.88E-3	2.58E+4	1	6.39 E-2	1.11 E-1
AZ101P-F11	12/5/01	9123	300	1752	2.3535	1.9231	744.58	911.21	3.59E-5	**51.6**	3.59E-3	2.78E+4	1	5.91 E-2	1.14 E-1
AZ101P-F12	12/5/01	8165	300	1561	2.2277	1.8203	700.62	857.41	3.38E-5	**56.1**	3.38E-3	2.96E+4	1	5.56 E-2	1.01 E-1
AZ101P-F13	12/5/01	8369	300	1602	2.3283	1.9025	687.87	841.81	3.32E-5	**60.8**	3.32E-3	3.01E+4	1	5.46 E-2	1.04 E-1
AZ101P-F14	12/6/01	7426	300	1413	2.0827	1.7018	678.43	830.26	3.27E-5	**65.5**	3.27E-3	3.05E+4	1	5.39 E-2	9.17 E-2
AZ101P-F15	12/6/01	6248	300	1177	2.0773	1.6974	566.78	693.62	2.74E-5	**70.1**	2.74E-3	3.66E+4	1	4.50 E-2	7.64 E-2
AZ101P-F16	12/6/01	5899	300	1108	1.9546	1.5972	566.64	693.46	2.73E-5	**74.7**	2.73E-3	3.66E+4	1	4.50 E-2	7.19 E-2

Lag column, loading phase, continued

Sample ID	Date	Net counts	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF	Position	^{137}Cs µCi/mL	^{137}Cs µCi
AZ101P-F17	12/6/01	6240	300	1176	2.1884	1.7882	537.27	657.51	2.59E-5	**79.4**	2.59E-3	3.86E+4	1	4.27 E-2	7.63 E-2
AZ101P-F18	12/6/01	5734	300	1075	2.0968	1.7134	512.48	627.17	2.47E-5	**84.2**	2.47E-3	4.04E+4	1	4.07 E-2	6.97 E-2
AZ101P-F19	12/6/01	4870	300	902	2.2089	1.8050	408.24	499.60	1.97E-5	**88.9**	1.97E-3	5.08E+4	1	3.24 E-2	5.85 E-2
AZ101P-F20	12/6/01	2858	300	499	1.951	1.5942	255.95	313.23	1.24E-5	**93.5**	1.24E-3	8.09E+4	1	2.03 E-2	3.24 E-2
AZ101P-F21	12/6/01	5467	300	1021	1.97139	1.6109	517.99	633.92	2.50E-5	**98.3**	2.50E-3	4.00E+4	1	4.11 E-2	6.62 E-2
AZ101P-F22	12/6/01	6394	300	1207	2.6781	2.1883	450.53	551.36	2.17E-5	**103.0**	2.17E-3	4.60E+4	1	3.58 E-2	7.83 E-2
AZ101P-F23	12/6/01	5213	300	970	2.3836	1.9477	407.10	498.21	1.96E-5	**107.6**	1.96E-3	5.09E+4	1	3.23 E-2	6.30 E-2
AZ101P-F24	12/7/01	6137	300	1155	2.7971	2.2856	412.99	505.41	1.99E-5	**112.2**	1.99E-3	5.02E+4	1	3.28 E-2	7.49 E-2
AZ101P-F25	12/7/01	4157	300	759	1.9071	1.5583	398.07	487.16	1.92E-5	**116.9**	1.92E-3	5.20E+4	1	3.16 E-2	4.93 E-2
													sum		**2.76 E+0**

Individual composite effluents

Sample ID	Date	Net counts	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	C/Co	Position	C/Co, %	DF
AZ102FEcomp1	12/6/01	970	300	122	1.9687	2	61.85	60.88	2.99E-6	1	2.99E-4	3.35E+5
AZ102FEcomp2	12/6/01	9570	300	1842	2.316	1.8925	795.23	973.21	3.84E-5	1	3.84E-3	2.61E+4
AZ102FEcomp3	12/6/01	19126	300	3753	2.3591	1.9277	1590.84	1946.88	7.68E-5	1	7.68E-3	1.30E+4
AZ102FEcomp4	12/6/01	19245	300	3777	2.367	1.9341	1595.59	1952.68	7.70E-5	1	7.70E-3	1.30E+4
AZ102FEcomp5	12/6/01	13109	300	2550	2.3548	1.9242	1082.71	1325.02	5.23E-5	1	5.23E-3	1.91E+4
AZ102FEcomp6	12/6/01	9757	300	1879	2.3396	1.9118	803.20	982.95	3.88E-5	1	3.88E-3	2.58E+4
AZ102FEcomp7	12/6/01	8775	300	1683	2.4275	1.9836	693.21	848.35	3.35E-5	1	3.35E-3	2.99E+4
AZ102FEcomp8	12/6/01	8041	300	1536	2.4254	1.9819	633.28	775.01	3.06E-5	1	3.06E-3	3.27E+4
AZ102FEcomp9	12/6/01	7472	300	1422	2.4249	1.9815	586.48	717.74	2.83E-5	1	2.83E-3	3.53E+4
AZ102FEcomp10	12/6/01	6442	300	1216	2.4351	1.9898	499.43	611.20	2.41E-5	1	2.41E-3	4.15E+4
AZ102FEcomp11	12/6/01	6034	300	1135	2.423	1.9799	468.25	573.04	2.26E-5	1	2.26E-3	4.42E+4
AZ102FEcomp12	12/7/01	5847	300	1097	2.4911	2.0355	440.43	539.00	2.13E-5	1	2.13E-3	4.70E+4
AZ102FEcomp13	12/7/01	5475	300	1023	2.4619	2.0117	415.44	508.41	2.01E-5	1	2.01E-3	4.99E+4

Feed displacement (position 1)

Sample ID	Date	Net counts	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	**BV+ Feed**	^{137}Cs µCi
AZ101-FD-1	12/7/01	5158	300	959.36	2.425	2.000	395.61	479.68	1.91E-5	0.3	1.91E-3	**117.2**	9.70 E-2
AZ101-FD-2	12/7/01	5323	300	992.36	2.435	2.000	407.59	496.18	1.97E-5	1.0	1.97E-3	**117.9**	2.30 E-1
AZ101-FD-3	12/7/01	4598	300	847.36	2.429	2.000	348.85	423.68	1.68E-5	1.7	1.68E-3	**118.6**	2.15 E-1
AZ101-FD-4	12/7/01	4548	300	837.36	2.430	2.000	344.59	418.68	1.66E-5	2.6	1.66E-3	**119.5**	2.54 E-1
AZ101-FD-5	12/7/01	4460	300	819.76	2.433	2.000	336.98	409.88	1.63E-5	3.7	1.63E-3	**120.6**	2.93 E-1
AZ101-FD-6	12/7/01	11494	300	2226.56	2.355	2.000	945.46	1113.28	4.56E-5	4.8	4.56E-3	**121.7**	8.77 E-1
AZ101-FD-7	12/7/01	4321	300	791.96	2.056	2.000	385.23	395.98	1.56E-5	6.2	1.56E-3	**123.1**	3.67 E-1
AZ101-FD-8	12/7/01	1983	300	324.36	2.012	2.000	161.19	162.18	6.40E-6	7.3	6.40E-4	**124.2**	1.14 E-1
AZ101-FD-9	12/7/01	1677	300	263.16	1.997	2.000	131.80	131.58	5.19E-6	8.2	5.19E-4	**125.1**	8.49 E-2
												sum	**2.53 E+0**

DI Rinse (count position 1)

Sample ID	Date	Net counts	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	**BV+feed+FD**	^{137}Cs µCi
AZ101-Fdi-1	12/7/01	1636	300	254.96	2.002	2.0020	127.35	127.35	5.02E-6	0.9	5.02E-4	**126.1**	7.91 E-2
AZ101-Fdi-2	12/7/01	1467	300	221.16	1.997	1.9973	110.73	110.73	4.37E-6	1.9	4.37E-4	**127.1**	7.24 E-2
AZ101-Fdi-3	12/7/01	1363	300	200.36	1.992	1.9918	100.59	100.59	3.97E-6	2.9	3.97E-4	**128.1**	6.75 E-2
AZ101-Fdi-4	12/7/01	1293	300	186.36	1.997	1.9972	93.31	93.31	3.68E-6	3.9	3.68E-4	**129.0**	6.00 E-2
AZ101-Fdi-5	12/7/01	1470	300	221.76	1.988	1.9881	111.54	111.54	4.40E-6	4.8	4.40E-4	**130.0**	7.15 E-2
AZ101-Fdi-6	12/7/01	1362	300	200.16	1.991	1.9909	100.54	100.54	3.97E-6	5.8	3.97E-4	**130.9**	6.45 E-2
AZ101-Fdi-7	12/7/01	1171	300	161.96	1.996	1.9964	81.13	81.13	3.20E-6	6.8	3.20E-4	**131.9**	5.32 E-2
AZ101-Fdi-8	12/7/01	1015	300	130.76	1.990	1.9899	65.71	65.71	2.59E-6	7.8	2.59E-4	**132.9**	4.53 E-2
AZ101-Fdi-9	12/7/01	878	300	103.36	1.986	1.9860	52.05	52.05	2.05E-6	8.9	2.05E-4	**134.0**	3.70 E-2
												sum	**5.50 E-1**

AZ-101 run starting 12/3/01.
Lead column, eluting phase

Sample ID	Date	Net counts	Time (sec)	net cpm	counted mass, g	counted volume, mL	net cpm/g	net cpm/mL	C/Co	**BV**	Position	^{137}Cs µCi/mL
AZ101L-E1-d	12/7/01	9771	300	1882	0.0389	3.71E-2	4.84E+4	5.07E+4	2.00E-3	**1.0**	1	3
AZ101L-E2-d	12/7/01	72066	300	14341	0.0394	3.76E-2	3.64E+5	3.81E+5	1.50E-2	**1.9**	1	25
AZ101L-E3-dd	12/7/01	280,783	300	56084	0.0002	1.67E-4	3.22E+8	3.36E+8	1.33E+1	**2.8**	1	21815
AZ101L-E4-dd	12/7/01	64980	300	12924	1.87E-4	1.79E-4	6.89E+7	7.22E+7	7.55E+1	**3.7**	6	124165
AZ101L-E5-dd	12/7/01	9193	300	1766	1.63E-4	1.58E-4	1.08E+7	1.12E+7	1.17E+1	**4.7**	6	19206
AZ101L-E6-dd	12/7/01	31262	300	6180	1.87E-4	1.77E-4	3.30E+7	3.50E+7	1.38E+0	**5.6**	1	2271
AZ101L-E7-dd	12/7/01	2304	300	389	1.84E-4	1.75E-4	2.12E+6	2.22E+6	8.74E-2	**6.6**	1	144
AZ101L-E8-d	12/7/01	105105	300	20949	1.93E-2	1.85E-2	1.09E+6	1.13E+6	4.48E-2	**7.5**	1	74
AZ101L-E9-d	12/7/01	99071	300	19742	3.73E-2	3.57E-2	5.29E+5	5.53E+5	2.18E-2	**8.4**	1	36
AZ101L-E10-d	12/7/01	61804	300	12289	0.0375	3.60E-2	3.28E+5	3.41E+5	1.35E-2	**9.4**	1	22
AZ101L-E11-d	12/7/01	39177	300	7763	0.0375	3.61E-2	2.07E+5	2.15E+5	8.47E-3	**10.4**	1	14
AZ101L-E12-d	12/10/01	26445	300	5217	0.0386	3.64E-2	1.35E+5	1.43E+5	5.66E-3	**11.3**	1	9
AZ101L-E13-d	12/10/01	19367	300	3801	0.0389	3.67E-2	9.77E+4	1.04E+5	4.08E-3	**12.3**	1	7
AZ101L-E14-d	12/10/01	15353	300	2998	0.0379	3.56E-2	7.92E+4	8.41E+4	3.32E-3	**13.3**	1	5
AZ101L-E15-d	12/10/01	25212	300	4970	2.0218	1.9978	2.46E+3	2.49E+3	2.60E-3	**14.3**	6	4

Deionized water rinse following elution

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	BV post Elution	^{137}Cs µCi	Position
AZ101-Edi-1	12/10/01	20148	300	3957	2.0168	2.0168	1962	1962	2.05E-3	1.2	15.4	4.00 E+1	6
AZ101-Edi-3	12/10/01	296570	300	59242	2.0271	2.0271	29225	29225	1.15E-3	2.2	16.5	2.04 E+1	1
AZ101-Edi-4	12/11/01	144283	300	28784	1.9938	1.9938	14437	14437	5.69E-4	3.2	17.5	9.73 E+0	1
AZ101-Edi-5	12/11/01	110047	300	21937	1.9880	1.9880	11035	11035	4.35E-4	4.1	18.4	6.98 E+0	1
AZ101-Fdi-10	12/11/01	118656	300	23659	1.9876	1.9876	11903	11903	4.69E-4	5.1	19.4	7.82 E+0	1
											sum	**8.49 E+1**	

Regeneration with 0.25 M NaOH

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	Position	BV + elution + rinse	^{137}Cs µCi
AZ-101-RGN	12/11/01	21594	300	4247	1.9824	1.9706	2142	2155	2.25E-3	6	20	1.72 E+2

Table C.3. First AZ-101 Cs Ion Exchange Column Test: CMC ^{137}Cs Results

CMC Analysis, ASR 6306								
Sample ID	**RPL ID**	**^{137}Cs, µCi/mL**	**% error**	**C/Co, %**	**BV**	**DF**		
AZ101LF0	02-909	1660	3	100	(not applicable)		2.08E+6	µCi AZ101
AZ101LF0D	02-909 dup	1630	3	100	(not applicable)			fed to column
	average	1645	3					
Load samples								
AZ101L-F5	02-910	0.0108	3	6.57E-04	24.2	1.52E+5		
AZ101L-F11	02-911	0.00761	3	4.63E-04	54.0	2.16E+5		
AZ101L-F17	02-912	0.201	3	1.22E-02	83.2	8.18E+3		
AZ101L-F20	02-913	56.7	3	3.45	98.0	2.90E+1		
AZ101L-F21	02-914	117	3	7.11	102.9	1.41E+1		
AZ101L-F22	02-915	234	3	14.22	107.8	7.03E+0		
AZ101L-F23	02-916	448	3	27.23	112.7	3.67E+0		
AZ101L-F24	02-917	521	3	31.67	117.5	3.16E+0		
AZ101L-F25	02-918	635	3	38.60	122.5	2.59E+0		
AZ101L-F24	re-prep	545	3	33.11	117.5	3.02E+0		
AZ101L-F25	re-prep	718	3	43.67	122.5	2.29E+0		
AZ101P-F5	02-919	0.138	3	8.39E-03	23.3	1.19E+4		
AZ101P-F10	02-920	0.0721	4	4.38E-03	47.2	2.28E+4		
AZ101P-F15	02-921	0.0499	3	3.03E-03	70.1	3.30E+4		
AZ101P-F20	02-922	0.0403	4	2.45E-03	93.5	4.08E+4		
AZ101P-F25	02-923	0.033	4	2.01E-03	116.9	4.98E+4		
AZ101-FEcomp	02-935	0.0649	4	3.95E-03	NA	2.53E+4		
Feed displacement and DI rinse samples								
AZ101FD-3	02-924	0.0285	3	1.73E-03	1.7	5.77E+4		
AZ101FD-8	02-925	0.0103	3	6.26E-04	7.3	1.60E+5		
AZ101Fdi-3	02-926	0.00627	3	3.81E-04	2.9	2.62E+5		
AZ101-Fdi-8	02-927	0.00388	4	2.36E-04	7.8	4.24E+5		
Elution samples	**RPL ID**	**^{137}Cs, µCi/mL**	**error**	**C/Co**	**BV**	**^{137}Cs µCi**		
AZ101L-E3	02-928	3.98E+04	3	24.19	2.8	3.85 E+5		
AZ101L-E4	02-929	1.72E+05	3	104.56	3.7	1.56 E+6		
AZ101L-E5	02-930	2.62E+04	3	15.93	4.7	2.66 E+5		
AZ101L-E6	02-931	3.23E+03	3	1.96	5.6	3.14 E+4		
AZ101L-E11	02-932	14.7	3	8.94E-03	10.4	1.44 E+2		
AZ101L-E15	02-933	3.67	4	2.23E-03	14.3	3.68 E+1		
					Eluate sum	**2.24 E+6**	108	% recovery
Regeneration sample	**RPL ID**	**^{137}Cs, µCi/mL**	**error**	**C/Co**				
AZ101-RGN	02-934	3.99	4	2.43E-03				

DF = decontamination factor

Table C.4 First AZ-101 Cs Ion Exchange Column Test: Cs Lead Column Breakthrough Integration

Activity of ^{137}Cs in Feed = 1645 µCi/mL

Lead column sample ID	Cumulative Processed Vol (mL)	Cs-137 C/C_o	Cs-137 Conc (µCi/mL)	Δ Vol (mL)	C/C_o Midpoint	Midpoint Conc (µCi/mL)	Area (µCi)
AZ101L-F1	50.0	6.02E-06	9.90E-03	50.0	6.02E-06	9.90E-03	4.95E-01
AZ101L-F2	98.1	6.83E-06	1.12E-02	48.2	6.42E-06	1.06E-02	5.09E-01
AZ101L-F3	149.7	6.86E-06	1.13E-02	51.6	6.84E-06	1.13E-02	5.81E-01
AZ101L-F4	200.2	6.28E-06	1.03E-02	50.4	6.57E-06	1.08E-02	5.45E-01
AZ101L-F5	249.4	5.91E-06	9.72E-03	49.2	6.10E-06	1.00E-02	4.94E-01
AZ101L-F6	299.8	5.45E-06	8.97E-03	50.4	5.68E-06	9.34E-03	4.71E-01
AZ101L-F7	350.3	5.44E-06	8.95E-03	50.5	5.45E-06	8.96E-03	4.52E-01
AZ101L-F8	400.4	4.91E-06	8.08E-03	50.1	5.18E-06	8.51E-03	4.26E-01
AZ101L-F9	460.2	4.35E-06	7.15E-03	59.8	4.63E-06	7.61E-03	4.55E-01
AZ101L-F10	508.2	4.59E-06	7.55E-03	48.1	4.47E-06	7.35E-03	3.53E-01
AZ101L-F11	555.8	4.60E-06	7.57E-03	47.6	4.60E-06	7.56E-03	3.60E-01
AZ101L-F12	606.1	4.37E-06	7.19E-03	50.2	4.49E-06	7.38E-03	3.71E-01
AZ101L-F13	656.3	3.84E-06	6.31E-03	50.3	4.11E-06	6.75E-03	3.39E-01
AZ101L-F14	707.0	3.62E-06	5.95E-03	50.6	3.73E-06	6.13E-03	3.10E-01
AZ101L-F15	757.6	4.88E-06	8.02E-03	50.6	4.25E-06	6.99E-03	3.54E-01
AZ101L-F16	807.4	1.64E-05	2.70E-02	49.8	1.07E-05	1.75E-02	8.74E-01
AZ101L-F17	857.4	1.12E-04	1.84E-01	50.0	6.42E-05	1.06E-01	5.28E+00
AZ101L-F18	908.9	8.09E-04	1.33E+00	51.5	4.61E-04	7.58E-01	3.90E+01
AZ101L-F19	959.0	4.29E-03	7.06E+00	50.1	2.55E-03	4.20E+00	2.10E+02
AZ101L-F20	1009.6	2.45E-02	4.02E+01	50.7	1.44E-02	2.37E+01	1.20E+03
AZ101L-F21	1060.0	6.09E-02	1.00E+02	50.4	4.27E-02	7.02E+01	3.54E+03
AZ101L-F22	1110.6	1.61E-01	2.64E+02	50.6	1.11E-01	1.82E+02	9.22E+03
AZ101L-F23	1161.0	2.76E-01	4.54E+02	50.4	2.18E-01	3.59E+02	1.81E+04
AZ101L-F24	1210.0	4.02E-01	6.62E+02	49.0	3.39E-01	5.58E+02	2.73E+04
AZ101L-F25	1261.4	5.31E-01	8.73E+02	51.4	4.67E-01	7.68E+02	3.94E+04
Full load:	**1263.2**	**mL (obtained by mass difference of feed bottle)**					
						(µCi Area sum)	9.91E+04µCi

Amount of Cs that Broke through Lead Column = Amount of Cs on Lag Column = 99,099µCi
= 0.0991Ci

Amount of Cs Fed to Lead Column (1645 µCi/mL * 1263.2 mL) = 2.08E+06µCi
= 2.078Ci

Percentage of Cs-137 on Lag Column (0.0991 Ci / 2.078 Ci) *100 = 4.8%

Amount of Cs Loaded on Lead Column (2.078 Ci – 0.0991 Ci) = 1.979Ci

Table C.5. First AZ-101 Cs Ion Exchange Column Test: Cs Isotopic and Mass Distributions

AZ-101 Cs isotopic distribution
ASR 6403 #02-2246 (Eluate)
Thermal ionization mass spectrometry weight fraction distribution ratio

	Cs-133	**Cs-135**	**Cs-137**
wt fraction	0.5185	0.169	0.3125

The eluate isotopic distribution is considered most accurate and is applied to the eluate, effluent, feed and regeneration solutions. Total Cs was calculated from 137Cs and the isotopic ratio.

ASR 6306 RPL # 02-0909 Feed 1263 mL

Cs IX feed	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	7.51	2194	3.42 E-3	9.49 E-3	
Cs-137	1645	87	18.91	2.08 E+0	2.39 E+1

Feed Cs Concentration

	Cs-133	Cs-135	Cs-137	Sum Conc.	total mass (mg) or millimoles
µg/mL Cs	31.372	10.225	18.908	60.506	7.64E+1
M Cs	2.36E-4	7.57E-5	1.38E-4	4.50E-4	5.68E-1

31.25 weight % of the Cs is Cs-137
Therefore, the total Cs concentration is 18.91 µg/mL divided by 0.3125
60.51 µg/mL Cs in the AZ-102 feed

ASR 6280 RPL # 02-0769 AZ-102C Cs eluate (reported in WTP-RPT-041)

Thermal ionization mass spectrometry wt fraction distribution ratio

	Cs-133	**Cs-135**	**Cs-137**
wt fraction	0.523	0.149	0.328
	SpA, µCi/µg	total Ci	total mg
Cs-137	87.0	0.12	1.38

	Cs-133	Cs-135	Cs-137	Sum, mg or mmoles
mg Cs	2.195	0.626	1.379	4.200
M Cs	1.65E-5	4.64E-6	1.01E-5	3.12E-5

32.8 weight % of the Cs is Cs-137
Therefore, the total Cs concentration is 1.38 µg divided by 0.328
4.20 mg Cs from AZ-102C loaded already on the lead column

ASR 6306 RPL # 02-935 (AZ-101 effluent) 1152.5 mL

Cs IX effluent	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	<5.0E-5	2194	<2.E-8	5.76 E-8	
Cs-137	6.49E-02	87	7.46 E-4	7.48 E-5	8.60 E-4

1st run effluent Cs Concentration

	Cs-133	Cs-135	Cs-137	Sum conc.	total mass (mg) or millimoles
µg/mL Cs	1.24 E-3	4.03 E-4	7.46 E-4	2.39 E-3	2.75E-3
M Cs	9.31E-9	2.99E-9	5.45E-9	1.77E-8	2.04E-5

31.25 weight % of the Cs is Cs-137
Therefore, the total Cs concentration is 7.46E-4 µg/mL divided by 0.3125
2.39 E-3 µg/mL Cs in the AZ-102 feed

Net lead column Cs loading

	mg Cs	**Ci Cs-137**
Feed	76.4	2.08
subtract breakthrough	1.14	0.099
add AZ-102C initial load	4.20	0.12
Net	79.5	2.10

ASR 6401 #02-2238 (AZ-101 eluate 1st run) 146.84 mL

Cs IX effluent	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	5.78 E+0	2194	2.63 E-3	8.49 E-4	
Cs-137	1.60E+04	87	1.84 E+2	2.35 E+0	27.0

1st run eluate Cs Concentration

	Cs-133	Cs-135	Cs-137	Sum conc.	total mass (mg) or millimoles
µg/mL Cs	3.05 E+2	9.95 E+1	1.84 E+2	5.89 E+2	8.64E+1
M Cs	2.29E-3	7.37E-4	1.34E-3	4.37E-3	6.42E-1

31.25 weight % of the Cs is Cs-137
Therefore, the total Cs concentration is 1.84E+2 µg/mL divided by 0.3125
5.89 E+2 µg/mL Cs in the AZ-102 feed

ASR 6306 RPL # 02-934 Regeneration solution 46.3 mL

AZ-101-RGN	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	<2.0E-3	2194	<9.E-7	9.26 E-8	
Cs-137	3.99	87	0.0459	1.85 E-4	2.12 E-3

Regeneration Solution Cs Concentration

	Cs-133	Cs-135	Cs-137	Sum conc.	total mass (mg) or millimoles
µg/mL Cs	7.61 E-2	2.48 E-2	4.59 E-2	0.147	6.79E-3
M Cs	5.72E-7	1.84E-7	3.35E-7	1.09E-6	5.05E-5

31.25 weight % of the Cs is Cs-137
Therefore, the total Cs concentration is 0.0459 µg/mL divided by 0.3125
0.147 µg/mL Cs in the AZ-102 feed

SpA = specific activity

Second AZ-101 Cs IX Processing

Table C.6. Second Cs Ion Exchange Column Test: Sample Process Flows

			Solution	Density	
SL-644 batch 010310SMC-IV-73 212-425 um particle size			HNO3	1.012	g/mL
Bed volume in AZ-101 Rerun, lead column =	10.2	mL	0.1M NaOH	0.999	g/mL
Bed volume in 0.25M NaOH, lead column =	10.9	mL	0.25M NaOH	1.006	g/mL
Apparatus volume, whole system =	42	mL	AZ-101	1.2238	g/mL
Apparatus volume, lead column =	23	mL	DI water	0.997	g/mL

Notes:
start = start time of flow through column
end = end time of flow through column
Δt = time interval
BV = bed volume
cumul. Vol = cumulative volume collected
BV (RGN) = bed volumes relative to regeneration condition
BV (feed) = bed volumes relative to feed condition of columns

Conditioning 12/3/01 Lead column + Lag column start	end	Δt	mass, g	volume, mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	volume, AV
0.25M NaOH wash								
1/28/2002 9:54	1/28/2002 12:50	2:56	80.9	80.4	0.46	2.52	7.4	1.91

AZ-101 actual waste loading starting on 1/28/02

Initial effluent collection 1 apparatus volume of feed start	end	Δt	mass, g	cumul. vol., mL	flowrate, mL/min	flowrate, BV/hr	volume, AV	Lead column sampling Sample ID	mass, g	volume, mL	cum. total vol., mL	BV (RGN)	BV (feed)	Running total sample vol
1/28/2002 13:00	1/28/2002 14:40	1:40	46.8	46.5	0.465	2.56	1.1	RZ101L-F1	2.7045	2.210	48.731	**4.5**	**4.8**	2.2099

Feed flow start	end	Δt	mass, g	cumul. vol., mL	flowrate, mL/min	flowrate, BV/hr	cum. vol., BV	Lead column sampling Sample ID	mass g	volume, mL	cum. total vol., mL	BV (RGN)	BV (feed)	Running total sample vol
1/28/2002 14:57	1/28/2002 16:20	1:23	45.4	83.6	0.447	2.46	7.7	RZ101L-F2	3.3003	2.697	91.400	**8.4**	**9.0**	4.9067
16:33	18:00	1:27	50.9	125.2	0.478	2.63	11.5	RZ101L-F3	2.8293	2.312	138.722	**12.7**	**13.6**	7.2186
18:12	19:40	1:28	51.3	167.1	0.476	2.62	15.3	RZ101L-F4	3.0364	2.481	185.890	**17.1**	**18.2**	9.6997
19:52	21:20	1:28	52.6	210.1	0.488	2.69	19.3	RZ101L-F5	2.5387	2.074	233.312	**21.4**	**22.9**	11.7741
21:30	23:00	1:30	54.3	254.5	0.493	2.71	23.3	RZ101L-F6	2.8014	2.289	282.207	**25.9**	**27.7**	14.0632
1/28/2002 23:13	1/29/2002 0:40	1:27	53.2	298.0	0.500	2.75	27.3	RZ101L-F7	2.9911	2.444	330.982	**30.4**	**32.4**	16.5074
0:51	2:20	1:29	48.4	337.5	0.444	2.45	31.0	RZ101L-F8	2.5352	2.072	375.081	**34.4**	**36.8**	18.5789
2:31	4:00	1:29	52.9	380.7	0.486	2.67	34.9	RZ101L-F9	2.4497	2.002	422.863	**38.8**	**41.5**	20.5807
4:10	5:40	1:30	54.0	424.9	0.490	2.70	39.0	RZ101L-F10	2.4312	1.987	471.365	**43.2**	**46.2**	22.5672
5:49	7:20	1:31	54.4	469.3	0.488	2.69	43.1	RZ101L-F11	2.3024	1.881	519.733	**47.7**	**51.0**	24.4486
7:29	9:00	1:31	51.3	511.2	0.461	2.54	46.9	RZ101L-F12	2.4427	1.996	565.934	**51.9**	**55.5**	26.4446
9:09	10:40	1:31	51.6	553.4	0.463	2.55	50.8	RZ101L-F13	2.4439	1.997	611.858	**56.1**	**60.0**	28.4416
10:49	11:59	1:10	44.1	589.4	0.515	2.83	54.1	RZ101L-F14	3.0713	2.510	652.659	**59.9**	**64.0**	30.9512
12:10	14:00	1:50	64.6	642.2	0.480	2.64	58.9	RZ101L-F15	2.6185	2.140	709.994	**65.1**	**69.6**	33.0909
14:11	15:20	1:09	40.8	675.5	0.483	2.66	62.0	RZ101L-F16	2.9360	2.399	748.040	**68.6**	**73.3**	35.4899
15:31	17:22	1:51	65.8	729.3	0.484	2.67	66.9	RZ101L-F17	3.8898	3.178	807.565	**74.1**	**79.2**	38.6684
17:38	19:00	1:22	49.5	769.8	0.493	2.72	70.6	RZ101L-F18	3.1311	2.559	853.243	**78.3**	**83.7**	41.2269
19:15	20:40	1:25	55.7	815.3	0.535	2.95	74.8	RZ101L-F19	2.2406	1.831	903.300	**82.9**	**88.6**	43.0578
20:50	22:20	1:30	53.1	858.7	0.482	2.65	78.8	RZ101L-F20	2.2033	1.800	950.756	**87.2**	**93.2**	44.8581
1/29/2002 22:30	1/30/2002 0:01	1:31	52.8	901.8	0.474	2.61	82.7	RZ101L-F21	2.3330	1.906	998.068	**91.6**	**97.8**	46.7645
1/30/2002 0:10	1/30/2002 1:40	1:30	53.0	945.1	0.481	2.65	86.7	RZ101L-F22	2.5000	2.043	1045.640	**95.9**	**102.5**	48.8073
Δt, total:	36:40:00			**Average:**	**0.48**	**2.66**								

Feed total:	1048.09	mL	through **lead** column
Feed total:	1288.0	g	
	1052.5	mL	
	4.4	mL loss	
Feed total:	999.3	mL	through the **lag** column

Lag column sampling

Date	Sample ID	mass, g	volume, mL	total vol., mL	BV (RGN)	BV (feed)	Running total sample vol., mL
1/28/02	RZ-101P-F1	3.5185	2.875	49.40	**4.5**	**4.8**	2.875
1/28/02	RZ-101P-F2	3.4386	3.418	89.91	**8.2**	**8.8**	6.293
1/28/02	RZ-101P-F3	3.3875	2.768	134.27	**12.3**	**13.2**	9.061
1/28/02	RZ-101P-F4	2.8965	2.367	178.56	**16.4**	**17.5**	11.428
1/28/02	RZ-101P-F5	2.7371	2.237	223.77	**20.5**	**21.9**	13.665
1/28/02	RZ-101P-F6	3.4991	2.859	271.00	**24.9**	**26.6**	16.524
1/29/02	RZ-101P-F7	3.0332	2.479	316.95	**29.1**	**31.1**	19.002
1/29/02	RZ-101P-F8	3.1261	2.554	359.06	**32.9**	**35.2**	21.557
1/29/02	RZ-101P-F9	2.9258	2.391	404.67	**37.1**	**39.7**	23.947
1/29/02	RZ-101P-F10	2.4894	2.034	450.83	**41.4**	**44.2**	25.982
1/29/02	RZ-101P-F11	2.7982	2.286	497.57	**45.6**	**48.8**	28.268
1/29/02	RZ-101P-F12	2.1587	1.764	541.25	**49.7**	**53.1**	30.032
1/29/02	RZ-101P-F13	2.7603	2.256	585.67	**53.7**	**57.4**	32.288
1/29/02	RZ-101P-F14	2.9481	2.409	624.12	**57.3**	**61.2**	34.696
1/29/02	RZ-101P-F15	2.8254	2.309	679.21	**62.3**	**66.6**	37.005
1/29/02	RZ-101P-F16	3.1564	2.579	715.13	**65.6**	**70.1**	39.584
1/29/02	RZ-101P-F17	3.2697	2.672	771.57	**70.8**	**75.6**	42.256
1/29/02	RZ-101P-F18	3.3191	2.712	814.73	**74.7**	**79.9**	44.968
1/29/02	RZ-101P-F19	2.7729	2.266	862.51	**79.1**	**84.6**	47.234
1/29/02	RZ-101P-F20	2.7681	2.262	908.16	**83.3**	**89.0**	49.496
1/30/02	RZ-101P-F21	2.7187	2.222	953.53	**87.5**	**93.5**	51.717
1/30/02	RZ-101P-F22	2.9942	2.447	999.28	**91.7**	**98.0**	54.164

0.1M NaOH feed displacement 1/30/2002

Sample ID	Start	End	Δt	Sample mass, g	Sample vol., mL	flowrate mL/min	flowrate, BV/hr	flowrate, BV/hr	cumul. BV	volume, AV	Cumul. AV	calculated density, g/mL
RZ101-FD-1	1:59	2:19	0:20	14.89	12.16	0.61	3.35	1.12	1.12	0.29	0.29	1.224
RZ101-FD-2	2:23	2:43	0:20	10.80	8.82	0.44	2.43	0.81	1.93	0.21	0.50	1.220
RZ101-FD-3	2:44	3:04	0:20	12.59	10.29	0.51	2.83	0.94	2.87	0.24	0.74	1.226
RZ101-FD-4	3:08	3:28	0:20	13.96	11.41	0.57	3.14	1.05	3.92	0.27	1.02	1.223
RZ101-FD-5	3:32	3:52	0:20	13.56	11.56	0.58	3.18	1.06	4.98	0.28	1.29	1.172
RZ101-FD-6	3:57	4:17	0:20	12.49	12.00	0.60	3.30	1.10	6.08	0.29	1.58	1.041
RZ101-FD-7	4:21	4:41	0:20	11.03	10.89	0.54	3.00	1.00	7.08	0.26	1.84	1.013
RZ101-FD-8	4:44	5:04	0:20	10.45	10.24	0.51	2.82	0.94	8.02	0.24	2.08	1.020
RZ101-FD-9	5:07	5:27	0:20	11.20	11.19	0.56	3.08	1.03	9.04	0.27	2.35	1.001
RZ101-FD-10	5:31	5:51	0:20	11.34	10.95	0.55	3.01	1.00	10.05	0.26	2.61	1.036
	Δt, total:	3:28			**Average**	**0.55**	**3.01**					
				Sum:	**109.52**							

DI water rinse	1/30/2002										
Sample ID	Start	End	Δt	Sample mass, g	Sample Vol., mL	flowrate, mL/min	flowrate, BV/hr	flowrate, BV/hr	cumul. BV	volume, AV	Cumul. AV
RZ101-Fdi-1	5:55	6:15	0:20	11.320	11.354	0.57	3.12	1.04	1.04	0.27	0.27
RZ101-Fdi-2	6:16	6:36	0:20	9.693	9.722	0.49	2.68	0.89	1.93	0.23	0.50
RZ101-Fdi-3	6:37	6:57	0:20	9.792	9.821	0.49	2.70	0.90	2.83	0.23	0.74
RZ101-Fdi-4	6:57	7:18	0:21	9.833	9.863	0.47	2.59	0.90	3.74	0.23	0.97
RZ101-Fdi-5	7:18	7:39	0:21	8.523	8.548	0.41	2.24	0.78	4.52	0.20	1.17
RZ101-Fdi-6	7:39	7:59	0:20	9.792	9.822	0.49	2.70	0.90	5.42	0.23	1.41
RZ101-Fdi-7	7:59	8:20	0:21	9.965	9.995	0.48	2.62	0.92	6.34	0.24	1.65
RZ101-Fdi-8	8:20	8:40	0:20	9.590	9.619	0.48	2.65	0.88	7.22	0.23	1.87
RZ101-Fdi-9	8:40	9:00	0:20	9.411	9.439	0.47	2.60	0.87	8.09	0.22	2.10
RZ101-Fdi-10	9:00	9:21	0:21	9.777	9.807	0.47	2.57	0.90	8.99	0.23	2.33
	Δt, total:	3:26			**Average**	**0.48**	**2.65**				
				Sum:	**98.0**						

Cs elution from lead column, Separate columns 1/30/02	Sample ID	start	end	Δt	mass collected, g	volume, mL	flowrate, mL/min	flowrate, BV/hr	cumul. BV
		HNO3 wash--lead column							
	RZ101L-E1	9:30	10:00	0:30	8.5995	8.50	0.28	1.56	0.78
	RZ101L-E2	10:01	10:32	0:31	10.0518	9.93	0.32	1.76	1.69
	RZ101L-E3	10:32	10:59	0:27	9.0996	8.99	0.33	1.83	2.52
	RZ101L-E4	11:00	11:30	0:30	9.8562	9.74	0.32	1.79	3.41
	RZ101L-E5	11:31	12:01	0:30	10.2762	10.15	0.34	1.86	4.34
	RZ101L-E6	12:02	12:30	0:28	9.9426	9.82	0.35	1.93	5.24
	RZ101L-E7	12:32	13:00	0:28	9.7265	9.61	0.34	1.89	6.12
	RZ101L-E8	13:01	13:30	0:29	9.7960	9.68	0.33	1.84	7.01
	RZ101L-E9	13:31	14:01	0:30	10.0866	9.97	0.33	1.83	7.93
	RZ101L-E10	14:02	14:33	0:31	10.5531	10.43	0.34	1.85	8.88
	RZ101L-E11	14:34	14:58	0:24	7.9945	7.90	0.33	1.81	9.61
	RZ101L-E12	14:59	15:30	0:31	10.5598	10.43	0.34	1.85	10.57
	RZ101L-E13	15:31	15:59	0:28	9.4878	9.38	0.33	1.84	11.43
	RZ101L-E14	16:00	16:30	0:30	10.2614	10.14	0.34	1.86	12.36
	RZ101L-E15	16:31	16:58	0:27	9.2268	9.12	0.34	1.86	13.19
	RZ101L-E16	16:59	17:29	0:30	9.9960	9.88	0.33	1.81	14.10
	RZ101L-E17	17:31	17:58	0:27	9.4442	9.33	0.35	1.90	14.95
	RZ101L-E18	17:59	18:29	0:30	10.0191	9.90	0.33	1.82	15.86
	Δt, total:	7:28:00		**Sum:**	**174.98**	**172.90**			
						Average:	**0.33**	**1.83**	

DI water rinse start 1/30/02

Sample ID	Start	End	Δt	Sample mass, g	Sample Vol., mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	Cum. vol., BV	volume, AV	Cumul. vol., AV
RZ101-Edi-1	18:35	18:54	0:19	9.908	9.938	0.52	2.88	0.9	0.9	0.43	0.43
RZ101-Edi-2	18:55	19:15	0:20	10.408	10.439	0.52	2.87	1.0	1.9	0.45	0.89
RZ101-Edi-3	19:16	19:36	0:20	10.145	10.175	0.51	2.80	0.9	2.8	0.44	1.33
RZ101-Edi-4	19:37	19:57	0:20	10.122	10.152	0.51	2.79	0.9	3.7	0.44	1.77
RZ101-Edi-5	19:58	20:18	0:20	8.936	8.963	0.45	2.47	0.8	4.6	0.39	2.16
RZ101-Edi-6	20:19	20:39	0:20	10.557	10.589	0.53	2.91	1.0	5.5	0.46	2.62
RZ101-Edi-7	20:40	21:00	0:20	9.966	9.996	0.50	2.75	0.9	6.4	0.43	3.05
		Δt, total:	1:43		**Average:**	**0.51**	**2.78**				
				Sum:	**70.25**						

Regeneration with 0.25M NaOH start 2/1/02

Sample ID	Start	End	Δt	Sample mass, g	Sample Vol., mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	Cum. vol., BV	volume, AV	Cumul. vol., AV
RZ101L-RGN	6:34	7:34	1:00	13.3	13.2	0.22	1.21	1.2	1.2	0.57	0.57
	7:36	9:27	1:51	15.9	15.8	0.14	0.78	1.5	2.7	0.69	1.26
	9:29	10:47	1:18	11.1	11.0	0.14	0.78	1.0	3.7	0.48	1.74
	10:47	10:56	0:09	7.9	7.9	0.87	4.80	0.7	4.4	0.34	2.08
	Δt, total:	4:22		**Sum:**	**47.9**						
					Average:	**0.17**	**0.93**				

This incorporated a draining of the fluid above the bed. Apparently a slight blockage was removed allowing the solution to flow freely resulting in a high flowrate.

Rinse with DI water start 2/1/02

Sample ID	Start	End	Δt	Sample mass, g	Sample Vol., mL	flowrate, mL/min	flowrate, BV/hr	volume, BV	Cum. vol., BV	volume, AV	Cumul. vol., AV
RZ101-DIRinse-Final	11:03	11:53	0:50	16.0	16.0	0.32	1.77	1.5	1.5	0.70	0.70
	11:54	12:24	0:30	13.3	13.3	0.44	2.45	1.2	2.7	0.58	1.28
	12:25	12:54	0:29	13.0	13.0	0.45	2.47	1.2	3.9	0.57	1.84
	12:55	13:16	0:21	8.7	8.7	0.42	2.29	0.8	4.7	0.38	2.22
	Δt, total:	1:51		**Sum:**	**51.2**						
					Average:	**0.41**	**2.24**				

Table C.7. Second Cs Ion Exchange Column Test: Counting Data

Counting Data for TI-RPP-WTP-152, Rev. 0

Background Counts

Date	Net counts	Uncertainty	Time (sec)	cpm	uncertainty %
1/29/02 10:00	1535	47	1000	92.10	3%
1/29/02 16:58	1437	48	1000	86.22	3%
1/29/02 21:42	4275	86	3000	85.50	2%
1/31/02 11:40	4427	86	3000	88.54	2%
1/31/02 16:39	4293	86	3000	85.86	2%
			average	**87.64**	

Notes: AZ-101 density = **1.2238** g/mL
cpm = counts per minute
DF = decontamination factor
position = indicates relative position sample is relative to detector

AZ101 Comparitor Standards

F0DD-A: 0.1140 g of AZ-101 into 10.0671g (total), then 0.1003g of this into 10.0489g (total), then 2 mL geometry (1.9310 g, diluent is 0.01M NaOH).
2.17E-04 g RZ-102

Date	Net counts	Error	(sec)	cpm	net cpm	mass, g	net cpm/g	mL	cpm/mL
F0DD-A, Position 1 on counter									
1/29/02 11:27	22367	169	300	4473	4386	2.17E-4	2.02E+7	1.77E-4	2.47E+7
1/29/02 15:03	21633	167	300	4327	4239	2.17E-4	1.95E+7	1.77E-4	2.39E+7
1/29/02 21:32	22752	172	300	4550	4463	2.17E-4	2.05E+7	1.77E-4	2.51E+7
1/30/02 14:19	23055	174	300	4611	4523	2.17E-4	2.08E+7	1.77E-4	2.55E+7
1/30/02 19:47	22719	172	300	4544	4456	2.17E-4	2.05E+7	1.77E-4	2.51E+7
1/31/02 14:50	22670	172	300	4534	4446	2.17E-4	2.05E+7	1.77E-4	2.51E+7
1/31/02 16:32	21771	172	300	4354	4267	2.17E-4	1.96E+7	1.77E-4	2.40E+7
2/1/02 8:52	23381	171	300	4676	4589	2.17E-4	2.11E+7	1.77E-4	2.59E+7
						Average	**2.04E+7**		**2.49E+7**
						standard deviation	**5.51E+5**		**6.75E+5**
						% standard deviation	**2.7**		**2.7**

F0D-A 0.1140 g of AZ-101 brought to 10.10671g total, then 2 g of this was placed into a 2-mL geometry (diluent is 0.01M NaOH).
0.0213956 g AZ-101

Date	Net counts	Error	(sec)	cpm	net cpm	mass, g	net cpm/g	mL	cpm/mL	
F0D-A, Position 6 on counter										
1/30/2002	86059	344	300	17212	17124	0.0214	8.00E+5	0.0175	9.79E+5	position 6
						Average	**8.00E+5**		**9.79E+5**	

AZ-101 run starting 1/28/02
Lead column, loading phase

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	BV	% C/Co	DF	Position	Cs-137 µCi	Cs, µCi/mL
RZ101L-F1	1/29/02	11264	300	2165	2.4449	1.9978	886	1084	4.35E-5	**4.5**	4.35E-3	2.30E+4	1	1.58E-1	0.07
RZ101L-F2	1/29/02	9422	300	1797	2.808	2.2945	640	783	3.14E-5	**8.4**	3.14E-3	3.18E+4	1	1.39E-1	0.05
RZ101L-F3	1/29/02	7460	300	1404	2.4267	1.9829	579	708	2.84E-5	**12.7**	2.84E-3	3.52E+4	1	1.08E-1	0.05
RZ101L-F4	1/29/02	7136	300	1340	2.6876	2.1961	498	610	2.45E-5	**17.1**	2.45E-3	4.08E+4	1	9.99E-2	0.04
RZ101L-F5	1/29/02	5979	300	1108	2.1161	1.7291	524	641	2.57E-5	**21.4**	2.57E-3	3.89E+4	1	8.78E-2	0.04
RZ101L-F6	1/29/02	6083	300	1129	2.4067	1.9666	469	574	2.30E-5	**25.9**	2.30E-3	4.34E+4	1	8.68E-2	0.04
RZ101L-F7	1/29/02	6134	300	1139	2.4817	2.0279	459	562	2.25E-5	**30.4**	2.25E-3	4.43E+4	1	9.07E-2	0.04
RZ101L-F8	1/29/02	5265	300	965	2.2334	1.8250	432	529	2.12E-5	**34.4**	2.12E-3	4.71E+4	1	7.24E-2	0.03
RZ101L-F9	1/29/02	4790	300	870	2.146	1.7536	406	496	1.99E-5	**38.8**	1.99E-3	5.02E+4	1	6.56E-2	0.03
RZ101L-F10	1/29/02	4474	300	807	1.9926	1.6282	405	496	1.99E-5	**43.2**	1.99E-3	5.03E+4	1	6.50E-2	0.03
RZ101L-F11	1/29/02	4360	300	784	2.1369	1.7461	367	449	1.80E-5	**47.7**	1.80E-3	5.55E+4	1	5.58E-2	0.03
RZ101L-F12	1/29/02	4476	300	808	2.2248	1.8179	363	444	1.78E-5	**51.9**	1.78E-3	5.61E+4	1	5.85E-2	0.03
RZ101L-F13	1/29/02	4612	300	835	2.3093	1.8870	361	442	1.78E-5	**56.1**	1.78E-3	5.63E+4	1	5.83E-2	0.03
RZ101L-F14	1/29/02	5727	300	1058	2.9435	2.4052	359	440	1.77E-5	**59.9**	1.77E-3	5.66E+4	1	7.29E-2	0.03
RZ101L-F15	1/29/02	5412	300	995	2.5218	2.0606	394	483	1.94E-5	**65.1**	1.94E-3	5.16E+4	1	6.82E-2	0.03
RZ101L-F16	1/29/02	7410	300	1394	2.7844	2.2752	501	613	2.46E-5	**68.6**	2.46E-3	4.07E+4	1	9.71E-2	0.04
RZ101L-F17	1/29/02	34252	300	6763	3.7716	3.0819	1793	2194	8.81E-5	**74.1**	8.81E-3	11353	1	4.61E-1	0.14
RZ101L-F18	1/29/02	109188	300	21750	2.8990	2.3689	7503	9182	3.69E-4	**78.3**	3.69E-2	2713	1	1.55E+0	0.61
RZ101L-F19	1/30/02	313041	300	62521	1.8296	1.4950	34172	41819	1.68E-3	**82.9**	1.68E-1	596	1	5.06E+0	2.76
RZ101L-F20	1/30/02	16032	300	3119	0.0216	0.0177	1.44E+5	1.77E+5	7.09E-3	**87.2**	7.09E-1	141	1	2.10E+1	11.66
RZ101L-F21	1/30/02	53019	300	10516	0.0222	0.0181	4.74E+5	5.81E+5	2.33E-2	**91.6**	2.33E+0	43	1	7.31E+1	38.34
RZ101L-F22	1/30/02	143173	300	28547	0.0213	0.0174	1.34E+6	1.64E+6	6.59E-2	**95.9**	6.59E+0	15	1	2.21E+2	108.39
													sum:	**323.96**	

AZ-101 run starting 1/28/02
Lag column, loading phase

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF	Position	Cs-137 µCi	Cs, µCi/mL
RZ101P-F1	1/29/02	81053	300	16123	3.1494	2.5735	5119	6265	2.51E-4	**4.5**	2.51E-2	3.98E+3	1	1.19E+0	0.41
RZ101P-F2	1/29/02	168654	300	33643	3.0314	2.4770	11098	13582	5.45E-4	**8.2**	5.45E-2	1.83E+3	1	3.07E+0	0.90
RZ101P-F3	1/29/02	215197	300	42952	2.9892	2.4426	14369	17585	7.06E-4	**12.3**	7.06E-2	1.42E+3	1	3.21E+0	1.16
RZ101P-F4	1/29/02	160528	300	32018	2.3583	1.9270	13577	16615	6.67E-4	**16.4**	6.67E-2	1.50E+3	1	2.60E+0	1.10
RZ101P-F5	1/29/02	145387	300	28990	2.4028	1.9634	12065	14765	5.93E-4	**20.5**	5.93E-2	1.69E+3	1	2.18E+0	0.97
RZ101P-F6	1/29/02	183142	300	36541	3.1233	2.5521	11699	14318	5.75E-4	**24.9**	5.75E-2	1.74E+3	1	2.70E+0	0.95
RZ101P-F7	1/29/02	130569	300	26026	2.493	2.0371	10440	12776	5.13E-4	**29.1**	5.13E-2	1.95E+3	1	2.09E+0	0.84

Lag column, loading phase, continued

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF	Position	Cs-137 µCi	Cs, µCi/mL
RZ101P-F8	1/29/02	122953	300	24503	2.7365	2.2361	8954.1	10958.06	4.40E-4	**32.9**	4.40E-2	2.27E+3	1	1.85E+0	0.72
RZ101P-F9	1/29/02	103776	300	20668	2.4103	1.9695	8574.68	10493.70	4.21E-4	**37.1**	4.21E-2	2.37E+3	1	1.66E+0	0.69
RZ101P-F10	1/29/02	86051	300	17123	2.123	1.7348	8065.26	9870.27	3.96E-4	**41.4**	3.96E-2	2.52E+3	1	1.33E+0	0.65
RZ101P-F11	1/29/02	92840	300	18480	2.4086	1.9681	7672.65	9389.79	3.77E-4	**45.6**	3.77E-2	2.65E+3	1	1.42E+0	0.62
RZ101P-F12	1/29/02	97564	300	19425	2.0083	1.6410	9672.44	11837.13	4.75E-4	**49.7**	4.75E-2	2.10E+3	1	1.38E+0	0.78
RZ101P-F13	1/29/02	113393	300	22591	2.629	2.1482	8592.98	10516.09	4.22E-4	**53.7**	4.22E-2	2.37E+3	1	1.57E+0	0.69
RZ101P-F14	1/29/02	106218	300	21156	2.8355	2.3170	7461.10	9130.90	3.67E-4	**57.3**	3.67E-2	2.73E+3	1	1.45E+0	0.60
RZ101P-F15	1/29/02	97078	300	19328	2.6991	2.2055	7160.89	8763.50	3.52E-4	**62.3**	3.52E-2	2.84E+3	1	1.34E+0	0.58
RZ101P-F16	1/29/02	97097	300	19332	3.0043	2.4549	6434.70	7874.78	3.16E-4	**65.6**	3.16E-2	3.16E+3	1	1.34E+0	0.52
RZ101P-F17	1/29/02	89127	300	17738	3.1351	2.5618	5657.80	6924.01	2.78E-4	**70.8**	2.78E-2	3.60E+3	1	1.22E+0	0.46
RZ101P-F18	1/29/02	87638	300	17440	3.1832	2.6011	5478.75	6704.89	2.69E-4	**74.7**	2.69E-2	3.72E+3	1	1.20E+0	0.44
RZ101P-F19	1/30/02	59758	300	11864	2.5345	2.0710	4680.98	5728.59	2.30E-4	**79.1**	2.30E-2	4.35E+3	1	8.57E-1	0.38
RZ101P-F20	1/30/02	50250	300	9962	2.219	1.8132	4489.57	5494.34	2.21E-4	**83.3**	2.21E-2	4.53E+3	1	8.21E-1	0.36
RZ101P-F21	1/30/02	49204	300	9753	2.3518	1.9217	4147.10	5075.22	2.04E-4	**87.5**	2.04E-2	4.91E+3	1	7.44E-1	0.34
RZ101P-F22	1/30/02	51943	300	10301	2.6068	2.1301	3951.57	4835.93	1.94E-4	**91.7**	1.94E-2	5.15E+3	1	7.81E-1	0.32
													sum	**6.84E+2**	

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF
RZ102FEcomp1	1/31/02	11634	300	2239	1.9773	2	1132.43	1119.58	5.56E-5	NA	5.56E-3	1.80E+4
RZ102FEcomp2	1/31/02	109626	300	21838	2.3884	1.9516	9143.17	11189.42	4.49E-4	NA	4.49E-2	2.23E+3
RZ102FEcomp3	1/31/02	54109	300	10734	2.3432	1.9147	4580.98	5606.21	2.25E-4	NA	2.25E-2	4.44E+3

Feed Displacement

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	**BV**	C/Co, %	BV + feed	Cs-137 µCi
RZ101-FD-1	1/31/02	51420	300	10196	2.354	1.923	4332	5302	2.13E-4	**1.1**	2.13E-2	92.8	4.26E+0
RZ101-FD-2	1/31/02	38842	300	7681	2.008	1.646	3825	4667	1.88E-4	**1.9**	1.88E-2	93.6	2.73E+0
RZ101-FD-3	1/31/02	46207	300	9154	2.358	1.923	3882	4760	1.91E-4	**2.9**	1.91E-2	94.5	3.23E+0
RZ101-FD-4	1/31/02	50338	300	9980	2.353	1.923	4242	5190	2.08E-4	**3.9**	2.08E-2	95.6	3.91E+0
RZ101-FD-5	1/31/02	56396	300	11192	2.254	1.923	4965	5820	2.44E-4	**5.0**	2.44E-2	96.7	4.64E+0
RZ101-FD-6	1/31/02	15304	300	2973	2.003	1.923	1485	1546	6.21E-5	**6.1**	6.21E-3	97.8	1.22E+0
RZ101-FD-7	1/31/02	6438	300	1200	1.948	1.923	616	624	2.50E-5	**7.1**	2.50E-3	98.8	4.49E-1
RZ101-FD-8	1/31/02	4712	300	855	1.962	1.923	436	444	1.78E-5	**8.0**	1.78E-3	99.7	3.01E-1
RZ101-FD-9	1/31/02	3953	300	703	1.925	1.923	365	366	1.47E-5	**9.0**	1.47E-3	100.7	2.70E-1
RZ101-FD-10	1/31/02	3741	300	661	1.992	1.923	332	344	1.38E-5	**10.0**	1.38E-3	101.7	2.48E-1
												sum	**2.13E+1**

DI Rinse

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	**BV**	C/Co, %	BV + feed+ FD	Cs-137 µCi
RZ101-Fdi-1	2/1/02	3881	300	689	1.938	1.9380	355	355	1.43E-5	**1.0**	1.43E-3	102.8	2.66E-1
RZ101-Fdi-2	2/1/02	3581	300	629	1.939	1.9392	324	324	1.30E-5	**1.9**	1.30E-3	103.7	2.08E-1
RZ101-Fdi-3	2/1/02	3634	300	639	1.940	1.9395	330	330	1.32E-5	**2.8**	1.32E-3	104.6	2.14E-1
RZ101-Fdi-4	2/1/02	8185	300	1549	1.938	1.9378	800	800	3.21E-5	**3.7**	3.21E-3	105.5	5.21E-1
RZ101-Fdi-5	2/1/02	3125	300	537	1.932	1.9324	278	278	1.12E-5	**4.5**	1.12E-3	106.2	1.57E-1
RZ101-Fdi-6	2/1/02	2881	300	489	1.949	1.9492	251	251	1.01E-5	**5.4**	1.01E-3	107.1	1.63E-1
RZ101-Fdi-7	2/1/02	2572	300	427	1.933	1.9334	221	221	8.86E-6	**6.3**	8.86E-4	108.1	1.46E-1
RZ101-Fdi-8	2/1/02	2162	300	345	1.926	1.9262	179	179	7.18E-6	**7.2**	7.18E-4	108.9	1.14E-1
RZ101-Fdi-9	2/1/02	1877	300	288	1.930	1.9302	149	149	5.98E-6	**8.1**	5.98E-4	109.8	9.29E-2
RZ101-Fdi-10	2/1/02	1633	300	239	1.923	1.9230	124	124	4.99E-6	**9.0**	4.99E-4	110.7	8.05E-2
												sum	**1.96E+0**

AZ-101 Re-run
Lead column, eluting phase

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	counted mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	**BV**	Position
RZ101L-E1-d	1/30/02	3109	63	300	534	0.0347	3.43E-2	1.54E+4	1.56E+4	6.25E-4	**0.8**	1
RZ101L-E2-d	1/30/02	2243	53	300	361	0.0174	1.72E-2	2.08E+4	2.10E+4	8.43E-4	**1.7**	1
RZ101L-E3-dd	1/30/02	65,060	294	300	12924	1.82E-4	1.80E-4	7.11E+7	7.20E+7	2.89E+0	**2.5**	1
RZ101L-E4-dd	1/30/02	41281	237	300	8169	1.70E-4	1.68E-4	4.81E+7	4.86E+7	4.97E+1	**3.4**	6
RZ101L-E5-dd	1/30/02	211838	542	300	42280	1.82E-4	1.79E-4	2.33E+8	2.36E+8	9.46E+0	**4.3**	1
RZ101L-E6-dd	1/30/02	17213	150	300	3355	1.75E-4	1.73E-4	1.92E+7	1.94E+7	7.78E-1	**5.2**	1
RZ101L-E7-dd	1/30/02	3138	65	300	540	1.72E-4	1.70E-4	3.14E+6	3.18E+6	1.28E-1	**6.1**	1
RZ101L-E8-d	1/30/02	160018	470	300	31916	1.83E-2	1.81E-2	1.75E+6	1.77E+6	7.09E-2	**7.0**	1
RZ101L-E9-d	1/30/02	128351	416	300	25583	3.61E-2	3.57E-2	7.09E+5	7.17E+5	2.88E-2	**7.9**	1
RZ101L-E10-d	1/30/02	71801	309	300	14273	0.0339	3.35E-2	4.21E+5	4.26E+5	1.71E-2	**8.9**	1
RZ101L-E11-d	1/30/02	130097	418	300	25932	0.0379	3.74E-2	6.85E+5	6.93E+5	2.78E-2	**9.6**	1
RZ101L-E12-d	1/30/02	32040	205	300	6320	0.0319	3.15E-2	1.98E+5	2.01E+5	8.06E-3	**10.6**	1
RZ101L-E13-d	1/30/02	23239	175	300	4560	0.0196	1.94E-2	2.32E+5	2.35E+5	9.44E-3	**11.4**	1
RZ101L-E14-d	1/30/02	15785	144	300	3069	0.0397	3.93E-2	7.72E+4	7.81E+4	3.14E-3	**12.4**	1
RZ101L-E15-d	1/31/02	11952	124	300	2303	0.0377	3.73E-2	6.10E+4	6.18E+4	2.48E-3	**13.2**	1
RZ101L-E16-d	1/31/02	9601	112	300	1833	0.0368	3.64E-2	4.98E+4	5.04E+4	2.02E-3	**14.1**	1
RZ101L-E17-d	1/31/02	8100	104	300	1532	0.0372	3.67E-2	4.12E+4	4.17E+4	1.68E-3	**15.0**	1
RZ101L-E18-d	1/31/02	7484	99	300	1409	0.0377	3.72E-2	3.74E+4	3.78E+4	1.52E-3	**15.9**	1

Deionized water rinse following elution

Sample ID	Date	Net counts	Time (sec)	net cpm	volume, mL	net cpm/mL	C/Co	BV	**BV post Elution**	Cs-137 µCi	Position
RZ101-Edi-1	2/1/02	6013	300	1115	0.0380	29325	1.18E-3	0.9	**16.8**	1.92E+1	1
RZ101-Edi-2	2/1/02	4698	300	852	0.0381	22354	8.97E-4	1.9	**17.7**	1.54E+1	1
RZ101-Edi-3	2/1/02	3933	300	699	0.0367	19030	7.64E-4	2.8	**18.7**	1.28E+1	1
RZ101-Edi-4	2/1/02	3775	300	667	0.0363	18365	7.37E-4	3.7	**19.6**	1.23E+1	1
RZ101-Edi-5	2/1/02	136700	300	27252	1.9228	14173	5.69E-4	4.6	**20.4**	8.39E+0	1
RZ101-Edi-6	2/1/02	78632	300	15639	1.9356	8080	3.24E-4	5.5	**21.4**	5.65E+0	1
RZ101-Edi-7	2/1/02	54390	300	10790	1.9291	5593	2.25E-4	6.4	**22.3**	3.69E+0	1
									sum	**7.75E+1**	

Regeneration with 0.25 M NaOH

Sample ID	Date	Net counts	Time (sec)	net cpm	mass, g	volume, mL	net cpm/g	net cpm/mL	C/Co	Position	**BV**	Cs-137 uCi
RZ-101-RGN	2/6/02	173734	300	34659	1.8902	1.8789	18336	18446	7.40E-4	1	**24**	5.84E+1

Table C.8. Second Cs Ion Exchange Column Test: CMC ^{137}Cs Results, Cs Eluate Sample Integration, and Cs Recovery

Comparitor samples									
Sample ID	**RPL ID**	**^{137}Cs, µCi/mL**	**error**	**C/Co, %**	**BV**	**DF**			
AZ101LF0	02-909	1660	3	100	(not applicable)		1.72E+6	µCi AZ101 fed to column	
AZ101LF0D	duplicate	1630	3	100	(not applicable)		1.72E+0	Ci AZ-101	
	average	1645	3						
Load samples									
RZ-101L-F7A	02-1390	3.68E-2	3	2.24E-3	30.4	4.47E+4			
RZ101L-F18A	02-1391	6.43E-1	3	3.91E-2	78.3	2.56E+3			
RZ101L-F19A	02-1392	2.89E+0	3	1.76E-1	82.9	5.69E+2			
RZ101L-F20A	02-1393	1.16E+1	3	7.05E-1	87.2	1.42E+2			
RZ101L-F21A	02-1394	3.93E+1	3	2.39E+0	91.6	4.19E+1			
RZ101L-F22A	02-1395	1.19E+2	3	7.23E+0	95.9	1.38E+1			
RZ101P-F7A	02-1396	8.90E-1	3	5.41E-2	29.1	1.85E+3			
RZ101P-F14A	02-1397	6.25E-1	3	3.80E-2	57.3	2.63E+3			
RZ101P-F21A	02-1398	3.76E-1	3	2.29E-2	87.5	4.38E+3			
							mL processed		
RZ101FEcomp2A	02-1399	8.13E-1	3	4.94E-2	NA	2.02 E+3	8.15E+2	6.26E+2	uCi Cs total FE comp 2
RZ101FEcomp3A	02-1400	4.00E-1	3	2.43E-2	NA	4.11E+3	1.85E+2	7.40E+1	uCi Cs total FE comp 3
								7.00E+2	grand total Cs in effluents
								1.00E+3	mL total
								7.00E-1	uCi/mL Cs-137 in composite effluent
Elution samples								2.35E+3	RZ-101composite effluent DF
Sample ID	**RPL ID**	**^{137}Cs, µCi/mL**	**error**	**C/Co**	**BV**	**^{137}Cs µCi**			
RZ101L-E3	02-1401	1.18E+04	3	7.17	2.5	1.06E+5			
RZ101L-E4	02-1402	1.44E+05	3	87.54	3.4	1.40E+6			
RZ101L-E5	02-1403	4.17E+04	3	25.35	4.3	4.23E+5			
RZ101L-E6	02-1404	2.84E+03	3	1.73	5.2	2.79E+4			
RZ101L-E7	02-1405	5.20E+02	3	0.32	6.1	5.00E+3			
RZ101L-E10	02-1406	2.65E+01	3	1.61E-2	8.9	2.76E+2			
RZ101L-E14	02-1407	5.61E+0	3	3.41E-3	12.4	5.69E+1			
					sum	1.97E+6	Cs-137 µCi		
Composite eluate	02-2239	1.20E+04	3	7.29E+0	NA	2.07 E+6			

Table C.9. Second Cs Ion Exchange Column Test: Cs Lead Column Breakthrough Integration

Activity of Cs-137 in Feed = 1.65E+03 μCi/mL

Sample	Processed Vol (mL)	Cs-137 C/C_o	Cs-137 Conc (μCi/mL)	Δ Vol (mL)	C/Co Midpoint	Midpoint Conc (μCi/mL)	Area (μCi)
RZ101L-F1	48.7	4.35E-5	7.16E-2	48.7	4.35E-5	7.16E-2	3.49E+0
RZ101L-F2	91.4	3.14E-5	5.17E-2	42.7	3.75E-5	6.16E-2	2.63E+0
RZ101L-F3	138.7	2.84E-5	4.68E-2	47.3	2.99E-5	4.92E-2	2.33E+0
RZ101L-F4	185.9	2.45E-5	4.03E-2	47.2	2.65E-5	4.35E-2	2.05E+0
RZ101L-F5	233.3	2.57E-5	4.23E-2	47.4	2.51E-5	4.13E-2	1.96E+0
RZ101L-F6	282.2	2.30E-5	3.79E-2	48.9	2.44E-5	4.01E-2	1.96E+0
RZ101L-F7	331.0	2.25E-5	3.71E-2	48.8	2.28E-5	3.75E-2	1.83E+0
RZ101L-F8	375.1	2.12E-5	3.49E-2	44.1	2.19E-5	3.60E-2	1.59E+0
RZ101L-F9	422.9	1.99E-5	3.28E-2	47.8	2.06E-5	3.39E-2	1.62E+0
RZ101L-F10	471.4	1.99E-5	3.27E-2	48.5	1.99E-5	3.28E-2	1.59E+0
RZ101L-F11	519.7	1.80E-5	2.97E-2	48.4	1.90E-5	3.12E-2	1.51E+0
RZ101L-F12	565.9	1.78E-5	2.93E-2	46.2	1.79E-5	2.95E-2	1.36E+0
RZ101L-F13	611.9	1.78E-5	2.92E-2	45.9	1.78E-5	2.93E-2	1.34E+0
RZ101L-F14	652.7	1.77E-5	2.90E-2	40.8	1.77E-5	2.91E-2	1.19E+0
RZ101L-F15	710.0	1.94E-5	3.19E-2	57.3	1.85E-5	3.05E-2	1.75E+0
RZ101L-F16	748.0	2.46E-5	4.05E-2	38.0	2.20E-5	3.62E-2	1.38E+0
RZ101L-F17	807.6	8.81E-5	1.45E-1	59.5	5.63E-5	9.27E-2	5.52E+0
RZ101L-F18	853.2	3.69E-4	6.06E-1	45.7	2.28E-4	3.76E-1	1.72E+1
RZ101L-F19	903.3	1.68E-3	2.76E+0	50.1	1.02E-3	1.68E+0	8.43E+1
RZ101L-F20	950.8	7.09E-3	1.17E+1	47.5	4.38E-3	7.21E+0	3.42E+2
RZ101L-F21	998.1	2.33E-2	3.83E+1	47.3	1.52E-2	2.50E+1	1.18E+3
RZ101L-F22	1045.6	6.59E-2	1.08E+2	47.6	4.46E-2	7.34E+1	3.49E+3

Full load: **1048.1mL (obtained by mass difference of feed bottle)** 5.15E+03μCi

Amount of Cs that Broke through Lead Column = amount of Cs on the Lag Column = 5152μCi
= 0.0052Ci

Amount of Cs Fed to Lead Column = 1.72E+06μCi
1.724Ci

Percentage of Cs-137 on Lag Column = 0.3%

Amount of Cs Loaded on Lead Column = 1.719Ci

Table C. 10. Second Cs Ion Exchange Column Test: Cs Isotopic and Mass Distributions

AZ-101 Cs isotopic distribution
ASR 6403 #02-2246 (Eluate)
Thermal ionization mass spectrometry weight fraction ratio

	Cs-133	**Cs-135**	**Cs-137**
wt fraction	0.5185	0.169	0.3125

The eluate isotopic distribution is considered most accurate and is applied to the eluate, effluent, feed and regeneration solutions. Total Cs was calculated from 137Cs and the isotopic ratio.

ASR 6306 RPL # 02-0909 Feed **1048.1 mL**

Cs IX feed	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	7.51	2194	3.42 E-3	7.87 E-3	
Cs-137	1640	87	18.85	1.72 E+0	1.98 E+1

	Cs-133	Cs-135	Cs-137	Sum Conc.	total mass (mg) or millimoles
µg/mL Cs	31.277	10.194	18.851	60.322	6.32E+1
M Cs	2.35E-4	7.55E-5	1.38E-4	4.48E-4	4.70E-1

The total Cs concentration is 18.85 µg/mL divided by 0.3125
60.32 µg/mL Cs in the AZ-102 feed

ASR 6360 RPL # 02-1399 AZ-101 effluent **769.8 mL**

Cs IX effluent	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	<1.0E-4	2194	<5.E-8	7.70 E-8	
Cs-137	8.13E-01	87	9.34 E-3	6.26 E-4	7.19 E-3

2nd run effluent Cs Concentration FEcomp 2

	Cs-133	Cs-135	Cs-137	Sum Conc.	total mass (mg) or millimoles
µg/mL Cs	1.55 E-2	5.05 E-3	9.34 E-3	2.99 E-2	2.30E-2
M Cs	1.17E-7	3.74E-8	6.82E-8	2.22E-7	1.71E-4

The total Cs concentration is 9.34E-3 µg/mL divided by 0.3125
2.99 E-2 µg/mL Cs in the AZ-102 FE comp 2

ASR 6401 #02-2239 (AZ-101 eluate 2nd run) **172.9 mL**

Cs IX effluent	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	4.09 E+0	2194	1.86 E-3	7.07 E-4	
Cs-137	1.20E+04	87	1.38 E+2	2.07 E+0	2.38 E+1

	Cs-133	Cs-135	Cs-137	Sum Conc.	total mass (mg) or millimoles
µg/mL Cs	2.29 E+2	7.46 E+1	1.38 E+2	4.41 E+2	7.63E+1
M Cs	1.72E-3	5.53E-4	1.01E-3	3.28E-3	5.67E-1

The total Cs concentration is 1.38E+2 µg/mL divided by 0.3125
4.41 E+2 µg/mL Cs in the AZ-102 feed

ASR 6360 RPL # 02-1400 AZ-101 effluent **175.3 mL**

Cs IX effluent	µCi/mL	SpA, uCi/µg	µg/mL	total Ci	total mg
Cs-134	<8.0E-5	2194	<4.E-8	<1.4E-8	
Cs-137	4.00E-01	87	4.60 E-3	7.01 E-5	8.06 E-4

2nd run effluent Cs Concentration FEcomp 3

	Cs-133	Cs-135	Cs-137	Sum Conc.	total mass (mg) or millimoles
µg/mL Cs	7.63 E-3	2.49 E-3	4.60 E-3	1.47 E-2	2.58E-3
M Cs	5.74E-8	1.84E-8	3.36E-8	1.09E-7	1.92E-5

The total Cs concentration is 4.60E-3 µg/mL divided by 0.3125
1.47 E-2 µg/mL Cs in the AZ-102 FE comp 3

Note: SpA = specific activity
0.30% broke through the lead column and onto the lag column

Amount loaded on the lag column

Cs-137	5.16E-3	µCi
Cs-137	59	µg
Cs-total	190	µg
Cs-total	1.4 E-3	mmoles

Net lead column Cs loading

Cs-137	1.72 E+6	µCi
Cs-137	1.97 E+4	µg
Cs-total	6.30 E+4	µg
Cs-total	4.68 E-1	mmoles

Appendix D

Analytical Data

Appendix D: Analytical Data

Table D. 1. Sample Identification

Sample Description	ASR	RPL ID	Sample Identification	Extended Sample Description
Batch Contact Testing				
Batch contact with SL-644 010319SMC-IV-73 212- to 425-μm particle size H-form resin	6413	02-2288	AZ-101-TI164-S0-FA	Batch contact control, unspiked,
		02-2289	AZ-101-TI164-S0-D-FA	Batch contact control, unspiked, duplicate
		02-2290	AZ-101-TI164-S0-73-FA	Batch contact with SL-644, unspiked
		02-2291	AZ-101-TI164-S0-73D-FA	Batch contact with SL-644, unspiked, duplicate
		02-2292	AZ-101-TI164-S1-FA	Batch contact control, 4E-3M Cs
		02-2293	AZ-101-TI164-S1-D-FA	Batch contact control, 4E-3 M Cs, duplicate
		02-2294	AZ-101-TI164-S1-73-FA	Batch contact with SL-644, 4E-3 M Cs
		02-2295	AZ-101-TI164-S1-73D-FA	Batch contact with SL-644, 4E-3 M Cs, duplicate
		02-2296	AZ-101-TI164-S2-FA	Batch contact control, 7E-3 M Cs
		02-2297	AZ-101-TI164-S2-D-FA	Batch contact control, 7E-3 M Cs, duplicate
		02-2298	AZ-101-TI164-S2-73-FA	Batch contact with SL-644, 7E-3
		02-2299	AZ-101-TI164-S2-73D-FA	Batch contact with SL-644, 7E-3 M Cs, duplicate
Column Run				
Initial Feed Sample and column processing samples First test	6306	02-0909	AZ101-F0	AZ-101/Cs IX feed
		02-0910—02-0918	AZ101L-F5, -F11, -F17, -F20, -F21, -F22, -F23, -F24, -F25	Lead column load samples
		02-0919—02-0923	AZ101P-F5, -F10, -F15, -F20, -F25	Lag column load samples
		02-0924—02-0925	AZ-101-FD-3 and –8	Feed displacement samples
		02-0926—02-0927	AZ-101-Fdi-3 and –8	Water rinse samples
		02-0928—02-0933	AZ101L-E3, -E4, -E5, -E6, -E11, -E15	Lead column elution samples
		02-0934	AZ101-RGN	Regeneration solution analytical sample
		02-0935	AZ101-FEcomp	Effluent composite
Column processing samples Second test	6360	02-1408	RZ101-F0	AZ-101 Cs IX feed sample
		02-1390—02-1395	RZ101L-F7, -F18, -F19, -F20, -F21, -F22	Lead column load samples
		02-1396—02-1398	RZ101P-F7, -F14, -F21	Lag column load samples
		02-1399	RZ101FEcomp2	723-mL effluent composite sample
		02-1400	RZ101FEcomp3	173-mL effluent composite sample
		02-1402—02-1407	RZ101L-E3, -E4, -E5, -E6, -E7, -E10, -E14	Eluate samples
Composite eluate	6401	02-2238	AZ101-CsEluate-comp	Composited eluate samples from first test
Composite eluate	6401	02-2239	RZ101-CsEluate-comp	Composited eluate samples from second test
Composite eluate	6403	02-2246	AZ101-CsEluate-comp-HLW	Combined eluates from first and second test
Composite eluate	6566	02-3345	AZ-101 Cs Eluate-comp	Composited eluate for Pu re-analysis from first test
		02-3346	RZ-101 Cs Eluate-comp	Composited eluate for Pu re-analysis from second test
		02-3347	AZ-101 Cs Eluate-comp-HLW	Combined eluates for Pu re-analysis

Notes for batch contact sample identifications: The suffix letter "F" indicates the sample was filtered; "A" indicates the sample was loaded out of the hot cell into a clean analytical vial. Unidentified samples are associated with other tests unrelated to this work.

ASR 6413

GEA

File: 02-2288.xls

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

3/25/2002

Client : Sandy Fiskum
ASR: 6413

Cognizant Scientist: L R Greenwood Date : 3/25/02

Concur : T Trang-Le Date : 3/25/02

Reference Date: March 20, 2002 Procedure: PNL-ALO-450

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Cs-137	Error+/-%
02-2288 AZ101-TI164-S0-FA	1.89E+3	3%
02-2289 AZ101-TI164-S0-D-FA	2.03E+3	3%
02-2290 AZ101-TI164-S0-73-FA	1.63E+2	3%
02-2291 AZ101-TI164-S0-73D-FA	1.77E+2	3%
02-2292 AZ101-TI164-S1-FA	1.98E+3	3%
02-2293 AZ101-TI164-S1-D-FA	7.62E+2	3%
02-2294 AZ101-TI164-S1-73-FA	7.73E+2	3%
02-2295 AZ101-TI164-S1-73-D-FA	7.91E+2	3%
02-2296 AZ101-TI164-S2-FA	1.82E+3	3%
02-2297 AZ101-TI164-S2-D-FA	1.88E+3	3%
02-2298 AZ101-TI164-S2-73-FA	9.79E+2	3%
02-2299 AZ101-TI164-S2-73-D-FA	1.01E+3	3%
02-2300 AZ102-TI164-S0-FA	2.18E+3	3%
02-2301 AZ102-TI164-S0-D-FA	2.25E+3	3%
02-2302 AZ102-TI164-S0-73-FA	7.35E+2	3%
02-2303 AZ102-TI164-S0-73D-FA	6.77E+2	3%
02-2304 AZ102-TI164-S1-FA	2.36E+3	3%

File: 02-2288.xls

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Cs-137	Error+/-%
02-2305 AZ102-TI164-S1-D-FA	2.34E+3	3%
02-2306 AZ102-TI164-S1-73-FA	1.48E+3	3%
02-2307 AZ102-TI164-S1-73-D-FA	1.42E+3	3%
02-2308 AZ102-TI164-S2-FA	2.32E+3	3%
02-2309 AZ102-TI164-S2-D-FA	2.28E+3	3%
02-2310 AZ102-TI164-S2-73-FA	1.53E+3	3%
02-2311 AZ102-TI164-S2-73-D-FA	1.60E+3	3%

ASR 6306

ICP-AES (Feed and Effluent)
ICP-AES (Regeneration Solution)
U (KPA)
IC (Feed)
IC (Effluent)
OH
GEA
Pertechnetate (Effluent Only – feed sample appears to be the acid-digested material)
Pu (Effluent)
Cs isotopic (TIMS)

Battelle PNNL/RSE/Inorganic Analysis … ICPAES Analysis Report
PO Box 999, Richland, Washington 99352

Project / WP#: 42365 / W60553
ASR#: 6306
Client: S. Fiskum
Total Samples: 3

RPL#:	02-00909	02-00935
Client ID:	AZ101-F0	AZ101-FEcomp
Sample Preparation: PNL-ALO-128 (SAL/vh). **AOIs:** Al, Ca, Cd, Cr, K, Li, Na, and U. **Notes:** ASR 6306 sample 02-00934 was not processed or analyzed with these samples.		

REVISION 1: Expanded Discussion on LCS Na and K Failure

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 02-13-02 (A0769)

See **Chemical Measurement Center 98620 file:** ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

M W Thice 10-18-02
Prepared by

B.M. Olson 10/18/02
Reviewed by

Two samples of AZ101 tank waste (RPL#'s 02-00909 and 02-00935), were processed using a nitric/hydrochloric acid digestion procedure (PNL-ALO-128) in the Shielded Analytical laboratory (SAL). The samples were prepared using approximately 1 mL (weighed) aliquots of liquid material. After digestion the samples were diluted to a final volume of about 25 mL (weighed). Sample density of liquid before processing was estimated to be about 1.24 g/mL based on the average weight of the three 1 mL sample aliquots (see worksheets in supporting documents for sample preparation information). The final digestate was weighed and the volume calculated based upon estimated densities. Analytes of interest (AOI) were specified in a table attached to the ASR. All other analytes that were not requested are reported, but have not been fully evaluated for QC performance.

A summary of the ICP analyses, including QC performance, is given in the attached ICPAES Data Report. ICPAES measurement results are reported in µg/mL and have been corrected for dilution resulting from sample processing.

The process blank had detectable amounts of Al and Na present. The Al was below estimated quantitation limits (EQL). The Na present in the process blank was at a concentration greater than EQL and likely originated from glassware used to digest the samples or (less likely) from the digestion reagents, nitric and hydrochloric acid. The Na concentration in the process blank, however, is only about 0.05% of that found in the AZ101 samples. All other analytes of interest (AOI's) were below detection limits.

Quality control standard results met tolerance requirements for the specific AOIs except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan. LCS/BS, matrix-spikes and duplicate were prepared with the samples and analyzed. LCS/blank-spike and matrix-spike were prepared using 1.5 and 0.5 mL, respectively, of multi-element solutions "BPNL-QC-1" and "BPNL-QC-2" per 25 mL of digestate volume.

Process Blanks:

Concentration of analytes of interest measured in the process blank were all within tolerance limit of ≤ EQL or less than ≤5% of the sample.

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample (RPL# 02-00909) were within tolerance limit of ≤ 15% RPD for all analytes of interest. Sodium was over range for the undiluted samples, but was within tolerance for the @5 dilutions.

Blank Spike:

Blank-Spike recovery for the analytes of interest was within tolerance of 80% to 120% except for K which was recovered at only 34% and Na which was recovered at 129%.

The under recovery for the K has been determined to be from lower than specified levels of K in the BPNL-QC-2 spike material. The laboratory analyzed the PBNL-QC-2 solution multiple times with an average recovery of about 35% of the certified concentration. The vendor who supplied the multi-element standard was notified and conducted an independent evaluation of archived material. The vendor measured a K concentration of 1725 µg/mL in the archive, where the certified value was 5000 µg/mL. This evaluation was documented on the vendor's "Inquiry Report Form CL# 4359 (02/04/02)". A copy of this form and the laboratory's results on PBNL-QC-2 after 3 months is included in the ICP-AES system file. The 1725 µg/mL is not a certified value and cannot be used for reporting the LCS and MS recoveries.

The failure of the LCS for Na is attributed to blank contamination from processing in glass using digestion process PNL-ALO-128, a nitric acid and hydrochloric acid digestion process. This digestion processing typically uses non-glass digestion vessels (e.g., Teflon, polyethylene, etc.) whenever Na, Si, or B (major glass components) is an analyte of interest. The Na concentration in the processing blank was approximately 2 times greater than the estimated quantitation limit (EQL); however, this contribution has been subtracted from the LCS. The elevated levels of Na measured in the LCS is most likely due to the presence of HF, which is present at 0.7% in PBNL-QC-2 to maintain the Si in the standard solution. The excessive recovery of Na is most likely confined to the LCS, which is the only samples containing the HF, except the MS for which the Na was over range and could not be determined). The failure of the LCS Na is not considered a systematic problem and is the result of processing standard solutions containing HF in glass digestion vessels. Since the absolute effect of the HF on the glass vessels is likely variable and cannot be quantified, it is not possible to correct the recoveries to account for this effect.

Matrix Spiked Sample:

Matrix-Spike recoveries for analytes of interest are within tolerance of 75% to 125% except for K, which showed a recovery of only 25%. The under recovery of K is attributed to the lower K concentration in the BPNL-QC-2 spike discussed above. Chromium and sodium exceeded spike concentration by more than five times and therefore were not recovered. Post-spikes and serial dilutions are used to evaluate chromium and sodium.

Post-Spiked Samples (Group A):

All post-spiked analytes of interest in the sample tested were recovered within tolerance of 75% to 125% except Al, Cr, and Na, which were not recovered. The spike concentrations for Al, Cr, and Na were less than 20% of the sample concentrations. High concentration of Na (approximately 110,000 ug/ml) was verified by analysis of serial diluted aliquots.

Post-Spiked Samples (Group B):

No analytes of interest were included in the Group B post-spike.

Five fold serial dilution:

All analytes above EQL in the sample tested were within tolerance limit of 10% after correcting for dilution. Sodium was over range for the @1/@5 dilution test, but was within tolerance for the @5/@25 test.

Comments:

1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

	Run Date=	2/13/2002	2/13/2002	2/13/2002	2/13/2002	2/13/2002	2/13/2002	2/13/2002
	Multiplier=	26.6	25.5	127.7	25.9	129.3	26.0	130.1
	RPL/LAB #=	02-00909-B	02-00909	02-00909 @5	02-00909-DUP	02-00909-DUP @5	02-00935	02-00935 @5
Det. Limit	Client ID=	process blank	AZ101-F0		AZ101-F0-Dup		AZ101-FEcomp	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	[5.4]	5,600		5,660		5,560	
0.250	Ca	--	--		--		--	
0.015	Cd	--	--		--		--	
0.020	Cr	--	624		631		613	
2.000	K	--	4,450		4,510		4,350	
0.030	Li	--	[0.85]		[0.93]		--	
0.150	Na	58.2	over range	111,000	over range	112,000	over range	112,000
2.000	U	--	--		[57]		--	
Other Analytes								
0.025	Ag	--	--		--		--	
0.250	As	--	[8.9]		[8.9]		[7.9]	
0.050	B	47.2	81.5		69.3		70.5	
0.010	Ba	--	[0.29]		[0.30]		--	
0.010	Be	--	--		--		--	
0.100	Bi	--	--		--		--	
0.200	Ce	--	--		--		--	
0.050	Co	--	--		--		--	
0.025	Cu	--	--		--		--	
0.050	Dy	--	--		--		--	
0.100	Eu	--	--		--		--	
0.025	Fe	[1.4]	[2.9]		[2.3]		[1.6]	
0.050	La	--	--		--		--	
0.100	Mg	--	--		--		--	
0.050	Mn	--	--		--		--	
0.050	Mo	--	93.3		93.7		92.8	
0.100	Nd	--	--		--		--	
0.030	Ni	--	--		--		--	
0.100	P	--	504		510		495	
0.100	Pb	--	[3.0]		[3.3]		--	
0.750	Pd	--	--		--		--	
0.300	Rh	--	--		--		--	
1.100	Ru	--	--		--		--	
0.500	Sb	--	--		--		--	
0.250	Se	--	--		--		--	
0.500	Si	[110]	182		[41]		[130]	
1.500	Sn	--	[54]		[57]		[48]	
0.015	Sr	--	--		--		--	
1.500	Te	--	--		--		--	
1.000	Th	--	--		--		--	
0.025	Ti	--	--		--		--	
0.500	Tl	--	--		--		--	
0.050	V	--	[1.5]		[1.5]		[1.4]	
2.000	W	--	[58]		[53]		[57]	
0.050	Y	--	--		--		--	
0.050	Zn	--	20.2		[1.3]		--	
0.050	Zr	--	[2.0]		[2.0]		--	

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 2/13/02

Criteria>	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-00909 & 02-00909-D	02-00909 LCS/BS	02-00909 & 02-00909-MS	02-00909 @5 + Post Spike A	02-00909 @5 + Post Spike B	02-00909 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	1.0	102	85	nr		3.0
Ca		101	92	102		
Cd		101	91	102		
Cr	1.0	103	nr	nr		4.2
K	1.3	34	29	96		0.1
Li		102	90	101		
Na	0.8 (b)	129	nr	nr		0.9 (b)
U		104	90		99	
Other Analytes						
Ag				99		
As				104		
B	16.1	164	110	100		6.3
Ba		102	88	100		
Be		99	90	102		
Bi		105	100	102		
Ce		98	90		94	
Co				104		
Cu		104	77	99		
Dy					100	
Eu					101	
Fe		104	91	104		
La		100	89		97	
Mg		103	93	108		
Mn		103	91	104		
Mo	0.4	104	93	102		3.1
Nd		100	89		95	
Ni		104	92	104		
P	1.2	102	93	101		0.2
Pb		116	106	116		
Pd						
Rh					92	
Ru						
Sb				99		
Se				104		
Si		66	16	125		
Sn					99	
Sr		103	94	103		
Te					102	
Th		97	90		100	
Ti		100	90	98		
Tl				102		
V		96	86	98		
W			84			
Y				99		
Zn		105	24	105		
Zr		102	89	103		

Shaded results exceed acceptance criteria

n.r. = not recovered; spike concentration less than 20% of sample concentration

(a)=RPD <3.5% for Na (only) *(b)=@5x dilution*

Project / WP#: 42365 / W60555 & W60553
ASR#: 6280 & 6306
Client: S. Fiskum
Total Samples: 2 (liquid)

	First	Last
RPL#:	02-00775	02-00934
Client ID:	"AZ102-RGN"	"AZ101-RGN"
Sample Preparation: Acid dilution (SAL/vh)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 04-29-2002 (A0801)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

M W Lee 5/2/02
Reviewed by

S. M. Olsen 5/2/02
Concur

Two ion exchange column regeneration samples from Analytical Service Requests (ASR) 6280 and 6303 were analyzed by ICPAES. The samples were prepared by the Shielded Analytical Laboratory (SAL) by diluted ~1 mL of sample with 0.5 molar nitric acid to a final volume of ~10 mL.

Analytes of interest (AOIs) were specified in the ASR and included Al, Ca, Cd, Cr, K, Li, Na, and U. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those identified as analytes of interest are reported, but have not been fully evaluated for QC performance.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (2 pages). The results are reported as µg/mL for each detected analyte. The reported data have been adjusted for the SAL dilution factor of 10.12.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the dilution processing, a reagent blank and duplicates were prepared along with the samples.

Process Blank:

A process blank was prepared by the SAL using reagents only. All AOIs measured in the blank were within the acceptance criteria of ≤ EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike (laboratory control sample):

No blank spike was provided for analysis.

Matrix Spiked Sample:

No matrix spike was provided for analysis.

Duplicate RPD (Relative Percent Difference):

A duplicate was prepared for each sample. RPDs for all AOIs measured above the EQL were with the acceptance tolerance of ±15% (±3.5% for Na).

Post-Spiked Samples (Spike A Elements):

All analytes of interest contained in the group A elements, measured above the EQL, were recovered within the acceptance criteria of 75% to 125%.

Post-Spiked Samples (Spike B Elements):

All analytes of interest contained in the group B elements, measured above the EQL, were recovered within the acceptance criteria of 75% to 125%. Uranium was recovered, but at a level below the EQL.

Five fold serial dilution:

Serial dilution was performed on Sample AZ101-RGN. All AOIs measured above the EQL were within the acceptance criteria of ±10%.

Other QC:

All required instrument-related QC tests for the analytes of interest were within the acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilutions performed on the sample during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICPAES measurements is 2.

Battelle PNNL/RSE/Inorganic Analysis.... ICPAES Report

	Multiplier=	10.12	10.12	10.12	10.12	10.12
	RPL/LAB #=	02-00934-BLK	02-00775	02-00775-DUP	02-00934	02-00934-DUP
	Client ID=	process blank	AZ102-RGN		AZ101-RGN	
Det. Limit	Run Date=	4/29/2002	4/29/2002	4/29/2002	4/29/2002	4/29/2002
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	--	--	--	[0.61]	[0.78]
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.020	Cr	--	[0.22]	[0.22]	[0.45]	[0.46]
2.000	K	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.150	Na	--	[10]	[10]	119	121
2.000	U	--	--	--	--	--
Other Analytes						
0.025	Ag	--	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	--	[0.72]	[0.73]	[1.2]	[1.2]
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	[0.30]	[0.32]
0.050	La	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	--	--	--	--	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	--	--	--	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
0.050	V	--	--	--	--	--
0.500	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 4/29/02

Criteria>	<15%[a]	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-00775 & 02-00775-D	02-00934 & 02-00934-D	LCS/BS (none)	MS (none)	02-00934 + Post Spike A	02-00934 + Post Spike B	02-00934 @1/@5 Serial Dil
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al					102		
Ca					108		
Cd					106		
Cr					102		
K					108		
Li					106		
Na		1.5			105		-0.2
U						105	
Other Analytes							
Ag					102		
As					107		
B					106		
Ba					101		
Be					102		
Bi					102		
Ce						100	
Co					105		
Cu					104		
Dy						105	
Eu						106	
Fe					103		
La						103	
Mg					108		
Mn					109		
Mo					105		
Nd						102	
Ni					105		
P					106		
Pb					111		
Pd						97	
Rh						100	
Ru							
Sb					105		
Se					110		
Si					112		
Sn							
Sr					102		
Te							
Th						105	
Ti					98		
Tl					104		
V					100		
W							
Y					100		
Zn					108		
Zr					102		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

(a) = RPD <3.5% for Na (only)

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

filename 02-0909
6/11/2002

Client: S. Fiskum
ASR 6306
Procedure RPG-CMC-4014, Rev 1, Uranium Analysis by Kinetic Phosphorescence
Chem-Chek Instruments model KPA-11R uranium analyzer

Prepared by: C. Soderquist 6-11-02
Reviewed by: LR Greenwood 6-12-02

Sample	Lab ID	Measured uranium concentration, mg U per liter ± 1s		Detection Limit
AZ101L-F0	02-0909	**2.23E+1**	**2%**	**5.E-2**
AZ101L-F0	02-0909 Dup	**2.22E+1**	**3%**	**5.E-2**
AZ101-FE COMP	02-0935	**2.24E-3**	**8%**	**2.E-4**
	Blank	**1.97E-1**	**18%**	**5.E-2**
	LCS	111%		
	Matrix spike 909	75%		

(Matrix spike was less then 10% of sample uranium concentration.)

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology – IC Report
PO Box 999, Richland, Washington 99352

Project / Charge Code: 42365 / W60553
ASR Number: 6306
Client: S. Fiskum
Total Samples: 2

	First in Series	Last in Series
RPL Numbers	02-0909	02-0935
Client IDs	AZ-101 Cs IX Feed	AZ-101 FE Comp

Analysis Procedure	PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Prep Procedure	None
Analyst	MJ Steele
Analysis Date	06/25/2002, 06/25/2002
Calibration Date	04/17/2002
Cal/Ver Stds Prep Date	04/11/2002
Excel Data File	ASR 6306 6347 6401 6403.xls
M&TE Numbers	IC system (WD25214)
	Balance (360-06-01-031)
All Analysis Records	Chemical Measurement Center 98620 RIDS IC System File

MW Thun — 7/17/02
Prepared By **Date**

MJ Steele — 7-18-02
Reviewed By **Date**

Sample Results

RPL No.	Sample ID	SAL Dil Fctr	F µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL (a)		0.13	0.13	0.25	0.13	0.25	0.25	0.25	0.25
	Dilution Blank	1	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	MRQ		150	300	3,000	n/a	3,000	2,500	2,300	n/a
	EQL (a)		26	26	5,260	263	526	53	526	53
02-0909	AZ-101 Cs IX Feed	21.04	1,100	72	70,900	900	61,500	1,600	16,800	1,000
	EQL (a)		13	13	250	13	250	25	25	25
02-0935	AZ-101 FE Comp	1	140	< 13	3,600	45	3,100	130	1,100	90
02-0935 Dup	AZ-101 FE Comp Dup	1	140	< 13	3,700	46	3,200	130	1,100	86
	RPD		0	(b)	3	2	3	0	0	4
02-0909MS	AZ-101 Cs-IX-Feed MS %Recovery		126	113	109	103	95	106	105	106
LCS062402	LCS/BS % Recovery		106	103	105	106	99	107	105	109

MRQ: minimum reportable quantity EQL: estimated quantitation limit RPD: relative percent difference

Shaded results exceed QC acceptance criteria

(a) EQL based on lowest calibration standard times dilution factors used to obtain sample results

(b) RPD not calculated unless both sample and duplicate results >EQL

Sample Analysis/Results Discussion

The AZ-101 Cs IC Feed sample was prediluted in the Shielded Analytical Laboratory by approximately 20x. This samples, as well as the AZ-101 FE Comp sample, required additional laboratory dilutions from 10x to 1,000x in order to ensure that the anions were measured within the calibration range and that the IC column was not overloaded during the analysis. The minimum reportable quantities (MRQ) and the estimated quantitation levels (EQL) are provided; the MRQs are based on a table provided with ASR 6306 and the EQLs are based on the lowest calibration standard adjusted for the dilutions used for reporting the results.

It should be noted that the results from these samples are significantly different than those analyzed on 02/21/2002 and reported on 03/22/2002. The original results indicated that the sample labeled AZ-101 Cs IX feed was an acid digested sample and the current AZ-101 Cs IX Feed results should represent the true sample anion concentration. However, based on the AZ-101 FE Comp results, there appears that the sample was diluted in the SAL by about 20x (although the SAL benchsheet indicates 'no dilution').

Quality Control Discussion

Duplicate: No sample duplicate was provided by the client. A laboratory duplicate was prepared using sample 'AZ-101 FE Comp' The laboratory duplicate relative percent difference

(RPD) is well within the acceptance criterion of <15% (as defined by the ASR) for all anions measured above the EQL.

Laboratory Control Sample/Blank Spike - (BS 062402 [HCV020411 @3x)]): The high range calibration verification standard diluted by a factor of three was used as the LCS/BS. The LCS/BS demonstrated recoveries within the 80% to 120% acceptance criteria for all anions measured.

Matrix Spike (CCV020411 @2x): A matrix spike (MS) was prepared using the mid-range calibration check standard and the 'AZ-101 Cs IX Feed' sample. The MS recoveries were within the 75% to 125% recovery acceptance criteria for the anions of measured, except for fluoride that recovered at 126%. Slight interferences were noted in the fluoride region, most likely due to low molecular organic anions (e.g., formate).

Process/Dilution Blank: A dilution blank (i.e., diluted eluant) was analyzed for all reported analytes and no anions were measured above the EQL.

IC System QC samples: No anions of interest were measured in the calibration blanks (ICB/CCB). The four calibration verification standards analyzed with the samples met the 90% to 110% recovery criteria for all anions measured.

Summary of Deviation: None

General Comments

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested. For routine analyses, no results are reported below the EQL.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference.

Battelle PNNL/RPG/Inorganic Analysis --- IC Report

PO Box 999, Richland, Washington 99352

Client:	S. Fiskum	Charge Code/Project:	W60553 / 42365
ASR Number:	6306	Sample Receipt Date:	12/31/01
Sample Prep Date:	N/A	Sample Analysis Date:	02/21/2002
Analyst:	MJ Steele		

Preparation Procedure: Hot cell and analytical dilution only.
Analysis Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
M&TE: IC system (WD25214); Balance (360-06-01-031)
Records: CMC 98620 RIDS "IC File" for all IC analysis records and chromatograms.

Table 1: Sample Results

RPL Number	Sample ID	F (a) µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL	0.13	0.13	0.25	0.13	0.25	0.25	0.25	0.25
	Dilution Blank	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	EQL	25	630	50	630	1,300	50	50	50
02-00909	AZ101-F0 (b)	2,000	127,000	< 50	< 630	415,000	< 50	16,500	870
	EQL	50	1,300	100	2,500	2,500	100	100	100
02-00909 Dup	AZ101-F0 Dup (b)	2,300	139,000	< 100	< 2,500	460,000	< 100	18,600	< 100
	RPD	15%	9%	(c)	NA	10%	(c)	12%	(c)
	EQL	25	25	1,300	650	1,300	50	1,300	50
02-00935	AZ101-Fe Comp	1,400	< 25	64,200	730	53,700	1,400	16,900	960
02-00935 MS	Matrix Spike %Rec	97%	96%	102%	97%	99%	95%	95%	100%
	BS/LCS % Rec	94%	96%	97%	98%	92%	95%	93%	100%

EQL = estimated quantitation level
RPD = relative percent difference (between laboratory duplicates)

(a) The fluoride results should be considered the upper bound concentration for the fluoride, since the fluoride peak shape and retention time suggests the presence of co-eluting anion(s), possibly formate or acetate.

(b) The sample does not appear to be consistent with AZ-101 chemistry; most likely a sample of an acid digestion, where major constituents are nitric and hydrochloric acid.

(c) Not applicable; sample and/or duplicate concentration <EQL.

The samples associated with Analytical Service Request (ASR) 6306 were delivered to the IC laboratory from the Shielded Analytical Laboratory (SAL). Sample 02-00909 (AZ101-F0) was diluted 10.3-fold in the SAL. The samples were prepared for ion chromatography anion analysis by diluting an additional 200-fold to 10,000-fold in order to ensure that the anions of interest were measured within the calibration range and that column overloading was minimized. The stated estimated quantitation levels (EQL) are based on the lowest calibration standard adjusted for the SAL and IC laboratory dilutions used to obtain the sample results reported in Table 1.

NOTE – Resampling and reanalysis of AZ-101 F0 required: It is unlikely that sample 02-00909 (AZ101-F0) is a dilution of the feed material. The sample labeled 02-00909 is more likely a diluted aliquot of an acid digestion of the feed material. The SAL benchsheet indicates only that a sample of feed material was diluted by 10.3-fold; specifically to reduce the dose of the feed material being submitted to the IC laboratory.

Q.C. Comments:

Duplicates: Although somewhat higher than normal, the relative percent difference (RPD) for the sample and duplicate prepared from sample 02-00909 meets the acceptance criteria of <15% as stated in the ASR. The poor precision is most likely due to the need to make very high dilutions due to the very high chloride and nitrate concentrations.

Laboratory Control Sample/Blank Spike - (HCV 010912 @3x [LCS 021902]): A Blank Spike was prepared as the Laboratory Control Sample (LCS) and demonstrated recoveries within the 80% to 120% acceptance criteria as stated in the ASR.

Matrix Spike (HCV 010912 @2x): A matrix spike was prepared using sample 02-00935 and the high-range HCV standard solution. The anion recoveries are within the 75% to 125% acceptance criteria as stated in the ASR.

Process/Dilution Blank: The diluent used for diluting the samples was analyzed for chloride and nitrate. No anions were detected in the diluent blank above the laboratory's QA plan acceptance criteria of <EQL.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Report Prepared by: MW Thu Date 3-22-02

Review/Approval: MJ Steele Date 3-22-02

Excel Archive Information: ASR 6306 Fiskum.xls

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Client: Sandra Fiskum Date: 05/23/02

Subject: Hydroxide Analyses for: AZ-101 tank waste
ASR: 6306 **Re-analysis**

A reanalysis of the three samples was necessary since the original dilution information was not available for the first analysis run. The dilution bench sheet accompanies this data.

A diluted sample aliquot (21.04X dilution) of AZ-101-CsIX-Feed (02-0909) plus a replicate of 02-0909, a direct aliquot of AZ-101-RGN (02-0934), and a direct sample aliquot of Cs removed AZ-101-CsIX- FE-comp (02-0935), were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228. Sample O2-0934, AZ-101-RGN was slightly acidic as recieved an thus no real inflection points were measured. All samples were analyzed using a Brinkman 636 Auto-Titrator. The titrant used was 0.0203 M HCl and the base standard 0.0103 M NaOH was used for QC verification standards and matrix spike.

The attached Report Summary indicates that the hydroxide results were well above the required MRQ value of 17 ug/mL for all the samples. The hydroxide recovery was 92% for the standard, the matrix spike recovery on 02-0909 was 101% and the matrix spike recovery on 02-0935 was 89%. The first, second and third inflection points, frequently associated with hydroxide, carbonate and bicarbonate, showed excellent RPD's on sample 02-0935, well below the required RPD of +/- 15%. However the RPD's on 02-0909 and 02-0909-dup were 24 to 28% for the first and second inflection points. This exceeded the required RPD of +/- 15% and probably is due to the 21X dilution needed on this sample to meet dose limits in the analyical laboratory. Averaging all the results for sample 02-0909 and dup gives RSD (relative standard deviation) values of 17-18%, only slightly above the required value. The QC data met the acceptance criteria for the standards and the matrix spike recoveries.

Following is the report summary, the sample results calculated from the raw data, and the record file for the standardized acid and base used. Also included in this report are copies of the titration curves.

Prepared by: _____ Date: 5-23-02

Reviewed by: _____ Date: 5-23-02

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Chemical Measurements Center

ASR # 6306

WP# W60553

Hydroxide and Alkalinity Determination
Procedure: **PNL-ALO-228** Equip # **WB76843**

Report Summary for ASR # -- 6306 ---Reanalysis

RPG #	Client ID		OH conc ug/mL	Concentration, moles / Liter: First Point	RPD	Second Point	RPD	Third Point	RPD
02-0909	AZ-101- (Cs IX- Feed)		1.1E+04	0.65		0.81		0.63	
02-0909	AZ-101- (Cs IX- Feed)	Rep	1.4E+04	0.83	24%	0.62	27%	0.71	13%
02-0909-dup	AZ-101- (Cs IX- Feed)		9.3E+03	0.54		0.92		0.65	
02-0909-dup	AZ-101- (Cs IX- Feed)	Rep	1.2E+04	0.71	27%	0.69	28%	0.73	11%
					RSD		RSD		RSD
Average	AZ-101-FE comp		1.2E+04	0.68	18%	0.76	17%	0.68	7%
02-934	AZ-101 -RGN	pH=	6.2	Re-generate sample was acidic initially.					
02-934	AZ-101 -RGN	pH=	6.4						
02-935	AZ-101-FE comp		4.0E+03	0.24		0.03		0.04	
02-935	AZ-101-FE comp	Rep	4.0E+03	0.24	0%	0.04	12%	0.04	6%

OH conc (ug/mL) = M (g/L) * 17,000

MDQ: 1.7E+01

Required RPD: +/- 15%

		Allowed Recovery Range
Standard 1	92%	+/- 20%
MS 02-909 Matrix spike	101%	+/- 25%
MS 02-935 Matrix spike	89%	+/- 25%

Note: Results are presented for the first, second, and third inflection points on the titration curves, as applicable. The first inflection point is generally associated with the hydroxide concentration. The second and third points generally represent the carbonate and bicarbonate concentrations.

Analyst: R S Swenson 5/23/02

Reviewer: H D Greenwood 5/23/02

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

File: 02-909
06/13/02

Client : Sandy Fiskum
ASR: 6306

Cognizant Scientist: L R Greenwood Date : 6/13/02

Concur : T Trang-Le Date : 6/14/02

Reference Date: * Dec. 26, 2001
Reference Date: Feb 21, 2002
Procedure: PNL-ALO-450 - Gamma Energy Analysis

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	RuRh-106 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
02-0909* AZ101L-F0	<9.E-2	<7.E+0	<2.E1	<2.E+0	8.04E-1 11%	1.66E+3 3%	<2.E-1	<6.E0	<2.E1
02-0909 Dup* AZ101L-F0 Dup	<7.E-2	<7.E+0	<2.E1	<2.E+0	7.44E-1 11%	1.63E+3 3%	<2.E-1	<6.E0	<2.E1
RPD					8%	2%			
02-0910 AZ101L-F5	3.07E-5 8%	1.49E-2 3%	7.44E-4 8%	2.36E-3 2%	<9.E-6	1.08E-2 3%	<2.E-5	<7.E-5	<8.E-5
02-0911 AZ101L-F11	2.03E-5 13%	1.47E-2 3%	1.03E-3 7%	2.35E-3 2%	<2.E-5	7.61E-3 4%	<2.E-5	<2.E-4	<3.E-4
02-0912 AZ101L-F17	2.42E-5 9%	1.43E-2 3%	9.93E-4 9%	2.20E-3 2%	<2.E-5	2.01E-1 3%	<2.E-5	<2.E-4	<4.E-4
02-0913* AZ101L-F20	<8.E-2	<2.E+0	<3.E0	<2.E-1	<2.E-1	5.67E+1 3%	<2.E-1	<2.E0	<3.E0
02-0914*	<5.E-2	<2.E+0	<3.E0	<3.E-1	<2.E-1	1.17E+2	<2.E-1	<2.E0	<4.E0

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	RuRh-106 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
AZ101L-F21						3%			
02-0915* AZ101L-F22	<8.E-2	<3.E+0	<4.E0	<4.E-1	<2.E-1	2.34E+2 3%	<2.E-1	<2.E0	<5.E0
02-0916* AZ101L-F23	<1.E-1	<4.E+0	<6.E0	<5.E-1	<3.E-1	4.48E+2 3%	<2.E-1	<3.E0	<6.E0
02-0917* AZ101L-F24	<5.E-2	<4.E+0	<6.E0	<6.E-1	<3.E-1	5.21E+2 3%	<2.E-1	<3.E0	<7.E0
02-0918* AZ101L-F25	<1.E-1	<5.E+0	<7.E0	<7.E-1	<2.E-1	6.35E+2 3%	<2.E-1	<4.E0	<8.E0
02-919 AZ101P-F5	3.07E-5 15%	1.52E-2 3%	<7.E-4	2.39E-3 2%	<4.E-5	1.38E-1 3%	<4.E-5	<3.E-4	<3.E-4
02-920 AZ101P-F10	3.23E-5 12%	1.47E-2 3%	<5.E-4	2.36E-3 3%	<3.E-5	7.21E-2 4%	<3.E-5	<3.E-4	<4.E-4
02-921 AZ101P-F15	2.01E-5 9%	1.48E-2 3%	7.54E-4 24%	2.29E-3 2%	<9.E-6	4.99E-2 3%	<2.E-5	<2.E-4	<4.E-4
02-922 AZ101P-F20	1.72E-5 14%	1.48E-2 4%	7.78E-4 9%	2.43E-3 3%	<2.E-5	4.03E-2 4%	<2.E-5	<2.E-4	<1.E-4
02-923 AZ101P-F25	<2.E-5	1.47E-2 4%	<4.E-4	2.40E-3 3%	<3.E-5	3.30E-2 4%	<4.E-5	<3.E-4	<2.E-4
02-924 AZ101-FD-3	<2.E-5	1.48E-2 3%	<4.E-4	2.36E-3 2%	<3.E-5	2.85E-2 3%	<4.E-5	<3.E-4	<3.E-4
02-925 AZ101-FD-8	3.79E-5 7%	7.60E-4 4%	<2.E-4	1.17E-4 3%	<7.E-6	1.03E-2 3%	<2.E-5	<6.E-5	<2.E-4
02-926 AZ101-Fdi-3	2.94E-5 9%	1.12E-4 17%	<2.E-4	<6.E-5	<8.E-6	6.27E-3 3%	<2.E-5	<6.E-5	<9.E-5
02-927	2.72E-5	<7.E-5	<2.E-4	<3.E-5	<9.E-6	3.88E-3	<3.E-5	<6.E-5	<8.E-5

ALO ID Client ID	Co-60 Error %	Sb-125 Error %	RuRh-106 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
AZ101-Fdi-8	14%					4%			
02-928* AZ101L-E3	<8.E-1	<2.E+2	<2.E2	<3.E+1	1.59E+1 10%	3.98E+4 3%	<3.E0	<8.E1	<2.E2
02-929* AZ101L-E4	<2.E0	<3.E+2	<5.E2	<8.E+1	7.19E+1 10%	1.72E+5 3%	<2.E1	<3.E2	<5.E2
02-930* AZ101L-E5	<5.E-2	<9.E+1	<2.E2	<2.E+1	1.04E+1 12%	2.62E+4 3%	<3.E0	<7.E1	<2.E2
02-931* AZ101L-E6	<8.E-1	<3.E+1	<5.E1	<5.E+0	<2.E+0	3.23E+3 3%	<2.E0	<3.E1	<5.E1
02-932* AZ101L-E11	<2.E-3	<7.E-2	<2.E-1	<4.E-2	<4.E-3	1.47E+1 3%	<3.E-3	<4.E-2	<4.E-2
02-933 AZ101L-E15	<8.E-4	<2.E-2	<2.E-2	<3.E-3	<8.E-4	3.67E+0 4%	<8.E-4	<2.E-2	<5.E-2
02-934* AZ101-RGN	<2.E-3	<3.E-2	<4.E-2	<1.E-2	<2.E-3	3.99E+0 4%	<2.E-3	<2.E-2	<3.E-2
02-935* AZ101-FE COMP	<4.E-5	1.46E-2 4%	<1.E-3	2.36E-3 3%	<5.E-5	6.49E-2 4%	<6.E-5	<8.E-4	<2.E-3

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

File: 02-909
05/31/02

Client : Sandy Fiskum
ASR: 6306

Cognizant Scientist: L R Greenwood Date : 5/31/02

Concur : T Trang-Le Date : 6/3/02

Procedure: PNL-ALO-432 - Tc-99 (Pertechnetate)

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Pertechnetate Tc-99 Error %
02-0909 AZ101L-F0	3.44E-1 3%
02-935 AZ101-FE COMP	1.27E-2 3%
02-935 DUP AZ101-FE COMP	1.34E-2 3%
RPD	5%
Matrix Spike*	96%
Blank Spike	85%
Blank	2.90E-4 4%

*Performed on sample 02-0828 in the same batch.

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

10/11/02

Client : Fiskum
ASR: 6306.01

Cognizant Scientist: L L Greenwood Date : 10/11/02

Concur : T Trang-Le Date : 10/14/02

Procedure: PNL-ALO-417& 496 for Pu & Am

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %
02-909 PB Process Blank	<7.E-7	<1.E-6	<7.E-7
02-909 AZ101-F0	<1.E-3	<8.E-4	<7.E-4
02-935 AZ101-FE COMP	<5.E-5	<7.E-5	<6.E-5
Matrix Spike 3347 Matrix Spike 3250	108% 111%		
Reagent Spike	112%		
Lab Blank #1 Lab Blank #2	<5.E-8 <6.E-8	<5.E-8 <5.E-8	<5.E-8 <4.E-8

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Project No.

Internal Distribution
File/LB

Date March 11, 2002

To Sandy Fiskum

From Stan Bos Stan Bos

Subject Cesium isotopic analysis (RPL # 02-00909)

Corrected Report

Sandy

Cesium isotopic distribution analyses of samples AZ101 FO and AZ101 FO (dup) have been completed. Approximately five micro liters of the sample were plated on a rhenium carbide filament and analyzed on the single stage mass spectrometer (M&TE # WB76849) in accordance with PNNL technical procedure PNNL-98523-264. Since natural cesium is mono-isotopic, a sample of rubidium chloride isotopic standard (NBS 984) was run to standardize the mass spectrometer. Work package W60553 will be charged for the analysis.

Please feel free to call me at 376-5384 with any questions you might have.

Sample Id. AZ-101 FO RPL # 02-00909

	^{133}Cs	^{135}Cs	^{137}Cs
	Weight percent	Weight percent	Weight percent
AZ-101 FO	51.4± 0.5	16.9 ± 0.2	31.7 ± 0.2
AZ-101 FO (dup)	51.5 ± 0.5	17.1 ± 0.2	31.4 ± 0.2

Note: ^{134}Cs was not detected above the background

Concurrence Pamela K Brown 2-22-02

E54-1900-001 (8/98)

ASR 6360

GEA

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

02/07/02

Client : Sandy Fiskum
ASR: 6360

Cognizant Scientist: LR Greenwood Date : 2/7/02

Concur : T Trang-Le Date : 2/11/02

Reference Date: Feb. 6, 2002 for Gea
Procedure: PNL-ALO-450 for GEA

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	RuRh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %
02-1390 RZ101L-F7A	8.36E-4 14%	1.51E-2 3%	2.32E-3 2%	<3.E-5	3.68E-2 3%
02-1391 RZ101L-F18A	<3.E-3	1.51E-2 4%	2.40E-3 7%	2.53E-4 9%	6.43E-1 3%
02-1392 RZ101L-F19A	<6.E-3	1.35E-2 7%	2.48E-3 11%	<2.E-4	2.89E+0 3%
02-1396 RZ101P-F7A	<3.E-3	1.59E-2 5%	2.23E-3 8%	3.49E-4 9%	8.90E-1 3%
02-1397 RZ101P-F14A	<3.E-3	1.49E-2 4%	2.45E-3 7%	<8.E-5	6.25E-1 3%
02-1398 RZ101P-F21A	<2.E-3	1.58E-2 3%	2.48E-3 2%	<7.E-5	3.76E-1 3%
02-1399 RZ101FEComp2A	<3.E-3	1.48E-2 5%	2.48E-3 4%	<1.E-4	8.13E-1 3%
02-1400 RZ101FEComp3A	<2.E-3	1.54E-2 4%	2.45E-3 3%	<8.E-5	4.00E-1 3%

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

2/13/02

Client : Sandy Fiskum
ASR: 6360

Cognizant Scientist: L R Greenwood Date : 2/13/02

Concur : T Trang-Le Date : 2/13/02

Reference Date: Feb. 12, 2001 for Gea
Procedure: PNL-ALO-450 for GEA

Measured Activities (uCi/ml) with 1-sigma error

ALO ID Client ID	Cs-134 Error %	Cs-137 Error %
02-1393 RZ101L-F20	<6.E-3	1.16E+1 3%
02-1394 RZ101L-F21	<2.E-2	3.93E+1 3%
02-1395 RZ101L-F22	<2.E-2	1.19E+2 3%
02-1401 RZ101L-E3	4.47E+0 7%	1.18E+4 4%
02-1402 RZ101L-E4	<2.E1	1.44E+5 4%
02-1403 RZ101L-E5	<7.E0	4.17E+4 3%
02-1404 RZ101L-E6	1.04E+0 8%	2.84E+3 4%
02-1405 RZ101L-E7	<9.E-2	5.20E+2 3%
02-1406 RZ101L-E10	<3.E-2	2.65E+1 3%
02-1407 RZ101L-E14	<2.E-3	5.61E+0 3%
02-1408 RZ101 F0	5.26E-1 8%	1.59E+3 4%

ASR 6401

ICP-AES
U (KPA)
IC
TOC Hot Persulfate Method
TOC Furnace Oxidation Method
GEA, ^{236}Pu, ^{238}Pu, $^{239+240}Pu$, ^{241}Am, $^{243+244}Cm$, ^{242}Cm, Sum of Alpha

Project / WP#: 42365 / W60553
ASR#: 6401
Client: S. Fiskum
Total Samples: 1 (liquid)

	First	Last
RPL#:	02-02238	–
Client ID:	"AZ101-Cs Eluate-Comp"	–
Sample Preparation: Sample dilution (SAL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 06-20-2002 (A0826)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

7/12/02
Reviewed by

7/2/02
Concur

One AZ101 sample submitted under Analytical Service Request (ASR) 6401was analyzed in duplicate by ICPAES. The sample was client prepared with subsequent acid dilution being performed in the Shielded Analytical Laboratory (SAL). Dilution involved a nominal 0.2 mL of sample diluted to a final volume of approximately 20 mL.

Analytes of interest (AOIs) were specified in the ASR and included Cr, K, Na, Ni, and U. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those identified AOIs are reported, but have not been fully evaluated for QC performance.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (2 pages). The results are reported as µg/mL for each detected analyte, and have been adjusted for the SAL dilution factor. Minimum Reportable Quantity (MRQ) values were specified in the ASR for each AOI. Method Detection Limits (MDL) for the ICPAES instrument are nominally specified at three times the Instrument Detection Limits (IDL). Using this criteria, the required MRQ levels were met for all AOIs except K and U.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the dilution processing, a diluent blank and blank spike were prepared along with the sample. The blank spike was prepared using a nominal 1.2 mL of multi-element spike BPNL-QC-1A. The spike solution included all specified AOIs.

Process Blank:

A process (diluent) blank was prepared with the sample. The concentrations of all AOIs were within the acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike/LCS:

A blank spike, with reagents and spike solution, was prepared with the sample. All AOIs showed recoveries within the acceptance criteria of 80% to 120%.

Matrix Spiked Sample:

No matrix spike sample was provided for analysis.

Duplicate RPD (Relative Percent Difference):

A duplicate sample was prepared with the sample. The RPDs for all AOIs measured above the EQL were within the acceptance criteria of ±15% (±3.5% for Na).

Post-Spiked Samples (Spike A Elements):

All AOIs contained in the group A elements were measured above the EQL, and were recovered within the acceptance criteria of 75% to 125%.

Post-Spiked Samples (Spike B Elements):

Uranium was the only AOI contained in the group B elements. Uranium was recovered within the acceptance criteria of 75% to 125%, but at a level below the EQL.

Serial dilution (Relative Percent Difference):

Five-fold serial dilution was performed on the sample. The results for all AOIs measured above the EQL were within the acceptance criteria of ±10%.

Other QC:

All required instrument-related QC tests for the analytes of interest passed within the acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

	Run Date=	6/20/2002	6/20/2002	6/20/2002
	Multiplier=	102.2	103.2	103.2
	RPL/LAB #=	02-02238-DB	02-02238	02-02238-DUP
Det. Limit	Client ID=	*diluant blank*	*AZ101-Cs Eluate-Comp*	*AZ101-Cs Eluate-Comp-Dup*
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)
0.020	Cr	--	52.0	51.7
2.000	K	--	[210]	[230]
0.150	Na	[18]	1,040	1,040
0.030	Ni	--	--	--
2.000	U	--	[300]	[310]
Other Analytes				
0.025	Ag	--	--	--
0.060	Al	--	[10]	[30]
0.250	As	--	--	--
0.050	B	--	[22]	[22]
0.010	Ba	[4.2]	[3.7]	[3.6]
0.010	Be	--	--	--
0.100	Bi	--	--	--
0.250	Ca	--	[32]	[31]
0.015	Cd	--	[2.7]	[2.8]
0.200	Ce	--	--	--
0.050	Co	--	--	--
0.025	Cu	--	[3.0]	[3.0]
0.050	Dy	--	--	--
0.100	Eu	--	--	--
0.025	Fe	--	[8.7]	[8.8]
0.050	La	--	--	--
0.030	Li	--	--	--
0.100	Mg	--	--	--
0.050	Mn	--	--	--
0.050	Mo	--	--	--
0.100	Nd	--	--	--
0.100	P	--	--	--
0.100	Pb	--	--	--
0.750	Pd	--	--	--
0.300	Rh	--	--	--
1.100	Ru	--	--	--
0.500	Sb	--	--	--
0.250	Se	--	--	--
0.500	Si	--	--	--
0.500	Sn	--	--	--
0.015	Sr	--	--	--
0.500	Te	--	--	--
1.000	Th	--	--	--
0.025	Ti	--	--	--
0.500	Tl	--	--	--
0.050	V	--	--	--
0.500	W	--	--	--
0.050	Y	--	--	--
0.050	Zn	[7.1]	[7.3]	[6.9]
0.050	Zr	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 6/20/02

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-02238 & 02-02238-D	LCS/BS	MS (none)	02-02238 + Post Spike A	02-02238 + Post Spike B	02-02238 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Cr	0.5	97		103		
K		98		104		
Na	0.4	98		99		2.2
Ni		97		102		
U		99			**104**	
Other Analytes						
Ag				104		
Al		96		100		
As				**105**		
B				103		
Ba		94		98		
Be		97		**103**		
Bi		98		101		
Ca		101		106		
Cd		100		104		
Ce		99			**106**	
Co				107		
Cu		95		102		
Dy					102	
Eu					103	
Fe		99		103		
La		99			103	
Li		98		100		
Mg		99		109		
Mn		101		**108**		
Mo				103		
Nd		99			105	
P				105		
Pb		99		111		
Pd					**105**	
Rh					**102**	
Ru						
Sb				**104**		
Se				**105**		
Si				111		
Sn					**111**	
Sr		97		101		
Te					**107**	
Th		99			**103**	
Ti				97		
Tl				**101**		
V		96		101		
W						
Y				103		
Zn		100		105		
Zr				101		

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

Battelle Pacific Northwest National Laboratory filename 02-2238
Radiochemical Chemical Science & Engineering -325 Building 6/12/2002

Client : S. Fiskum
ASR: 6401

Cognizant Scientist: C. Soderquist Date : 6-12-02

Concur : L. R. Greenwood Date : 6/12/02

Reference Date: June 4, 2002 for U KPA
Procedure: RPG-CMC-4014 Rev 1, *Uranium Analysis by Kinetic Phosphorescence*

RPL ID / Client ID	Measured Concentration (µg/ml) with 1-sigma error Uranium ± 1s	MDL
02-2238 AZ101-Cs Eluate-Comp	2.04E+2 4%	5E-2
Blank	1.97E-1 18%	
LCS	111%	
Matrix Spike 909	75% Matrix spike concentration was only 10% of sample uranium concentration.	

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology – IC Report
PO Box 999, Richland, Washington 99352

Project / Charge Code: 42365 / W60553
ASR Number: 6401
Client: S. Fiskum
Total Samples: 1

	First in Series	Last in Series
RPL Numbers	02-2238	
Client IDs	AZ-101 Cs Eluate Comp	

Analysis Procedure	PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Prep Procedure	None
Analyst	MJ Steele
Analysis Date	06/25/2002, 06/25/2002
Calibration Date	04/17/2002
Cal/Ver Stds Prep Date	04/11/2002
Excel Data File	ASR 6306 6347 6401 6403.xls
M&TE Numbers	IC system (WD25214)
	Balance (360-06-01-031)
All Analysis Records	Chemical Measurement Center 98620 RIDS IC System File

Prepared By 7/18/02 **Date**

Reviewed By 7/18/02 **Date**

Sample Results

RPL No.	Sample ID	Dil Fctr	F µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL (a)		0.13	0.13	0.25	0.13	0.25	0.25	0.25	0.25
	Dilution Blank	1	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	MRQ		n/a	10	n/a	n/a	3,000	n/a	n/a	n/a
	EQL (a)		13	13	26	130	2,600	26	26	26
02-2238	AZ-101 Cs Eluate Comp	103.83	< 13	120	< 26	< 130	33,500	68	230	150
02-2238 MS	AZ-101 Cs Eluate-Comp MS %Recovery		99	94	99	103	106	105	104	107
LCS062402	LCS/BS % Recovery		106	103	105	106	99	107	105	109
	RPD (b)		1	(c)	3	2	3	3	1	4

MRQ: minimum reportable quantity EQL: estimated quantitation limit RPD: relative percent difference

(a) EQL based on lowest calibration standard times dilution factors used to obtain sample results
(b) Batch duplicate RPD calculated from sample AZ-101 FE Comp (ASR 6306, RPL No. 02-0935)
(c) RPD not calculated unless both sample and duplicate results >EQL

Sample Analysis/Results Discussion

The AZ-101 Cs Eluate Comp sample was prediluted in the Shielded Analytical Laboratory (SAL) by approximately 100x. This samples required an additional 100x laboratory dilution in order to ensure that the anions were measured within the calibration range and that the IC column was not overloaded during the analysis. The minimum reportable quantities (MRQ) and the estimated quantitation levels (EQL) are provided; the MRQs are based on a table provided with ASR 6401 and the EQLs are based on the lowest calibration standard adjusted for the dilutions used for reporting the results.

Quality Control Discussion

Duplicate: No sample duplicate was provided by the client. An analysis batch laboratory duplicate was prepared using sample 'AZ-101 FE Comp' from ASR 6306 (02-0935). The laboratory duplicate relative percent difference (RPD) is well within the acceptance criterion of <20% (as defined by the laboratory QA plan) for all anions measured above the EQL.

Laboratory Control Sample/Blank Spike - (BS 062402 [HCV020411 @3x)]): The high range calibration verification standard diluted by a factor of three was used as the LCS/BS. The LCS/BS demonstrated recoveries within the 80% to 120% acceptance criteria for all anions measured.

Matrix Spike (CCV020411 @2x): A matrix spike (MS) was prepared using the mid-range calibration check standard and the sample. The MS recoveries were within the 75% to 125% recovery acceptance criteria for the anions of measured.

Process/Dilution Blank: A dilution blank (i.e., diluted eluant) was analyzed for all reported analytes and no anions were measured above the EQL.

IC System QC samples: No anions of interest were measured in the calibration blanks (ICB/CCB). The four calibration verification standards analyzed with the samples met the 90% to 110% recovery criteria for all anions measured.

Summary of Deviation: None

General Comments

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested. For routine analyses, no results are reported below the EQL.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference.

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology
TOC/TIC Report – Hot Persulfate Oxidation Method
PO Box 999, Richland, Washington 99352

Project Number:	42365
Charge Code:	W60553/W60555
ASR Number:	6374, 6401, 6403
Client:	S. Fiskum
Total Samples:	3 (liquids)

ASR	RPL Number	Client ID
6378 4	02-1698	AZ102C-CsE-Comp
6401	02-2238	AZ101-Cs Eluate-Comp
6403	02-2246	AZ101-Cs Eluate-Comp-HLW

Analysis Procedure	PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"
Prep Procedure	None
Analyst	D. Sanders and M. Steele
Analysis Date	07/16/2002
Cal/Verify Standards	TOC CMS-53219
LCS/MS Standards	TOC CMS-161713
Excel Data File	ASR 6374 6378 6401 6403 HP.xls
M&TE Numbers	Carbon System (WA92040)
	Balance (360-06-01-023)
All Analysis Records	Project File

MW Thurs — 8/1/02
Prepared By — **Date**

DR Johnson — 8-1-02
Reviewed By — **Date**

Carbon Results

RPL Number	Sample ID	Pre-Dilution	TOC MDL µgC/mL	TOC Results µgC/mL
	MRQ			1,500
ASR 6374				
02-01698	AZ-102C-CsE-Comp	103.83	2,500	< 2,500
ASR 6401				
02-02238	AZ-101-Cs-Eluate-Comp	103.83	1,900	5,100
ASR 6403				
02-02246	AZ-101-Cs-Eluate-Comp-HLW	103.83	3,700	20,500
02-02246 Dup	AZ-101-Cs-Eluate-Comp-HLW Dup	103.83	3,700	20,100
	RPD			2%
	%RSD			1%
02-02246 MS	Matrix Spike %Rec	103.83		116%
LCS/Blank Spike	LCS/BS %Rec	1.00		95%

Sample matrices are 0.5M nitric; no TIC analysis performed
TOC: total organic carbon MDL: method detection limit
RPD: relative percent difference RSD: relative standard deviation

Sample Analysis/Results Discussion

The TOC analyses of the three aqueous samples submitted under Analytical Service Requests (ASR) 6387, 6401, and 6403 are to be performed by both the hot persulfate and furnace oxidation methods. This report presents the results from the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition of any TIC and acidic potassium persulfate oxidation at 92-95°C for TOC.

The table above shows the results, rounded to two to three significant figures. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-381.

Quality Control Discussion

The calibration and QC standards for TOC analysis are solid pure chemicals from Aldrich, and Sigma (calcium carbonate for TC and α-D-glucose for TOC). The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The QC for the method involves calibration blanks, sample duplicates (laboratory), laboratory control sample/blank spikes (LCS/BS), and matrix spikes (MS). Each ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs. The ASRs also provide specific QC acceptance criteria for LCS/BS, MS, and duplicate RSD, as well as define the MRQ; the performance of the QC samples is compared to these criteria. Due to the large dilution performed in the Shielded Analytical Laboratory to reduce the dose of the samples, the MDL for the samples exceeded the MRQ. For those samples

with measurable TOC, the failure to meet the MRQ has little impact. However, for sample 'AZ-102C-CsE-Comp', the MDL exceeds the MRQ by nearly a factor of two.

The calibration of the coulometer analysis system is checked by analyzing calibration check standards at the beginning, middle, and end the analysis run. The average recovery from the calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average TOC calibration check standard recovery was 97%.

Laboratory Control Sample/Blank Spike: A LCS/BS was analyzed with the samples. At 95% TOC, the LCS/BS recovery is well within acceptance criterion of 80% to 120%.

Duplicate: The analysis batch duplicate analysis was performed on 'AZ101-Cs-Eluate-Comp-HLW'. The TOC results met the acceptance criterion established by the ASRs (i.e., <15% RSD), as well as acceptance criterion established by the Laboratory's QA Plan (i.e., <20% RPD)

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the MS. A MS was prepared from sample 'AZ101-Cs-Eluate-Comp-HLW' and an organic standard (see cover page for standard identification). Although somewhat higher than normal, the TOC MS recovery is within the acceptance criterion of 75% to 125% recovery.

Deviation from Procedure

None

General Comments

1) The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences.
3) The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results < 5xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated.
4) For both the TC and TOC, the analysis MDL is based on three times the standard deviation of a set of historical 'system blank' data. The sample MDL (in µgC/mL or µgC/g) are calculated by using the analysis MDL adjusted for the sample volume or mass.

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology
TOC/TIC Report – Furnace Oxidation Method
PO Box 999, Richland, Washington 99352

Project Number: 42365
Charge Code: W60555 | W60553 | W60555
ASR Number: 6374 | 6401 | 6403.01
Client: S. Fiskum
Total Samples: 3 (liquids)

	ASR 6374	ASR 6401	ASR 6403.01
RPL Numbers	02-1698	02-2238	02-2246
Client IDs	AZ102C-CsE-Comp	AZ101-Cs-Eluate-Comp	AZ101-Cs-Eluate-Comp-HLW

Analysis Procedure	PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"
Prep Procedure	None
Analyst	M. Steele
Analysis Date	08/20/2002 (TOC)
Cal/Verify Standards	TOC: CMS-53219
MS/LCS Standards	TOC: CMS-161713
Excel Data File	ASR 6374 6401 6403 700 Furn.xls
M&TE Numbers	Carbon System (WD13071)
	Balance (360-06-01-023)
All Analysis Records	Project File

Prepared By — **Date** 8/21/02

Reviewed By — **Date** 8-26-02

Carbon Results

RPL Number	Sample ID	Hot Cell Dilution	TOC MDL µgC/mL	TOC Results µgC/mL	TOC RPD
Hot Cell Diluent	2% Nitric Acid	1	26	30	
@104x Equivalent		104	2,700	3,100	
	MRQ			1,500[(a)]	
ASR 6374					
02-1698	AZ102C-CsE-Comp	104	1,800	6,300	
ASR 6403					
02-2246	AZ101-CsEluate-Comp-HLW	104	2,700	3,300	
ASR 6401					
02-2238	AZ101-CsEluate-Comp	104	2,700	2,900	
02-2238 Dup	AZ101-CsEluate-Comp Dup	104	2,700	< 2,700	N/A
02-2238 MS	MS Recovery			103%	
Blank Spike/LCS-1	LCS Recovery			103%	
Blank Spike/LCS-2	LCS Recovery			98%	

TOC: total organic carbon MDL: method detection limit
RPD: relative percent difference MRQ: minimum reportable quantity
N/A: not applicable; RPD not calculated unless sample and duplicate >5xMDL
(a) Due to the high dilutions necessary in the SAL hot cells to reduce the dose of the samples, the TOC MDL higher (about 2x) than the TOC MRQ.

Sample Analysis/Results Discussion

The TOC analyses of the samples submitted under ASR 6374, 6401, and 6403 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 700 °C for 10 minutes. No total inorganic carbon (TIC) was determined, since the samples are 0.5 M nitric acid.

The table above shows the results. The raw data bench sheets and calculation work sheets showing all calculations are attached. All sample results are corrected for average percent recovery of system calibration standards and are also corrected for contribution from the blank, as per procedure PNL-ALO-380.

Quality Control Discussion

The calibration and QC standards for TOC analysis are solid pure α-D-glucose from Aldrich and Sigma. The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets for traceability.

The QC for the method involves calibration blanks, sample duplicates (laboratory), laboratory control sample/blank spike (LCS/BS), and matrix spike (MS). The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of

Regulatory Programs". The ASRs provide QC acceptance criteria for LCS/BS, MS, and precision (RPD); the performance of the QC samples is compared to these criteria.

The calibration of the coulometer analysis system is checked by analyzing calibration check standards at the beginning, middle, and end of each day's run. The samples were analyzed for TOC as a batch. The average recovery from the calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for the TOC was 99%.

Analysis Run 08/20/2002: TOC QC

The TOC results for all the samples are less than 10 times the MDL and have a very high uncertainty (estimated at up to ±100%). The high dilutions from the SAL hot cells results in MDLs greater than the MRQ.

Laboratory Control Sample/Blank Spike: Two TOC LCS/BS (organic standards) were analyzed with samples. At 103% and 98% recovery, the LCS/BS recoveries are well within acceptance criterion of 80% to 120%.

Duplicate: A duplicate was prepared in the laboratory from sample 02-2238 (AZ101-CS-Eluate-Comp). The TOC RPD was not calculated since the sample and duplicate results were less than five times the MDL.

Matrix Spike: A MS was prepared from sample 02-2238 (AZ101-CS- Eluate-Comp) by adding a know quantity of an organic standard. The TOC MS recovery of 103% is well within the acceptance criterion of 75% to 125% recovery.

Deviations from Procedure

None

Comparison: Furnace Oxidation and Hot Persulfate Oxidation Results

RPL Number	Sample ID	Hot Cell Dilution	TOC-F MDL ugC/mL	TOC-F Results ugC/mL	TOC-HP MDL ugC/mL	TOC-HP Results ugC/mL
Diluent	2% Nitric Acid	104	2,700	3,100		NM
ASR 6374						
02-1698	AZ102C-CsE-Comp	104	1,800	6,300	2,500	< 2,500
ASR 6401						
02-2238	AZ101-Cs-Eluate-Comp	104	2,700	2,900	1,900	5,100
ASR 6403						
02-2246	AZ101-Cs-Eluate-Comp-HLW	104	2,700	3,300	3,700	20,500

HP= Hot Persulfate Method F= Furnace Combustion Method

The quantity of carbon measured for most samples was within three times the MDL for both the hot persulfate and furnace method. This low TOC concentration and the high dilution from the SAL hot cells make accurate quantitation and comparison of the TOC result impossible. All results except the hot persulfate results for 02-2246 (AZ101-Cs-Eluate-Comp-HLW) are essentially the same result within the level of measurement uncertainty. Sample 02-2446 measure by hot persulfate method produced a measurable carbon values about five times the

MDL. Carbon results less than 10 times the MDL are considered semi-quantitative due to the high measurement uncertainties, primarily due to the variability in the system blank levels.

General Comments

1) The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
2) Routine precision and bias are typically ±15% or better for non-complex samples that are free of interferences and have a concentration exceeding 10 times the MDL
3) The estimated quantitation limit (EQL) is defined as 10 times the MDL. Results <10xMDL have higher uncertainties, and RPDs (or RSDs, if applicable) are not calculated if the results are <5xMDL.
4) For the TOC, the analysis MDL is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's t values for n-1 degrees of freedom to establish the daily analysis MDL. The sample MDL (in µgC/ml or µgC/g) are calculated by using the analysis MDL adjusted for the sample volume or mass.

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

06/13/02

Client : S. Fiskum
ASR: 6401

Cognizant Scientist: L R Greenwood Date : 6/13/02

Concur : T Trang-Le Date : 6/13/02

Reference Date: June 4, 2002 for GEA
Procedure: PNL-ALO-450 for GEA
Procedure: PNL-ALO-417 & 496 for Pu & Am

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	Cs-134 Error %	Cs-137 Error %	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	(Am-242m Cm-242** Error %	Sum of Total Alpha Error +/-
02-2238 AZ101-Cs Eluate-Comp	5.78E+0 6%	1.60E+4 3%	1.43E-2 3%	1.50E-3 4%	<2.E-5	4.37E-4 4%	2.57E-5 14%	<3.E-6	1.63E-2 3%
02-2239 RZ101-Cs-Eluate-Comp	4.09E+0 8%	1.20E+4 2%							
Matrix Spike			104%			98%			
Reagent Spike			103%			100%			
Blank			<3.E-6	<2.E-6	<2.E-6	1.06E-5 23%	<4.E-6	<4.E-6	

** Cm-242 is the daughter of Am-242m (141y). The activity of Am-242m is a factor of 1.21 times higher than that of Cm-242.

ASR 6403

ICP-AES
U (KPA)
ICP-MS
IC
NH_3
TOC Hot Persulfate Method (reported with ASR 6401)
TOC Furnace Oxidation Method (reported with ASR 6401)
Hg
GEA
Cs isotopic (TIMS)
^{3}H
^{14}C
^{63}Ni
^{79}Se
^{151}Sm
Total Beta, ^{90}Sr, Total Alpha, ^{236}Pu, ^{238}Pu, $^{239+240}Pu$, ^{241}Am, $^{243+244}Cm$, ^{242}Cm, Sum of Alpha
^{241}Pu
Weight Percent Solids and Weight Percent Oxides
Chelators
Organic Anions

Project / WP#: 42365 / W60650 & W60655
ASR#: 6403
Client: S. Fiskum
Total Samples: 1 (liquid)
REVISION 1: Expanded Discussion of Th LCS Failure

	First	Last
RPL#:	02-02246	--
Client ID:	"AZ101-Cs-Eluate"	--
Sample Preparation: Sample dilution (SAL)		

Procedure: PNNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).

Analyst: D.R. Sanders

Analysis Date (File): 06-17-2002 (A0824)

See Chemical Measurement Center 98620 file: ICP-325-405-1
(Calibration and Maintenance Records)

M&TE Number: WB73520 (ICPAES instrument)
360-06-01-029 (Mettler AT400 Balance)

B.M. Dunn 10/14/02
Prepared by

M W Urie 10-14-02
Reviewed by

One AZ101 sample submitted under Analytical Service Request (ASR) 6403 was analyzed in duplicate by ICPAES. The sample was client prepared with subsequent acid dilution being performed in the Shielded Analytical Laboratory (SAL). Dilution involved a nominal 0.2 mL of sample diluted to a final volume of approximately 20 mL.

Analytes of interest (AOIs) were specified in the ASR and included forty of the standard forty-five analytes available for the ICP analyses. Analytes not included as AOIs were Pd, Rh, Ru, Tl, and W. The quality control (QC) results for each of the AOIs have been evaluated and are presented below. Analytes other than those identified as AOIs are reported, but have not been fully evaluated for QC performance.

A summary of the ICPAES analyses, including QC performance, is given in the attached ICPAES Data Report (2 pages). The results are reported as µg/mL for each detected analyte, and have been adjusted for the SAL dilution factor. Minimum Reportable Quantity (MRQ) values were specified in the ASR for a number of the AOIs. Method Detection Limits (MDL) for the ICPAES instrument are nominally specified at three times the Instrument Detection Limits (IDL). Using this criteria, the required MRQ levels were met for all specified AOIs except Ba, K, Sn, and U. Potassium, tin, and uranium, however, were not detected in the sample above the IDL. Barium was detected, but at a level below the instrument EQL.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. For the dilution processing, a diluent blank and blank spike were prepared along with the sample. The blank spike was prepared using a nominal 1.2 mL of multi-element spikes BPNL-QC-1A, -2A, and -3. The spike solution included all specified AOIs except for As, Co, Dy, Eu, Sb, Se, Sn, Te, and Y.

Process Blank:

A process (diluent) blank was prepared with the sample. The concentrations of all AOIs were within the acceptance criteria of ≤EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike/LCS:

A blank spike, with reagents and spike solution, was prepared with the sample. Except for thorium, all AOIs included in the spike showed recoveries within the acceptance criteria of 80% to 120%. Thorium was recovered at a level of only 40%. Thorium was, however, recovered in the post-spike sample (discussed below), although at a level below the EQL. The low thorium recovery appears to be due to combining multi-element spikes BPNL-QC-1A and BPNL-QC-2A. Since BPNL-QC-2A contains about 0.7% HF (to maintain the Si, W, and Zr in solution), thorium is most likely lost as precipitation as thorium fluoride. It is unlikely that thorium behaves in the same manner in the AZ-101-Cs-Eluate sample, due to the absence of any significant quantity of fluoride. Additional justification for accepting the sample thorium results is that an LCS containing only the BPNL-QC-1A spiking solution (i.e., no HF), which was prepared and analyzed at the same time to support the analysis of the AZ102C-Cs-E-Comp sample (02-01698), produced excellent thorium recovery (i.e., 99%).

Matrix Spiked Sample:

No matrix spike sample was provided for analysis.

Duplicate RPD (Relative Percent Difference):

A duplicate sample was prepared for analysis. The RPDs for all AOIs measured above the EQL were within the acceptance criteria of ±15% (±3.5% for Na).

Post-Spiked Samples (Spike A Elements):

All AOIs contained in the group A elements measured above the EQL were recovered within the acceptance criteria of 75% to 125%. The AOIs As, Be, Mn, Sb, and Se were recovered but at levels below the EQL.

Post-Spiked Samples (Spike B Elements):

All AOIs contained in the group B elements measured above the EQL were recovered within the acceptance criteria of 75% to 125%. The AOIs Ce, Sn, Te, Th, and U were recovered but at levels below the EQL.

Serial dilution (Relative Percent Difference):

Five-fold serial dilution was performed on the sample. The results for all AOIs measured above the EQL were within the acceptance criteria of ±10%.

Other QC:

All required instrument-related QC tests for the analytes of interest passed within the acceptance criteria.

Comments:

1) "Final Results" have been corrected for all laboratory dilutions performed on the samples during processing and analysis unless specifically noted.
2) Detection limits (Det. Limit) shown are for acidified water. Detection limits for other matrices may be determined if requested. Method detection limits (MDL) can be estimated by multiplying the 'Multiplier' times the Detection Limit.
3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight). Note that bracketed values listed in the data report are within ten times instrument detection limit (adjusted for processing factors and laboratory dilutions) and have a potential uncertainty much greater than 15%.
4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.
5) The maximum number of significant figures for all ICP measurements is 2.

	Run Date=	6/17/2002	6/17/2002	6/17/2002
	Multiplier=	103.2	103.2	103.2
	RPL/LAB #=	02-02246-DB	02-02246	02-02246-DUP
Det. Limit	Client ID=	*diluant blank*	*AZ101-Cs Eluate*	*AZ101-Cs Eluate-Dup*
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--
0.060	Al	--	--	--
0.250	As	--	--	--
0.050	B	--	[9.5]	[9.4]
0.010	Ba	--	[2.6]	[2.4]
0.010	Be	--	--	--
0.100	Bi	--	--	--
0.250	Ca	--	[27]	[27]
0.015	Cd	--	[2.0]	[1.9]
0.200	Ce	--	--	--
0.050	Co	--	--	--
0.020	Cr	--	32.7	32.6
0.025	Cu	--	--	--
0.050	Dy	--	--	--
0.100	Eu	--	--	--
0.025	Fe	--	[6.9]	[6.7]
2.000	K	--	--	--
0.050	La	--	--	--
0.030	Li	--	--	--
0.100	Mg	--	--	--
0.050	Mn	--	--	--
0.050	Mo	--	--	--
0.150	Na	--	810	796
0.100	Nd	--	--	--
0.030	Ni	--	--	--
0.100	P	--	--	--
0.100	Pb	--	--	--
0.500	Sb	--	--	--
0.250	Se	--	--	--
0.500	Si	--	--	--
1.500	Sn	--	--	--
0.015	Sr	--	--	--
1.500	Te	--	--	--
1.000	Th	--	--	--
0.025	Ti	--	--	--
2.000	U	--	--	--
0.050	V	--	--	--
0.050	Y	--	--	--
0.050	Zn	[6.5]	[7.4]	[6.3]
0.050	Zr	--	--	--
Other Analytes				
0.750	Pd	--	--	--
0.300	Rh	--	--	--
1.100	Ru	--	--	--
0.500	Tl	--	--	--
0.500	W	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.

2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.

3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance 6/17/02

Criteria>	<15%[a]	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-02246 & 02-02246-D	02-02246 - LCS/BS	MS (none)	02-02246 + Post Spike A	02-02246 + Post Spike B	02-02246 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Ag		105		105		
Al		99		103		
As				**110**		
B		101		106		
Ba		100		104		
Be		96		**106**		
Bi		104		106		
Ca		101		107		
Cd		101		105		
Ce		99			**102**	
Co				109		
Cr	0.0	101		106		
Cu		101		109		
Dy					103	
Eu					103	
Fe		103		107		
K		104		111		
La		101			103	
Li		104		108		
Mg		103		113		
Mn		102		**109**		
Mo		100		106		
Na	1.8	105		109		-2.7
Nd		101			103	
Ni		100		108		
P		101		103		
Pb		102		109		
Sb				**106**		
Se				**111**		
Si		104		113		
Sn					**102**	
Sr		101		107		
Te					**107**	
Th		**40**			**104**	
Ti		98		102		
U		100			**108**	
V		96		102		
Y				103		
Zn		104		110		
Zr		100		106		
Other Analytes						
Pd					**102**	
Rh					**105**	
Ru						
Tl				**108**		
W		**99**				

Shaded results exceed acceptance criteria

Bold results for information only - spiked concentration less than EQL

nr = not recovered; spike concentration less than 20% of sample concentration.

(a) = RPD <3.5% for Na (only)

Battelle
Richland, WA
Radiochemical Science and Engineering, 325 Building

filename 02-2246
06/12/02

Client: S. Fiskum
ASR 6403

Prepared by: C. Soderquist 6-12-02

Concur: L R Greenwood 6/12/02

Procedure RPG-CMC-4014 Rev 1, *Uranium Analysis by Kinetic Phosphorescence*
Analysis Date: June 4, 2002

Sample	Lab ID	Measured Uranium Concentration, µg/mL ± 1s		MDL
AZ-101 Cs Eluate	02-2246	**1.59E+2**	**± 4%**	5E-2
	LCS	111%		
	Blank	1.97E-1	± 18%	
	Matrix Spike 909	75% Matrix spike concentration was only 10% of sample uranium concentration		

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60654
ASR#:	6403
Client:	S.K. Fiskum
Total Samples:	1

Analysis: Multi-Elemental

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:

R102924	PQ-Excell, Thermal Elemental
512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 18 August 2002

Analysis Files: 12 Aug 2002, 13 Aug 2002

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): Hans Carman / Orville Thomas Farmer III

Reviewed By — Date 11/1/02

Concur — Date 11/1/02

Sample Solutions Submitted for Analysis:

RPL #	Client I.D.
02-02246-B	Process Blank
02-02246	AZ-101 Cs Eluate Comp-HLW
02-02246-DUP	AZ-101 Cs Eluate Comp-HLW-DUP
02-02246-BS2	ICP/MS Blank Spike
02-02246-MS2	AZ-101 Cs Eluate Comp-HLW ICP/MS Matrix Spike

One (1) sample (AZ-101 Cs Eluate Comp-HLW) prepared using PNL-ALO-128 was submitted for analysis and analyzed per 329-OP-SC01 on a radioactive-material-contained ICP/MS for all requested analyte(s).

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis. Results are reported in mg/L for analytes of interest.

Final calculated results for elemental concentration are assumed to have natural isotopic composition, however this is not the case for some analytes of interest such as (Pd, Ru). The results reported for these elements, should be considered to have bias with respect to their altered isotopic composition produced mainly from U-235 fission yield production. Table 1, provides an example of the isotopic production of fission yield element production from U-235 burn-up.

TABLE 1

Isotope	Natural Abundance (%)	U-235 Fission Yield (%)	Altered Isotopic Abundance (%)	Affect on final Result
Ru-98	1.90			
Ru-99	12.7	<0.1		
Ru-100	12.6			
Ru-101	17.0	5.20	45.7	Bias high
Ru-102	31.6	4.30	37.8	Bias high
Ru-104	18.7	1.88	16.5	Bias Low
Pd-102	1.00			
Pd-104	11.1			
Pd-105	22.3	0.960	60.5	Bias high
Pd-106	27.3	0.402	25.3	Bias Low
Pd-107		0.146	9.20	
Pd-108	26.5	0.054	3.40	Bias Low
Pd-110	11.7	0.025	1.60	Bias Low

For this analysis, Pd-105 was used to calculate the total elemental concentration of Pd. The above table suggests that the actual reported values are biased high for Pd by a factor of 2.71. Ru-101 and Ru-102 were both measured to better evaluate the total Ru concentration. The calculated difference for total Ru using Ru-101 and Ru-102 was determined to be 2.05, which demonstrates that the Ru isotopic concentration in the sample is from fission production. Elemental concentration determination of fission products by ICP/MS requires that the elemental isotopic distribution of the sample be know, otherwise chemical separation is required prior to

analysis. No chemical separation was performed for either of these elements prior to analysis by ICP/MS.

The preparative batch QC for ^{99}Tc (and all radionuclides) included a preparation blank (PB) and sample duplicate. Because of the high activity in the samples, large dilutions were required at the analytical workstation. Therefore ^{99}Tc (and all radionuclides) were not spiked into a separate BS/LCS or MS. Matrix effects for radionuclides were evaluated instead on post-spike recoveries at the analytical workstation, as is typical with radioisotope measurements. Stable elements such as Ru, Rh, Pd, Pr, etc. were spiked in the MS and BS/LCS. A limited evaluation of matrix effects on radioisotope analysis could be made with respect to stable element recoveries.

The analytical result for AMU-241 was determined using the standard response of an Am-241 standard solution. The Pu-242 sample concentration was calculated using the Pu-239 standard response curve. All minor isotope concentrations of uranium (U-233, U-234, U-235 and U-236) were calculated using the standard response of U-238.

The analytical results in the tables below show the isotope used for elemental analysis. It assumes naturally occurring isotopic abundances for the elements analyzed. For the radioactive isotopes (T-99, I-129, Np-237, U-233, U-234, U-235, U-236, U-238, Pu-239, Pu-240, Amu-241 and Pu-242) no assumption is make about isotopic abundance, the results are a direct determination of that isotopes concentration in the sample. No chemical separations were performed, for any analysis on ASR-6403.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions, which were evaluated, at the beginning of the analytical run. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and the sample total dilution factor.

Instrument QCs

Instrument QC checks are used to assure that the instrument is stable through out the analysis and are reported for information only. The Initial and Continuing Control Blanks (ICB,CCB) monitor instrument backgrounds, the Initial and Continuing Calibration Verification Standards (ICV and CCV) check to assure instrument responses are constant through out the analysis and Interference Correction Standard and Interference Correction Standard Duplicate (ICS and ICSD) and Serial Dilution (DT) are used to evaluate potential analyte interferences. All ICB,CCB, ICV and CCVs met their respective QC success criteria.

2. Final Results

2.1 ALO-128 Results

Sample ID	Analyte 99Tc		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			5.01E-04	8.50E-0	2.52E-07	1	100
ICB		<	2.52E-06		2.52E-07	1	
02-02246-B			2.08E-02	4.66E-03	7.31E-04	2902	
DT (02-02246)			1.31E-01	7.64E-03	3.41E-03	13527	N.A.
ICS (02-02246)			2.21E-02	1.15E-03	6.82E-04	2705	
ICSD (02-02246)			2.02E-02	6.65E-04	6.82E-04	2705	9.0
ICSDS (02-02246)			1.36E+00	5.59E-03	6.82E-04	2705	99
02-02246-PBS			1.47E+00	4.80E-04	7.31E-04	2902	100
02-02246			1.96E-02	9.92E-04	6.82E-04	2705	
02-02246-DUP			1.72E-02	6.36E-04	7.00E-04	2776	12.4
02-02246-BS2			1.12E-01	3.24E-04	6.70E-04	2657	N.S.
02-02246-MS2			1.26E-01	7.06E-04	6.72E-04	2665	N.S.
CCV-1			5.02E-04	2.38E-06	2.52E-07	1	100
CCB-1		<	2.52E-06		2.52E-07	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked with ^{99}Tc, instrument response above MDL is due to natural Ru present in the multi-element spiking solution.

Narrative:

The ^{99}Tc concentration in the PB was equivalent to the activity found in the sample and duplicate, and was well above the instrument detection limit. The level of ^{99}Tc contamination present in the blank relative to the ^{99}Tc concentration present in the sample did not meet the QC acceptance criteria for blanks (i.e. <5% of sample concentration). The hot cell is a difficult environment in which to work; samples prepared in the hot cells have typically been returned to the ICP-MS workstation with detectable quantities of ^{99}Tc from low-level contamination. This contamination occurs during sample preparation in cell and cannot be completely eliminated. Using the hot cells for sample preparation more than likely resulted in an achievable method detection limit of 2E-2 mg/L. The ^{99}Tc concentration in these samples, therefore, can be reported as < 2E-2 mg/L for this run.

The post matrix spike (PMS) and post blank spike (PBS) recoveries were well within the acceptance criteria of 80-120%. The responses shown for the BS and MS were due to ^{99}Ru (^{99}Ru is indistinguishable from ^{99}Tc using ICP-MS) that was present in the multi-element spiking solution.

A Dilution test, two (2) sample replicates (ICS and ICSD) were also analyzed with the submitted samples. This test did not meet the QC success criteria of 90 to 110% (or <10% difference). However, since the sample concentration for the serial dilution (DT) was not above 50 X MDL this test is not a valid QC test. The response for the BS and MS are due to Ru-99 that was present in the multi-element spiking solution.

Sample ID	Analyte 101Ru		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.98E-04	3.52E-07	2.44E-07	1	100
ICB		<	2.44E-06		2.44E-07	1	
02-02246-B		<	7.08E-03		7.08E-04	2902	
DT (02-02246)			5.64E-02	4.15E-03	3.30E-03	13527	100
ICS (02-02246)			5.65E-02	1.04E-03	6.60E-04	2705	
ICSD (02-02246)			5.65E-02	4.17E-04	6.60E-04	2705	0.1
ICSDS (02-02246)			1.39E+00	5.05E-03	6.60E-04	2705	99
02-02246-PBS			1.46E+00	8.65E-03	7.08E-04	2902	100
02-02246			5.64E-02	1.29E-03	6.60E-04	2705	
02-02246-DUP			5.45E-02	2.78E-04	6.77E-04	2776	3.5
02-02246-BS2			9.73E-01	2.58E-03	6.48E-04	2657	100
02-02246-MS2			1.05E+00	5.56E-03	6.50E-04	2665	102
CCV-1			5.00E-04	2.60E-06	2.44E-07	1	100
CCB-1		<	2.44E-06		2.44E-07	1	

Narrative:

All batch and instrument QC solution met their respective success criteria.

Sample ID	Analyte 102Ru		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV	25733		4.98E-04	2.47E-06	2.78E-07	1	100
ICB	6	<	2.78E-06		2.78E-07	1	
02-02246-B	24	<	8.08E-03		8.08E-04	2902	
DT (02-02246)	112		2.93E-01	6.40E-04	3.77E-03	13527	107
ICS (02-02246)	527		2.76E-02	8.69E-05	7.53E-04	2705	
ICSD (02-02246)	516		2.70E-02	6.06E-04	7.53E-04	2705	2.1
ICSDS (02-02246)	26201		1.37E+00	4.07E-03	7.53E-04	2705	99
02-02246-PBS	26019		1.46E+00	3.58E-03	8.08E-04	2902	101
02-02246	523		2.74E-02	2.54E-04	7.53E-04	2705	
02-02246-DUP	484		2.60E-02	2.51E-04	7.73E-04	2776	5.0
02-02246-BS2	19038		9.79E-01	8.56E-04	7.40E-04	2657	101
02-02246-MS2	19913		1.03E+00	7.83E-03	7.42E-04	2665	103
CCV-1	25887		5.01E-04	1.64E-06	2.78E-07	1	100
CCB-1	38	<	2.78E-06		2.78E-07	1	

Narrative:

All batch and instrument QC solution met their respective success criteria.

NOTE: The data for ^{101}Ru and ^{102}Ru are not identical because this analyte is produced by fission. The actual value assuming 235U fission yields shown in Table 1 would be 0.020 mg/L total Ru. This value can be calculated by taking the ratios difference from natural to fission yield (e.g. 0.37x 101Ru = 0.055 mg/L or by 0.84 x ^{102}Ru = 0.027 mg/L). Another way to calculate total Ru total by calculating total Ru without natural abundance considerations however without 104Ru considered the sum result will be low about 10%.

Sample ID	Analyte 103Rh		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			5.00E-04	1.54E-06	2.88E-06	1	100
ICB		<	2.88E-05		2.88E-06	1	
02-02246-B		<	8.36E-02		8.36E-03	2902	
DT (02-02246)		<	1.68E-02		3.90E-02	13527	95
ICS (02-02246)		<	7.79E-02		7.79E-03	2705	
ICSD (02-02246)		<	7.79E-02		7.79E-03	2705	N.A.
ICSDS (02-02246)			1.36E+00	7.55E-03	7.79E-03	2705	99
02-02246-PBS			1.46E+00	1.28E-03	8.36E-03	2902	100
02-02246		<	7.79E-02		7.79E-03	2705	
02-02246-DUP		<	7.99E-02		7.99E-03	2776	3.0
02-02246-BS2			9.68E-01	1.47E-03	7.65E-03	2657	100
02-02246-MS2			1.01E+00	3.35E-03	7.67E-03	2665	102
CCV-1			5.02E-04	1.47E-06	2.88E-06	1	100
CCB-1		<	2.88E-05		2.88E-06	1	

N.A. = not applicable

Narrative:

All batch and instrument QC solution met their respective success criteria. No Minimum Reportable Quantity was requested.

Sample ID	Analyte 105Pd		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.91E-04	1.69E-06	2.64E-06	1	98
ICB		<	2.64E-05		2.64E-06	1	
02-02246-B		<	7.66E-02		7.66E-03	2902	
DT (02-02246)		<	3.57E-01		3.57E-02	13527	N.A.
ICS (02-02246)		<	7.14E-02		7.14E-03	2705	
ICSD (02-02246)		<	7.14E-02		7.14E-03	2705	8.1
ICSDS (02-02246)			1.35E+00	6.54E-03	7.14E-03	2705	99
02-02246-PBS			1.46E+00	3.86E-03	7.66E-03	2902	99
02-02246		<	7.14E-02		7.14E-03	2705	
02-02246-DUP		<	7.33E-02		7.33E-03	2776	N.A.
02-02246-BS2			7.64E-01	4.66E-03	7.02E-03	2657	76
02-02246-MS2			8.69E-01	1.91E-03	7.04E-03	2665	88
CCV-1			4.98E-04	3.00E-07	2.64E-06	1	100
CCB-1		<	2.64E-05		2.64E-06	1	

N.A. = not applicable

Narrative:

All Batch QC met their respective success criteria. The Blank Spike recovery was low but passed the 70-130% recovery requested in ASR. Components in the matrix may be assisting Pd solution stability since the matrix spike shows better recovery than the Blank Spike.

Sample ID	Analyte 141Pr		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.99E-04	1.77E-06	1.56E-06	1	100
ICB		<	1.56E-05		1.56E-06	1	
02-02246-B		<	4.53E-02		4.53E-03	2902	
DT (02-02246)		<	2.11E-01		2.11E-02	13527	N.A.
ICS (02-02246)		<	4.23E-02		4.23E-03	2705	
ICSD (02-02246)		<	4.23E-02		4.23E-03	2705	N.A.
ICSDS (02-02246)			1.35E+00	4.57E-04	4.23E-03	2705	99
02-02246-PBS			1.45E+00	2.12E-03	4.53E-03	2902	100
02-02246		<	4.23E-02		4.23E-03	2705	
02-02246-DUP		<	4.34E-02		4.34E-03	2776	N.A.
02-02246-BS2			9.81E-01	2.96E-03	4.15E-03	2657	101
02-02246-MS2			1.00E+00	5.97E-03	4.16E-03	2665	103
CCV-1			5.06E-04	1.02E-06	1.56E-06	1	101
CCB-1		<	1.56E-05		1.56E-06	1	

N.A. = not applicable

Narrative:

All batch and instrument QC solution met their respective success criteria. The sample concentrations are reported below the EQL (10XMDL). A concentration of < 0.04 mg/L is estimated for this sample. No Minimum Reportable Quantities were requested for this analyte.

Sample ID	Analyte 181Ta		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.93E-04	1.11E-06	1.57E-06	1	99
ICB		<	1.57E-05		1.57E-06	1	
02-02246-B		<	4.56E-02		4.56E-03	2902	
DT (02-02246)		<	2.13E-01		2.13E-02	13527	N.A.
ICS (02-02246)		<	4.25E-02		4.25E-03	2705	
ICSD (02-02246)		<	4.25E-02		4.25E-03	2705	N.A.
ICSDS (02-02246)			1.38E+00	5.51E-03	4.25E-03	2705	102
02-02246-PBS			1.47E+00	2.58E-03	4.56E-03	2902	101
02-02246		<	4.25E-02		4.25E-03	2705	
02-02246-DUP		<	4.36E-02		4.36E-03	2776	N.A.
02-02246-BS2			3.24E-01	1.13E-03	4.18E-03	2657	**33**
02-02246-MS2			3.84E-01	1.19E-03	4.19E-03	2665	**40**
CCV-1			5.15E-04	1.88E-06	1.57E-06	1	103
CCB-1		<	1.57E-05		1.57E-06	1	

N.A. = not applicable

Narrative:

Both batch sample spikes (BS, MS) recovered low, however the PBS and PMS and met their respective QC success criteria of 90-110%. The Dilution test and ICS/ICSD was not significant due to low analyte concentrations. Failure of Blank Spike and Matrix Spike indicate that the preparation method was unsatisfactory for the analysis of this analyte.

Sample ID	Analyte 195Pt		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			5.10E-04	1.89E-06	4.25E-06	1	102
ICB		<	4.25E-05		4.25E-06	1	
02-02246-B		<	1.23E-01		1.23E-02	2902	
DT (02-02246)		<	5.75E-01		5.75E-02	13527	N.A.
ICS (02-02246)		<	1.15E-01		1.15E-02	2705	
ICSD (02-02246)		<	1.15E-01		1.15E-02	2705	N.A.
ICSDS (02-02246)			1.39E+00	6.81E-03	1.15E-02	2705	102
02-02246-PBS			1.48E+00	2.30E-03	1.23E-02	2902	101
02-02246		<	1.15E-01		1.15E-02	2705	
02-02246-DUP		<	1.18E-01		1.18E-02	2776	N.A.
02-02246-BS2			1.02E+00	4.76E-03	1.13E-02	2657	104
02-02246-MS2			1.05E+00	3.00E-03	1.13E-02	2665	108
CCV-1			5.13E-04	5.50E-07	4.25E-06	1	103
CCB-1		<	4.25E-05		4.25E-06	1	

N.A. = not applicable

Narrative:

All batch and instrument QC solutions met their respective success criteria. The sample concentrations are reported below the EQL (10XMDL). A concentration of < 0.12 mg/L is estimated for this sample. No Minimum Reportable Quantities were requested for this analyte.

Sample ID	Analyte 237Np		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.95E-04	8.08E-07	1.49E-06	1	99
ICB		<	1.49E-05		1.49E-06	1	
02-02246-B		<	4.32E-02		4.32E-03	2902	
DT (02-02246)		<	2.02E-01		2.02E-02	13527	98
ICS (02-02246)		<	4.03E-02		4.03E-03	2705	
ICSD (02-02246)		<	4.03E-02		4.03E-03	2705	N.A.
ICSDS (02-02246)			1.40E+00	2.87E-03	4.03E-03	2705	102
02-02246-PBS			1.45E+00	3.20E-03	4.32E-03	2902	100
02-02246		<	4.03E-02		4.03E-03	2705	
02-02246-DUP		<	4.14E-02		4.14E-03	2776	N.A.
02-02246-BS2		<	3.96E-02		3.96E-03	2657	N.S.
02-02246-MS2		<	3.97E-02		3.97E-03	2665	N.S.
CCV-1			5.03E-04	1.19E-06	1.49E-06	1	101
CCB-1		<	1.49E-05		1.49E-06	1	

N.A. = not applicable

N.S. = The Batch QC sample was not spiked.

Narrative:

All batch and instrument QC solutions met their respective success criteria. Batch QC spiking of radionuclides was not performed for this work, however a Post Matrix Spike (PMS) and Post Blank Spike (PBS) were prepared and analyzed and passed their respective QC success criteria. The sample concentrations are reported below the EQL (10XMDL). A concentration of < 0.04 mg/L or 3.5E-05uCi/L is estimated for this sample. No Minimum Reportable Quantities were requested for this analyte.

Sample ID	Analyte 238U		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.77E-04	2.41E-06	1.08E-05	1	95
ICB		<	1.08E-04		1.08E-05	1	
02-02246-B		<	3.14E-01		3.14E-02	2902	
DT (02-02246)			1.87E+02	4.13E+00	1.46E-01	13527	113
ICS (02-02246)			1.65E+02	4.57E-01	2.92E-02	2705	
ICSD (02-02246)			1.66E+02	6.72E-01	2.92E-02	2705	0.7
ICSDS (02-02246)			1.67E+02	2.85E-01	2.92E-02	2705	108
02-02246-PBS			1.50E+00	1.06E-02	3.14E-02	2902	101
02-02246			1.68E+02	2.12E-01	2.92E-02	2705	
02-02246-DUP			1.66E+02	4.24E-01	3.00E-02	2776	1.3
02-02246-BS2		<	2.87E-01		2.87E-02	2657	N.S.
02-02246-MS2			1.79E+02	4.00E-01	2.88E-02	2665	N.S.
CCV-1			5.07E-04	5.92E-06	1.08E-05	1	101
CCB-1		<	1.08E-04		1.08E-05	1	

N.S. = The Batch QC sample was not spiked.

Narrative:

All batch and instrument QC solution met their respective success criteria except the dilution test. Batch QC spiking of radionuclides was not performed for this work, however a Post Matrix Spike (PMS) and Post Blank Spike (PBS) were prepared and analyzed and passed their respective QC success criteria. Failure of dilution test >10% is unknown; however the high concentration of U found in this sample may result in an error in dead time correction. Often, the optimum range for obtaining the most data can lead to poor results for concentrated analytes due to instrument saturation (high dead time). The high recovery of the DT suggests that dead time corrections for the less diluted sample was insufficient and there fore more accuracy credibility should be given to the Dilution Test result.

Sample ID	Analyte 233U		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			1.82E-07	1.85E-07	1.68E-08	1	(a)
ICB			1.76E-07	1.82E-07	1.68E-08	1	
02-02246-B			5.16E-04	5.30E-04	4.87E-05	2902	
DT (02-02246)			1.01E-02	1.40E-02	2.27E-04	13527	N.A.
ICS (02-02246)			8.13E-03	6.19E-04	4.54E-05	2705	
ICSD (02-02246)			8.03E-03	7.15E-04	4.54E-05	2705	1.2
ICSDS (02-02246)			8.15E-03	6.24E-04	4.54E-05	2705	(a)
02-02246-PBS			5.28E-04	5.24E-04	4.87E-05	2902	(a)
02-02246			8.26E-03	6.19E-04	4.54E-05	2705	
02-02246-DUP			8.16E-03	6.92E-04	4.65E-05	2776	1.2
02-02246-BS2			4.72E-04	4.84E-04	4.46E-05	2657	N.S.
02-02246-MS2			8.82E-03	5.82E-04	4.47E-05	2665	N.S.
CCV-1			1.82E-07	1.84E-07	1.68E-08	1	(a)
CCB-1		<	1.68E-07		1.68E-08	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.
(a) = The instrument QC samples were not spiked for minor isotopes of Uranium.

Narrative:

All batch and instrument QC solutions met their respective success criteria based on ^{238}U QC checks. The ICB, although >EQL is nearly identical to EQL and orders of magnitude below the sample concentration. Batch QC spiking of radionuclides was not performed for this work. All isotopes of uranium were determined on a direct analysis of the sample solution.

Sample ID	Analyte 234U		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			1.87E-07	1.85E-07	1.63E-08	1	(a)
ICB			1.78E-07	1.82E-07	1.63E-08	1	
02-02246-B			5.27E-04	5.28E-04	4.72E-05	2902	
DT (02-02246)			1.33E-02	1.45E-02	2.20E-04	13527	N.A.
ICS (02-02246)			1.15E-02	5.19E-04	4.40E-05	2705	
ICSD (02-02246)			1.16E-02	7.14E-04	4.40E-05	2705	N.A.
ICSDS (02-02246)			1.15E-02	7.97E-04	4.40E-05	2705	(a)
02-02246-PBS			5.50E-04	5.33E-04	4.72E-05	2902	(a)
02-02246			1.18E-02	6.15E-04	4.40E-05	2705	
02-02246-DUP			1.15E-02	6.50E-04	4.52E-05	2776	2.0
02-02246-BS2			4.89E-04	4.80E-04	4.32E-05	2657	N.S.
02-02246-MS2			1.24E-02	6.04E-04	4.34E-05	2665	N.S.
CCV-1			1.91E-07	1.84E-07	1.63E-08	1	(a)
CCB-1		<	1.63E-07		1.63E-08	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.
(a) = The instrument QC samples were not spike for minor isotopes of Uranium.

Narrative:

All batch and instrument QC solutions met their respective success criteria based on ^{238}U QC checks. The ICB, although >EQL is nearly identical to EQL and orders of magnitude below the sample concentration. Batch QC spiking of radionuclides was not performed for this work. All isotopes of uranium were determined on a direct analysis of the sample solution.

Sample ID	Analyte 235U		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV		<	1.17E-05		1.17E-06	1	(a)
ICB		<	1.17E-05		1.17E-06	1	
02-02246-B		<	3.39E-02		3.39E-03	2902	
DT (02-02246)			1.23E+00	3.78E-02	1.58E-02	13527	N.A.
ICS (02-02246)			1.22E+00	1.59E-03	3.16E-03	2705	
ICSD (02-02246)			1.23E+00	1.07E-03	3.16E-03	2705	N.A.
ICSDS (02-02246)			1.21E+00	6.85E-03	3.16E-03	2705	(a)
02-02246-PBS		<	3.39E-02		3.39E-03	2902	(a)
02-02246			1.25E+00	2.10E-03	3.16E-03	2705	
02-02246-DUP			1.23E+00	1.45E-03	3.24E-03	2776	1.5
02-02246-BS2		<	3.10E-02		3.10E-03	2657	N.S.
02-02246-MS2			1.31E+00	4.14E-03	3.11E-03	2665	N.S.
CCV-1		<	1.17E-05		1.17E-06	1	(a)
CCB-1		<	1.17E-05		1.17E-06	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.
(a) = The instrument QC samples were not spike for minor isotopes of Uranium.

Narrative:

All batch and instrument QC solutions met their respective success criteria based on ^{238}U QC checks. Batch QC spiking of radionuclides was not performed for this work. All isotopes of uranium were determined on a direct analysis of the sample solution.

Sample ID	Analyte 236U		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV		<	8.21E-07		8.21E-08	1	(a)
ICB		<	8.21E-07		8.21E-08	1	
02-02246-B		<	2.38E-03		2.38E-04	2902	
DT (02-02246)			9.24E-02	1.40E-02	1.11E-03	13527	N.A.
ICS (02-02246)			9.31E-02	6.07E-04	2.22E-04	2705	
ICSD (02-02246)			9.30E-02	8.43E-04	2.22E-04	2705	N.A.
ICSDS (02-02246)			9.14E-02	1.08E-03	2.22E-04	2705	(a)
02-02246-PBS		<	2.38E-03		2.38E-04	2902	(a)
02-02246			9.58E-02	7.18E-04	2.22E-04	2705	
02-02246-DUP			9.45E-02	1.32E-03	2.28E-04	2776	1.4
02-02246-BS2		<	2.18E-03		2.18E-04	2657	N.S.
02-02246-MS2			9.92E-02	9.21E-04	2.19E-04	2665	N.S.
CCV-1		<	8.21E-07		8.21E-08	1	(a)
CCB-1		<	8.21E-07		8.21E-08	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.
(a) = The instrument QC samples were not spike for minor isotopes of Uranium.

Narrative:

All batch and instrument QC solutions met their respective success criteria based on ^{238}U QC checks. Batch QC spiking of radionuclides was not performed for this work. All isotopes of uranium were determined on a direct analysis of the sample solution.

Sample ID	Analyte 239Pu		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.82E-04	4.95E-06	8.32E-07	1	98
ICB		<	8.32E-06		8.32E-07	1	
02-02246-B		<	2.41E-02		2.41E-03	2902	
DT (02-02246)			1.19E-01	4.75E-03	1.13E-02	13527	N.A.
ICS (02-02246)			1.02E-01	5.77E-04	2.25E-03	2705	
ICSD (02-02246)			1.02E-01	3.40E-04	2.25E-03	2705	0.3
ICSDS (02-02246)			1.34E+00	7.06E-03	2.25E-03	2705	93
02-02246-PBS			1.42E+00	6.76E-03	2.41E-03	2902	100
02-02246			1.06E-01	7.39E-04	2.25E-03	2705	
02-02246-DUP			1.01E-01	2.57E-04	2.31E-03	2776	5.2
02-02246-BS2		<	2.21E-02		2.21E-03	2657	N.S.
02-02246-MS2			1.08E-01	4.20E-04	2.22E-03	2665	N.S.
CCV-1			4.91E-04	4.42E-06	8.32E-07	1	100
CCB-1		<	8.32E-06		8.32E-07	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.

Narrative:

All batch and instrument QC solution met their respective success criteria. Batch QC spiking of radionuclides was not performed for this work, however a Post Matrix Spike (PMS) and Post Blank Spike (PBS) were prepared and analyzed and passed their respective QC success criteria. The dilution test is not a valid test due to low concentration (i.e. <5X EQL).

Sample ID	Analyte 240Pu		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			9.85E-06	2.23E-07	3.49E-08	1	96
ICB		<	3.49E-07		3.49E-08	1	
02-02246-B		<	1.01E-03		1.01E-04	2902	
DT (02-02246)			8.47E-03	7.04E-03	4.72E-04	13527	N.A.
ICS (02-02246)			7.55E-03	2.84E-04	9.44E-05	2705	
ICSD (02-02246)			7.61E-03	5.43E-04	9.44E-05	2705	0.9
ICSDS (02-02246)			3.50E-02	3.10E-04	9.44E-05	2705	99
02-02246-PBS			2.90E-02	6.02E-04	1.01E-04	2902	96
02-02246			7.95E-03	3.63E-04	9.44E-05	2705	
02-02246-DUP			7.66E-03	3.76E-04	9.68E-05	2776	3.6
02-02246-BS2		<	9.27E-04		9.27E-05	2657	N.S.
02-02246-MS2			8.14E-03	3.73E-04	9.29E-05	2665	N.S.
CCV-1			1.01E-05	2.64E-07	3.49E-08	1	99
CCB-1		<	3.49E-07		3.49E-08	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.

Narrative:

All batch and instrument QC solution met their respective success criteria. Batch QC spiking of radionuclides was not performed for this work, however a Post Matrix Spike (PMS) and Post Blank Spike (PBS) were prepared and analyzed and passed their respective QC success criteria. The dilution test is not a valid test due to low concentration (i.e. <5X EQL).

Sample ID	Analyte 241AMU		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV			4.79E-05	1.34E-07	1.14E-08	1	96
ICB		<	1.14E-07		1.14E-08	1	
02-02246-B		<	3.30E-04		3.30E-05	2902	
DT (02-02246)		<	1.54E-03		1.54E-04	13527	N.A.
ICS (02-02246)			5.17E-04	5.77E-05	3.07E-05	2705	
ICSD (02-02246)			5.44E-04	3.92E-05	3.07E-05	2705	5.1
ICSDS (02-02246)			1.34E-01	4.08E-04	3.07E-05	2705	99
02-02246-PBS			1.41E-01	4.78E-04	3.30E-05	2902	97
02-02246			5.89E-04	3.07E-05	3.07E-05	2705	
02-02246-DUP			5.71E-04	3.75E-05	3.15E-05	2776	3.0
02-02246-BS2		<	3.02E-04		3.02E-05	2657	N.S.
02-02246-MS2			5.61E-04	1.39E-05	3.03E-05	2665	N.S.
CCV-1			4.85E-05	6.97E-08	1.14E-08	1	97
CCB-1		<	1.14E-07		1.14E-08	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.

Narrative:

All batch and instrument QC solution met their respective success criteria. Batch QC spiking of radionuclides was not performed for this work, however a Post Matrix Spike (PMS) and Post Blank Spike (PBS) were prepared and analyzed and passed their respective QC success criteria. Since it is not known what fraction of this isotope is 241Pu or 241Am, calculation of activity cannot be performed. However, the conversion factors to uCi/L are 127uCi/ug 241Pu and 4.23uCi/ug 241Am. The dilution test is not a valid test due to low concentration (i.e. <5X EQL).

Sample ID	Analyte 242Pu		Sample Concentration mg/L	+/- 1 sigma mg/L	MDL mg/L	Dilution Factor	% Rec / RPD
ICV		<	1.45E-07		1.45E-08	1	(a)
ICB		<	1.45E-07		1.45E-08	1	
02-02246-B		<	4.20E-04		4.20E-05	2902	
DT (02-02246)		<	1.96E-03		1.96E-04	13527	N.A.
ICS (02-02246)		<	3.92E-04		3.92E-05	2705	
ICSD (02-02246)		<	3.92E-04		3.92E-05	2705	N.A.
ICSDS (02-02246)		<	3.92E-04		3.92E-05	2705	(a)
02-02246-PBS		<	4.20E-04		4.20E-05	2902	(a)
02-02246		<	3.92E-04		3.92E-05	2705	
02-02246-DUP		<	4.02E-04		4.02E-05	2776	N.A.
02-02246-BS2		<	3.85E-04		3.85E-05	2657	N.S.
02-02246-MS2		<	3.86E-04		3.86E-05	2665	N.S.
CCV-1		<	1.45E-07		1.45E-08	1	(a)
CCB-1		<	1.45E-07		1.45E-08	1	

N.A. = not applicable
N.S. = The Batch QC sample was not spiked.
(a) = The instrument QC samples was not spiked for Pu-242.

Narrative:

All batch and instrument QC solution met their respective success criteria based on ^{239}Pu QC checks. Batch QC spiking of radionuclides was not performed for this work. All isotopes of plutonium were determined on a direct analysis of the sample solution.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W60654
ASR#:	6403
Client:	S.K. Fiskum
Total Samples:	1

Analysis: 129-Iodine

Procedure: PNL-SCO1 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number: R102924 PQ-Excell, Thermal Elemental
512-06-01-014 Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Report Date: 23 Oct 2002

Analysis Files: 28 Sep 2002

For Calibration and Maintenance Records, see ICPMS Service Center 98038 RIDS

Analyst(s): Hans Carman / Orville Thomas Farmer III

Reviewed By 23/oct 02
Date

Concur 23/oct/02
Date

Sample Solutions Submitted for Analysis:

RPL #	Client I.D.
02-02246-B	Process Blank
02-02246	AZ-101 Cs Eluate Comp-HLW

One (1) sample (AZ-101 Cs Eluate Comp-HLW) prepared by diluting with water was submitted for analysis and analyzed per 329-OP-SC01 on a radioactive-material-contained ICP/MS for all requested analyte(s). A duplicate was not prepared, however, duplicates from the sample were prepared at the ICP/MS workstation and identified as ICS(02-2246) and ICSD(02-2246). A Post Blank Spike identified as 02-02246-PBS (i.e. a Process Blank spiked with ^{129}I) and a Post Matrix Spike identified as ICSDS(02-02246) (i.e. the sample spiked with ^{129}I) was prepared to assure ICP/MS work station preparation/dilution processes satisfied QC recovery requirements. The ASR did not specify an MRQ.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis. Results are reported in mg/L.

The preparative batch QC for ^{129}I (and all radionuclides) included a preparation blank (PB). Iodine 129 (and all radionuclides) was not spiked into a separate BS/LCS or MS. Matrix effects for radionuclides were evaluated, instead, on post-spike recoveries at the analytical workstation, as is typical with radioisotope measurements.

A ^{129}I calibration standard was used to determine the concentration of ^{129}I in the sample. No chemical separations were performed, for any analysis on ASR-6403. However, a correction for natural Xe present in the sample matrix was performed based on the ^{131}Xe to ^{129}Xe ratio in the average blank solution. That is, the measure of ^{131}Xe count rate times the average blank 131/129 ratio (1.216) is subtracted from the counts observed for the 129 mass. With all standards, samples and blanks treated identically.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 7 instrument standard blank solutions, which were evaluated, at the beginning of the analytical run. The IDL was calculated by multiplying the observed standard deviation of the 7 standard blanks solutions by 3.14. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and the sample total dilution factor.

Instrument QCs

Instrument QC checks are used to assure that the instrument is stable through out the analysis. All of the instrument QC checks passed their respective success criteria.

2. Final Results

2.1 ^{129}I Results

Sample ID	Analyte		Sample Conc. (mg/L)	(+/-) 1 sigma	MDL (mg/L)	Dilution Factor	% RSD	% Rec.
02-02246-B	129I	<10X IDL			4.81E-05	100		
02-02246	129I	<10X IDL			4.81E-05	100		
ICS (02-02246)	129I	<10X IDL			4.81E-05	100		
ICSD (02-02246)	129I	<10X IDL			4.81E-05	100	====	
ICSDS (02-02246)	129I		4.85E-03	2.23E-04	4.81E-05	100		94
PBS (02-02246)	129I		4.89E-03	2.40E-04	4.81E-05	100		96

Narrative:

The ^{129}I concentration in the PB and sample were below 10X IDL (4.81E-05 mg/L or 3.14E-04 uCi/mL). The reporting base was not specified in ASR 6403. However, the Shielded Analytical Laboratory Bench Sheet indicated 0.1 ml was taken up in 9.9 ml of DI water. Consequently, we have reported the Results in ml/L.

The post matrix spike (ICSDS) and post blank spike (PBS) recoveries were well within the acceptance criteria of 80-120%.

A Dilution test, two (2) sample replicates (ICS and ICSD) were also analyzed with the submitted samples. The DT (not shown) was not a viable QC check due to low sample concentration. Also the duplicate RSD of the ICS and ICSD is not a viable QC check due also to low sample concentration.

Battelle - Pacific Northwest National Laboratory
Radiochemical Science and Technology – IC Report
PO Box 999, Richland, Washington 99352

Project / Charge Code: 42365 / W60655
ASR Number: 6403
Client: S. Fiskum
Total Samples: 1

	First in Series	Last in Series
RPL Numbers	02-2246	
Client IDs	AZ-101 Cs Eluate Comp HLW	

Analysis Procedure	PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"
Prep Procedure	None
Analyst	MJ Steele
Analysis Date	06/25/2002, 06/25/2002
Calibration Date	04/17/2002
Cal/Ver Stds Prep Date	04/11/2002
Excel Data File	ASR 6306 6347 6401 6403.xls
M&TE Numbers	IC system (WD25214)
	Balance (360-06-01-031)
All Analysis Records	Chemical Measurement Center 98620 RIDS IC System File

MW Thu — 7/17/02
Prepared By **Date**

MJ Steele — 7/18/02
Reviewed By **Date**

Sample Results

RPL No.	Sample ID	Dil Fctr	F µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL (a)		0.13	0.13	0.25	0.13	0.25	0.25	0.25	0.25
	Dilution Blank	1	< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	MRQ		150	10	3,000	n/a	3,000	2,500	2,300	n/a
	EQL (a)		13	13	26	130	2,600	26	26	26
02-2246	AZ-101 CS Eluate Comp HLW	103.83	< 13	180	< 26	< 130	31,200	< 26	300	170
02-2246 MS	AZ-101 Cs Eluate-Comp HLW MS %Recovery		103	104	102	104	108	105	105	108
LCS062402	LCS/BS % Recovery		106	103	105	106	99	107	105	109
	RPD (b)		1	(c)	3	2	3	3	1	4

MRQ: minimum reportable quantity EQL: estimated quantitation limit RPD: relative percent difference

(a) EQL based on lowest calibration standard times dilution factors used to obtain sample results
(b) Batch duplicate RPD calculated from sample AZ-101 FE Comp (ASR 6306, RPL No. 02-0935)
(c) RPD not calculated unless both sample and duplicate results >EQL

Sample Analysis/Results Discussion

The 'AZ-101 Cs Eluate Comp HLW' sample was prediluted in the Shielded Analytical Laboratory (SAL) by approximately 100x. This samples required an additional 100x laboratory dilution in order to ensure that the anions were measured within the calibration range and that the IC column was not overloaded during the analysis. The minimum reportable quantities (MRQ) and the estimated quantitation levels (EQL) are provided; the MRQs are based on a table provided with ASR 6403 and the EQLs are based on the lowest calibration standard adjusted for the dilutions used for reporting the results.

Quality Control Discussion

Duplicate: No sample duplicate was provided by the client. An analysis batch laboratory duplicate was prepared using sample 'AZ-101 FE Comp' from ASR 6306 (02-0935). The laboratory duplicate relative percent difference (RPD) is well within the acceptance criterion of <15% (as defined by the ASR) for all anions measured above the EQL.

Laboratory Control Sample/Blank Spike - (BS 062402 [HCV020411 @3x)]): The high range calibration verification standard diluted by a factor of three was used as the LCS/BS. The LCS/BS demonstrated recoveries within the 80% to 120% acceptance criteria for all anions measured.

Matrix Spike (CCV020411 @2x): A matrix spike (MS) was prepared using the mid-range calibration check standard and the sample. The MS recoveries were within the 75% to 125% recovery acceptance criteria for the anions of measured.

Process/Dilution Blank: A dilution blank (i.e., diluted eluant) was analyzed for all reported analytes and no anions were measured above the EQL.

IC System QC samples: No anions of interest were measured in the calibration blanks (ICB/CCB). The four calibration verification standards analyzed with the samples met the 90% to 110% recovery criteria for all anions measured.

Summary of Deviation: None

General Comments

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- The low calibration standards are defined as the estimated quantitation limit (EQL) for the reported results and assume non-complex aqueous matrices. Actual detection limits or quantitation limits for specific sample matrices may be determined, if requested. For routine analyses, no results are reported below the EQL.
- Routine precision and bias are typically ±15% or better for non-complex aqueous samples that are free of interference.

Battelle
Richland, WA
Radiochemical Science and Engineering, 325 Building

filename 02-2246
7/16/2002

Client: S. Fiskum
ASR 6403

Prepared by: C. Soderquist 7-16-02

Concur: L R Greenwood 7-16-02

Procedure RPG-CMC-226 Rev 0, *Measurement of Ammonia in Aqueous Samples*
Analysis Date: June 11, 2002

Sample	Lab ID	Measured Ammonia Concentration, µg/mL ± 1s		MDL	EQL	MRQ
AZ-101 Cs Eluate	02-2246	**1.8E+0**	**± 23%**	9	133	--
	LCS 1	97%				
	LCS 2	75%				
	Matrix Spike 824	121%				
	Matrix Spike 825	116%				
	Instrument blank 1	10				
	Instrument blank 2	9				
	Instrument blank 3	17				
	Instrument blank 4	9				

Sample AZ-101 Cs Eluate was diluted with water in a hot cell, then surveyed out for analysis in a laboratory hood. Since the sample was acidic to begin with, no preservation was necessary. The laboratory control samples and matrix spikes were prepared in cell in different batch, but were analyzed in the laboratory in the same batch as sample AZ-101 Cs Eluate.

The MDL is the instrument detection limit corrected for sample dilutions. The EQL is the lower end of the linear range of the instrument, corrected for sample dilutions. The instrument blank results have been corrected for the the sample dilutions.

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Radiological Processing Group (RPG)

Inorganic Analysis - Mercury Data Report

Project / WP#: 42365 / W60655
ASR#: 6403
Client: Sandra Fiskum
Total Samples: 1

RPL #	Client ID
02-2246	AZ-101 Cs Eluate

Procedure: RPG-CMC-131 Rev. 0, *Mercury Digestion*
RPG-CMC-201 Rev. 0, *Mercury Analysis*

M&TE Number: WD30853 CETAC, Mercury Analyzer, Model M-6000A
1113052270 Mettler AT400 Balance

Digestion Date: 6/12/02
Analysis Dates: 6/12/02, 8/26/02

Analysis Files: 02061202.DB, 02082601.DB

Analysts: PK Berry, LMP Thomas

Supporting data and records are located in the data package stored in the project file. Balance calibration records can be found in the Radiochemical Science and Engineering RIDS.

9/4/02
Prepared By

9/3/02
Reviewed By

One sample, AZ-101 Cs Eluate, was submitted for mercury analysis. The sample was aliquoted and prepared by digestion in the hot-cell, and analyzed by cold vapor atomic absorption spectroscopy (CVAA) in a fume hood. Preparative and analytical quality control included a preparation blank, laboratory control standard, duplicate, and matrix spike.

1. Analysis

Results from the analysis of the AZ-101 Eluate sample is provided in the table below. Approximately 0.5 mLs of sample was digested and brought to a digest volume of 25 mLs (prepared 6/12/02). Ue to the high dose level, a 0.5 mL aliquot of the digest was transferred to a fume hood and diluted to 10 mLs for analysis. The samples were also analyzed on 6/12/02, however the results between the sample and duplicate were very disparate. Additional aliquots of the digests were then obtained and analyzed on 8/26/02. The concentration is reported in µg of mercury per L of sample.

Table 1. Mercury Results for AZ-101 Eluate.

RPD success criteria: < 20%				
RPL ID	**Sample ID**	**Det Lim µg/L**	**Measured µg/L**	**RPD %**
Analysis 8/26/02				
02-2246 ~~02-829~~	AZ-101 Cs Eluate	2.7	2.7	
02-2246 ~~02-829~~	AZ-101 Cs Eluate DUP	2.7	2.9	
	Average		2.8	
	Est MDL [1]		2.7	
	EQL [2]		50	
	Preparation DF (v/v)		50	
	Analysis DF (v/v)		20	

mwtt 9/5/02

DF - dilution factor

[1] The estimated MDL is based on an MDL evaluated for liquid samples (ASR 6145) adjusted by the appropriate dilution factors.

[2] The EQL is based on the lowest calibration standard, 0.05 µg/L, multiplied by the total dilution factor.

2. Quality Control

Duplicate (DUP). The RPD is not presented since the mercury concentration in the samples does not exceed the EQL.

Matrix Spike (MS). The matrix spike recovery meets the QC success criteria. The RPD between replicate analysis of the matrix spike is 1.9%.

Table 2. Mercury Results for AZ-101 Eluate Matrix Spike.

MS success criteria: 75% to 125% of expected value						
RPL ID	Sample ID	Det Lim µg/L	Spike µg/L	Sample µg/L	Measured µg/L	Rec %
Analysis 6/12/02						
02-2246	AZ-101 Cs Eluate MS	2.8	2590	2.80	2100	81
02-2246	AZ-101 Cs Eluate MS PS	5.4	4950	2100	6860	96
Analysis 8/26/02						
02-2246	AZ-101 Cs Eluate MS	2.7	2590	2.80	2100	82

Preparation Blank (PB) and Laboratory Control Standard (LCS). The PB and LCS were analyzed on 6/12/02 and the results are presented in the table below. The results of the PB are based on the average sample size. The PB and LCS meet the success criteria.

Table 3. Mercury Results for the PB and LCS.

Sample ID	PB success criteria: < EQL		LCS success criteria: 80% - 120% of expected value		
	Success Criteria µg/L	Measured µg/L	Expected µg/L	Measured µg/L	Recovery %
PB/LCS liquid	< 50	< 2.7	1600	1650	103

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards. The ICB/CCB standards meet the success criteria.

Table 4. Mercury Results for the ICB and CCB Standards

ICB/CCB success criteria: < EQL		
Sample ID	Criteria µg/L [1]	Measured µg/L [1]
Analysis 6/12/02		
ICB	< 0.05	< 0.05
CCB 1	< 0.05	< 0.05
CCB 2	< 0.05	< 0.05
CCB-3	< 0.05	< 0.05
CCB 4	< 0.05	< 0.05
Analysis 8/26/02		
ICB	< 0.05	< 0.05
CCB 1	< 0.05	< 0.05
CCB 2	< 0.05	< 0.05
CCB-3	< 0.05	< 0.05
CCB 4	< 0.05	< 0.05
[1] Units are based on per liter of sample at the instrument.		

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards. The ICV/CCV standards meet the success criteria.

Table 5. Mercury Results for the ICV and CCV Standards

ICV/CCV success criteria: 80% to 120% recovery Calibration Range: 0 - 5 µg/L		
Sample ID	**Measured µg/L[1]**	**Recovery %**
Expected	2.00	
Analysis 6/12/02		
ICV	2.02	101
CCV 1	2.00	100
CCV 2	1.96	98
CCV-3	1.99	99
CCV-4	1.96	98
Analysis 8/26/02		
ICV	1.97	98
CCV 1	2.01	101
CCV 2	2.00	100
CCV-3	1.90	94
CCV-4	1.97	98
[1] Units are based on per liter of sample at the instrument.		

Low-Level Standard (LLS). The LLS meets the success criteria.

Table 6. Mercury Results for the LLS

LLS success criteria: 75% to 125% recovery Lowest calibration std: 0.05 µg/L		
Expected µg/L[1]	**Measured µg/L[1]**	**Recovery %**
Analysis 6/12/02		
0.050	0.046	93
Analysis 8/26/02		
0.050	0.051	103
[1] Units are based on per liter of sample at the instrument.		

3. Comments

a). The mercury results have been corrected for all dilution factors performed on the sample during preparation and analysis.

b). The detection limit is based on detection limit studies using water (for the determination of the detection limit in liquid matrices) and sand (for the determination of the detection limit in solid matrices) and documented in ASR 6145. The estimated quantitation limit (EQL) is defined as the lowest calibration standard.

c). Routine precision and bias is typically ±15% or better for non-complex aqueous samples that are free of interference.

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

06/07/02

Client : Sandy Fiskum
ASR: 6403

Cognizant Scientist: L.R. Greenwood Date : 6/7/02

Concur : T Trang-Le Date : 6/7/02

Reference Date: June 5, 2002 for GEA
Procedure: PNL-ALO-450 for GEA

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	Cr-51 Error %	Fe-59 Error %	Co-60 Error %	Y-88 Error %	Nb-95 Error %	Ru-103 Error %	Ru-106 Error %	Sn-113 Error %
02-2246 AZ101-CsEluate	<2.E1	<2.E-1	<7.E-2	<2.E-1	<2.E-1	<2.E0	<9.E0	<3.E0

RPL ID Client ID	Sb-125 Error %	Sn/Sb-126 Error %	Cs-134 Error %	Cs-137 Error %	Ce-144 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Th-232 Error %
02-2246 AZ101-CsEluate	<6.E0	<3.E0	4.39E+0 3%	1.30E+4 3%	<8.E0	<3.E-1	<2.E-1	<4.E0	<3.E0

Note: The sample was counted for 14 hours. Due to the high level of Cs-137 activity, it was not possible to achieve the requested MRQ values for Eu-154 and Eu-155.

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Project No.

Internal Distribution
File/LB

Date July 2, 2002

To Sandy Fiskum

From Stan Bos Stan Bos

Subject Cesium isotopic analysis (RPL # 02-02246)

Sandy

Cesium isotopic distribution analysis of a sample from AZ101 has been completed. Approximately two micro liters of the sample was plated on a rhenium carbide filament and analyzed on the single stage mass spectrometer (M&TE # WB76849) in accordance with PNNL technical procedure PNNL-98523-264. Since natural cesium in mono-isotopic, a sample of rubidium chloride isotopic standard (NBS 984) was run to standardize the mass spectrometer. Work package W60655 will be charged for the analysis.

Please feel free to call me at 376-5384 with any questions you might have.

Sample Id. AZ-101 RPL # 02-02246

	^{133}Cs	^{135}Cs	^{137}Cs
	Weight percent	Weight percent	Weight percent
AZ 101	51.9 ± 0.5	16.9 ± 0.2	31.2 ± 0.2
AZ 101 (dup)	51.8 ± 0.5	16.9 ± 0.2	31.3 ± 0.2

Concurrence P.K Barney 7-10-02

E54-1900-001 (8/98)

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

6/14/2002

Client : Sandy Fiskum
ASR: 6403

Cognizant Scientist: L.R. Greenwood Date : 6/14/02

Concur : T Trang-le Date : 6/14/02

Procedure: PNL-ALO-418 for H-3
Date Reference: June 13, 2002

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	H-3 Error +/-
02-2246 AZ101-CsEluate	<3.E-3
02-2246 DUP AZ101-CsEluate	<3.E-3
Matrix Spike	89%
Blank Spike	91%
Blank	<3.E-3

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

06/26/02

Client : Sandy Fiskum
ASR: 6403

Cognizant Scientist: L R Greenwood Date : 6/26/02

Concur : T Trang-Le Date : 6/27/02

Procedure: PNL-ALO-482 for HC-14
Date Reference: June 12, 2002

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	C-14	+/-%
02-2246 AZ101-CsEluate	<3.E-3	
02-2246 DUP AZ101-CsEluate	<3.E-3	
Matrix Spike 02-2246	107%	
Blank Spike	102%	
Blank	<3.E-3	

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

filename 02-2246
10/03/02

Client : S. Fiskum
ASR: 6403

Cognizant Scientist: C. Jackson Date : 10-3-02

Concur : J R Freeman Date : 10/03/02

Procedure RPG-CMC-4018, Rev 0, *Nickel-63 Analysis*
Reference Date: July 23, 2002

Sample	Lab ID	Ni-63 Measured Activity µCi/ml ± 1s		MDA
AZ-101-Cs Eluate	02-2246	2.28E-3	± 35%	<3.E-3
AZ-101-Cs Eluate	02-2246 dup	4.14E-3	± 20%	<3.E-3
	Lab blank	<3.E-3		
	Matrix spike 2788	96%		
	Reagent spike	96%		

Note: The beta energy spectrum does not clearly show a Ni-63 peak. Results are reported based on counts in the expected Ni-63 energy region.

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Date: 06/11/02

Subject: **Se-79** Analysis Report on: Client ID: AZ-101-Cs Eluant Comp-HLW

ASR: **6403**

Sample RPG ID: **02-2246**

WP#: W-60650

Project: 42365

To: S. Fiskum

Se-79 was measured in triplicate on the Acid Digest Fraction (103.83x dilution) of AZ101 -Cs Eluate according to procedure PNL-ALO-440 and the results are listed below. Since Se-79 is not available as a radioactive standard, it was not possible to provide a LCS or matrix spike. Se carrier was used in the analysis for establishing the yield and C-14 was used to establish the instrument efficiency since it has a very similar beta max energy (156 Kev vs 149 Kev for Se-79). One mL aliquots of the HNO_3 diluted slurry material provided to the lab were analyzed in triplicate. The gravimetric recoveries for the reagent blank, lab blank, and samples, are listed below. The Se-79 activities were measured by liquid scintillation counting according to procedure PNL-ALO-474. No peaks were observed in the Se-79 R.O.I. beta energy spectral plots provided and no other higher energy beta contaminants were observed. The average MDC value was < 2.2 E-4 uCi/mL. No MRQ value was provided for Se-79.

		Se	Se-79 Result	Se-79 1s TPU	Se-79 MDC	
I.D.	Client ID	Recovery	uCi/mL	uCi/mL	uCi/mL	TPU,%
02-2246-B	Process Blk	0.77	-1.03E-5	5.33E-5	1.78E-4	517%
02-2246	AZ-101-Cs Elua	0.62	-8.67E-5	6.36E-5	2.20E-4	73%
02-2246-rep	AZ-101-Cs Elua	0.66	-8.45E-5	5.97E-5	2.06E-4	71%
02-2246-trip	AZ-101-Cs Elua	0.58	-3.26E-5	7.04E-5	2.37E-4	216%
		Ave =	-6.79E-5		**2.2E-4**	
		Std. Dev.=	3.06E-5			

Prepared by: _____ Date: 6-12-02

Reviewed by: _____ Date: 6-12-02

Battelle Pacific Northwest National Laboratory
Radiochemical Science & Engineering -325 Building

filename 02-2246
01/15/03
Rev. 2

Client : S. Fiskum
ASR: 6403

Cognizant Scientist: L R Greenwood Date : 1/15/03

Concur : C. Soderquist Date : 1-15-03

Procedure RPG-CMC-417, Rev 1
Reference Date: July 26, 2002

Sample	Lab ID	Sm-151 Measured Activity µCi/ml ± 1s counting error		MDA
AZ-101-Cs Eluate	02-2246	5.73E-3	± 6%	6E-4
AZ-101-Cs Eluate	02-2246 dup	6.52E-3	± 6%	6E-4
	RPD	13%		
	Lab blank	< 6E-4		
	Matrix spike 2246	76%		
	Reagent spike	73%		

Note: The Sm-151 results were revised due to the discovery of an error on the preparation of the Sm-151 standard that was used to determine the liquid scintillation counting efficiency.

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

06/13/02

Client : Sandy Fiskum
ASR: 6403

Cognizant Scientist: L R Greenwood Date : 6/13/02

Concur : T. Trang-Le Date : 6/13/02

Procedure: PNL-ALO-408 for Alpha
Procedure: PNL-ALO-476 for Sr-90
Procedure: PNL-ALO-417 & 496 for Pu & Am
Procedure: PNL-ALO-4001 for Beta

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	Beta Error +/-	Sr-90 Error +/-	Alpha Error +/-	Pu-239+ Pu-240 Error +/-	Pu-238 Error +/-	Pu-236 Error +/-	Am-241 Error +/-	Cm-243+ Cm-244 Error +/-	(Am-242m) Cm-242** Error +/-	Sum of Total Alpha Error +/-
02-2246 AZ101-CsEluate	1.39E+4 4%	3.41E+0 6%	5.63E-3 23%	7.94E-3 3%	8.33E-4 5%	<2.E-5	2.24E-4 5%	1.83E-5 16%	<3.E-6	9.02E-3 3%
02-2246 DUP AZ101-CsEluate	1.40E+4 4%	3.19E+0 6%	8.86E-3 17%	7.93E-3 3%	8.19E-4 5%	<9.E-6	2.31E-4 5%	2.18E-5 15%	<3.E-6	9.00E-3 3%
RPD	1%	7%	45%	0%	2%		3%	17%		0%
Matrix Spike	*	101%	110%	104%			98%			
Blank Spike	102%	100%	102%	103%			100%			
Blank	<2.E-2	<3.E-1	<3.E-3	<3.E-6	<2.E-6	<2.E-6	<4.E-6	<3.E-6	<4.E-6	

*The matrix spike was too low compared to the sample activity such that a meaningful yield could not be determined.
** Cm-242 is the daughter of Am-242m (141y). The activity of Am-242m is a factor of 1.21 times higher than that of Cm-242.

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

09/17/02

Client : Sandy Fiskum
ASR: 6403

Cognizant Scientist: L R Greenwood Date : 9/17/02

Concur : T Trang-Le Date : 9/18/02

Procedure: PNL-ALO-476 for Sr-90

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	Recount Sr-90 Error +/-	Original Sr-90 Error +/-
02-2246 AZ101-CsEluate	3.14E+0 3%	3.41E+0 6%
02-2246 DUP AZ101-CsEluate	2.93E+0 3%	3.19E+0 6%
RPD	7%	7%
Matrix Spike	84%	101%
Blank Spike	87%	100%
Blank	<7.E-3	<3.E-1

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

07/02/02

Client : Sandy Fiskum
ASR: 6403

Cognizant Scientist: L R Greenwood Date : 7/2/02

Concur : C Soderquist Date : 7-3-02

Procedure: PNL-ALO-417/494 for Pu-241
Date Reference: June 20, 2002

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID		Pu-241	+/-%
02-2246 AZ101-CsEluate		3.93E-2	7%
02-2246 DUP AZ101-CsEluate		3.88E-2	7%
	RPD	1%	
Matrix Spike AZ101-CsEluate		114%	
LCS		113%	
Blank		3.65E-4	21%

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Project No. 42365

Internal Distribution
File/LB

Date May 23, 2002

To Sandra Fiskum

From Karl Pool 5/23/02

Subject Weight Percent Oxides, ASR 6403

Mike,

Attached are the bench sheet results for Total Dissolved Solids (TDS) analyses and the work sheet results for the Weight Percent Oxides Determination of the AZ-101 Cs Elute Comp-HLW sample. The sample was analyzed in accordance with "ASR 6403 Special Instructions". The sample preparations were performed within the Hot Cells of the 325 laboratory. All samples were weighed using a Mettler AE160 balance (M&TE # 360-06-01-016) located in Hot Cell number two of the Shielded Analytical Laboratory. All TDS temperature readings were obtained from a calibrated thermocouple (M&TE # 3118) and temperature readout (M&TE # 2115). The high temperature furnace readings were obtained from a calibrated thermocouple (M&TE # 14088) and temperature readout (M&TE # 2115). The TDS and weight percent oxides determinations were performed in triplicate. The sample has been assigned RPL log number 02-02246. Work package number W60650 will be charged for the analyses.

Fiskum RPL=02-2246
ASR 6403 Special Instructions - SAL

WP# = W60650

Determination of Wt% total Solids and Wt% total Oxides

Balance Calibration ID 360-06-01-016
Calibration Expiration Date Feb. 2003
Balance Location Cell 2

Furnace Thermocouple Calibration ID 14088
Calibration Expiration Date 7/31/02
Furnace Location Cell 3

1) Transfer about three, 3 ml of AZ-101-Cs Eluate-Comp-HL (02-02246) into pre-weighed ceramic crucibles. Weigh the loaded crucibles.

Crucible #1 ✓	Crucible #2 ✓	Crucible #3 ✓
Total 19.8374 g	Total 34.9872 g	Total 38.9649 g
Tare 16.8771 g	Tare 32.0401 g	Tare 36.0292 g
Slurry 2.9603 g	Slurry 2.9471 g	Slurry 2.9357 g

2) Dry the slurrried material in a drying oven set at 50 ± 5 °C for 48 hours.

3) Remove the slurried sample material from the drying oven. Allow the crucibles to cool in a dessicator to room temperature (typically about 1 hr) and reweigh. Record the date and time.

Time 0900 Date 5/20/02

Crucible #1 16.8898 g Crucible #2 32.0553 g Crucible #3 36.0437 g

4) Raise the temperature of the drying oven to 105 ± 3 °C and dry for an additional 24 hours.

5) Remove the slurried sample material from the drying oven. Allow the crucibles to cool in a dessicator to room temperature (typically about 1 hr) and reweigh. Record the date and time.

Time 1130 Date ~~5/21/03~~ 5/21/02 ZNP 9/18/02

Crucible #1 16.8900 g Crucible #2 32.0558 g Crucible #3 36.0440 g

6) As directed by the cognizant scientist, repeat steps 4 and 5 until samples have reached a constant mass. Record Mass data on Wt% Solids/TDS data sheet.

7) Place crucibles in furnace

8) Heat the furnace and hold for 30 minutes at between 100 and 1050 °C.

9) Turn off furnace and cool to about 150 °C (typically about 1 hour).

10) Transfer crucibles using tongs to a dessicator and cool to room temperature (typically 1 to 2 hours).

11) Reweigh the crucibles and record any observations

Crucible #1 16.8848 g Crucible #2 32.0453 g Crucible #3 36.0330 g

Signature [signature] 5/31/02 Date [signature] 5/30/02

PNL-ALO-504 Wt% Solids/TDS Data Sheet

Client name: S. Fiskum
Work Auth. Doc (WAD): ASR 6403
Tank/Core/Project: AZ-101-Cs Eluate-Comp-HLW
Special instructions: Use 3 ml aliquots in triplicate. Prepare samples in pre-fired ceramic crucibles. Dried Samples in the ceramic crucibles will be used for total oxides determnation after TDS is determined.

Work package number: W60650
Project number: 42365
PNL QA plan: HASQARD
PNL impact level: N/A
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number: N/A

	RPL Sample ID	Client sample ID	TARE WEIGHT (g)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS/TDS
1	02-02246	AZ-101 Cs Eluate Comp-HLW	16.8771	19.8374	16.8898	0.43
2	02-02246 - Dup	AZ-101 Cs Eluate Comp-HLW-Dup	32.0401	34.4872	32.0553	0.52
3	02-02246 - Triplicate	AZ-101 Cs Eluate Comp-HLW-Trip	36.0292	38.9649	36.0437	0.49

Wt% SOLIDS/TDS = (B - TARE) / (A - TARE) x 100

DATE/TIME IN: 5/17/02 1100 OVEN TEMPERATURE: 58° °C
DATE/TIME OUT: 5/20/02 1000 OVEN TEMPERATURE: 103 °C
DATE/TIME OUT: 5/21/02 1130 OVEN TEMPERATURE: 103 °C

BALANCE: CELL 2 (360-06-01-016) ___X___

TEMPERATURE READOUT: 2115 EXPIRATION DATE: 5/16/02

THERMOCOUPLE: 3118 EXPIRATION DATE: 4/02

Analyst/Date: [signature] 5/21/02 Reviewer/Date: [signature] 5/23/02

D.96

PNL-ALO-504

Wt% Solids/TDS Data Sheet

Client name: S. Fiskum
Work Auth. Doc (WAD): ASR 6403
Tank/Core/Project: AZ-101-Cs Eluate-Comp-HLW
Special instructions: Use 3 ml aliquots in triplicate. Prepare samples in pre-fired ceramic crucibles.
Dried Samples in the ceramic crucibles will be used for total oxides determnation after TDS is determined.

Work package number: W60650
Project number: 42365
PNL QA plan: HASQARD
PNL impact level: N/A
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number: N/A

	RPL Sample ID	Client sample ID	TARE WEIGHT (g)	(A) SAMPLE WET WEIGHT PLUS TARE	(B) SAMPLE DRY WEIGHT PLUS TARE	WEIGHT % SOLIDS/TDS
1	02-02246	AZ-101 Cs Eluate Comp-HLW	16.8771	19.8374	16.8898	0.43
2	02-02246 - Dup	AZ-101 Cs Eluate Comp-HLW-Dup	32.0401	34.9872	32.0553	0.52
3	02-02246 - Triplicate	AZ-101 Cs Eluate Comp-HLW-Trip	36.0292	38.9649	36.0437	0.49

$$\text{Wt\% SOLIDS/TDS} = \frac{\text{B - TARE}}{\text{A - TARE}} \times 100$$

DATE/TIME IN: 5/17/02 11:00 AM OVEN TEMPERATURE: 58 °C
DATE/TIME OUT: 5/20/02 10:00 AM OVEN TEMPERATURE: 103 °C
DATE/TIME OUT: 5/21/02 11:30 AM OVEN TEMPERATURE: 103 °C

BALANCE: CELL 2 (360-06-01-016) X

TEMPERATURE READOUT: 2115 EXPIRATION DATE: 5/16/02

THERMOCOUPLE: 3118 EXPIRATION DATE: 4/02

Analyst/Date: 5/29/02 Reviewer/Date: 5/29/02

AZ-101 Cs Eluate Comp-HLW Wt% Oxides

Test Plan: ASR 6403 Special Instructions

02-02246

Date: 5/23/2002

Crucible Data			Liquid Fraction		
Step #	Description	units	LA	LB	LC
1,2	Pre-fired Crucible Tare	g	16.8771	32.0401	36.0292
1,2	Crucible + AZ-101Cs Eluate material	g	19.8374	34.9872	38.9649
1,2	net wt. AZ-101 Cs Eluate material	g	2.9603	2.9471	2.9357
8	Stable dry wt. @ 105 deg C.	g	16.8898	32.0553	36.0437
N/A	net dry wt. @ 105 deg C.	g	0.0127	0.0152	0.0145
13	Total Wt. after 1000-1050 deg C.	g	16.8848	32.0453	36.0330
N/A	Net Wt. after 1000-1050 deg C.	g	0.0077	0.0052	0.0038

Step #	Description	Units	A	B	C	AVE	std.dev
NA	Wt.% Total Dissolved Solids	%	0.43%	0.52%	0.49%	0.48%	0.05%

			Liquid Fraction		
Step	Description	units	LA	LB	LC
N/A	Wt% Sample after 105 deg C	%	0.43%	0.52%	0.49%
		Ave	0.48%		
		Std. Dev.	0.05%		

			Liquid Fraction		
Step	Description	Units	LA	LA	LC
N/A	Wt% Oxides after 1050 deg C	%	0.26%	0.18%	0.13%
		Ave	0.19%		
		Std. Dev.	0.07%		

Analyst: [signature]

Review: [signature] 5/29/02

Battelle, PNNL / RPG / Organic Analysis … Chelator Data Report

Project / WP#:	42365 / W60656
ASR#:	6403
Client:	Sandra Fiskum
Total Samples:	1

RPL #	Client ID
02-2246	AZ-101-Cs-Eluate

Procedure: TP-RPP-WTP-049, *Ion Exchange for Activity Reduction*
TP-RPP-WTP-048 *Derivatization GC/FID Analysis of Chelators and Degradation Products*

M&TE Number:

Gas Chromatograph/Flame Ionization Detector	WD14807
Mettler PC4400 Balance	SN: 41100
Mettler AC100 Balance	SN: 821319

Analyst(s): BR Valenzuela and AM Aman

Analysis Date: 6/4,11/02

Analysis Files:

Calibration –	060402CH
Sample Analysis -	060402CH
	061102CH (reruns)

For Calibration and Maintenance Records, see Calibration Data Packet 060402CH and Instrument Logbook

Rhonda Skaggs for Blandina Valenzuela
Prepared By 7-29-02

Reviewed By 7/29/02

CHELATOR RESULTS

1. Sample Analysis
AZ-101-Cs-Eluate Results

Analyte	CAS #	MDL mg/L	Sample Results	
			02-2246 mg/L	Data Flag
Chelators and Degradation Products				
EDTA	60-00-4	4.9	4.9	U
HEDTA	150-39-0	8.8	8.8	UX
ED3A[(a)]	(b)	4.9	4.9	U
NTA	139-13-9	5.6	5.6	U
NIDA/IDA[(c)]	25081-31-6	11	11	U
Citric acid[(d)]	77-92-9	5.8	5.8	U
Succinic acid	110-15-6	6.1	21	JB
AA (surrogate)	124-04-9	(e)	61%[(e)]	

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) IDA completely converted to NIDA in the presence of nitrite in tank waste.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids.Value represents percent recovery of the surrogate standard.
(e) Value represents percent recovery of the surrogate standard; no MDL calculated.

Narrative

Analysis was performed on "AZ-101-Cs-Eluate" for chelators and chelator-degradation products. The chelators, ethylenediaminetetraacetic acid (EDTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), ethylenediaminetriacetic acid (ED3A), iminodiacetic acid (IDA), nitrosoiminodiacetic acid (NIDA), succinic acid, and nitrilotriacetic acid (NTA), have low volatility and high polarity precluding direct analysis by GC/FID. Derivatizing the chelators with a BF_3/methanol mixture results in a methyl ester product that is amenable to GC/FID separation and analysis. The derivatization process and analysis are still considered experimental.

A sample of "AZ-101-Cs-Eluate," was submitted for chelator analysis. The 5-gram (nominal) sub-sample of the AZ-101 eluate was diluted with 5 mL of DI water and subjected to an IX procedure, TP-RPP-WTP-049, *Ion Exchange for Activity Reduction* to reduce the sample dose. The diluted AZ-101 eluate sample was removed from the hot cell and derivitization of the sample according to procedure TP-RPP-WTP-048 *Derivatization GC/FID Analysis of Chelators and Degradation Products* was performed in a fume hood in 329. Adipic acid was added to 2-mL aliquots of each sub-sample (prior to derivatization step) as a derivatization monitor. Only succinic acid was found present in the sample. Quality control check standards relative to the sample preparation and instrument performance were also prepared and analyzed.

The EQL was calculated using the lowest calibration standard (10 mg/L; 20 mg/L for NIDA) and multiplied by the preparation dilution factor recorded for the batch preparation blank.

Historically, the EQL was 10 times the value of the estimated MDL. Therefore, for the current analysis the MDL will be calculated by dividing the EQL by 10. The result for succinic acid was above the MDL and below the EQL and is "J" flagged. Further, since succinic acid was detected in the PB at a concentration greater than 5% of the concentration measured in the sample, that result is also "B" flagged.

The results for HEDTA indicate high recoveries for the LCS. The results for reprepared and reanalyzed samples indicate high recovery as well. As a result, the data is X flagged and should be considered qualitative.

2. Quality Control Criteria

MS and MSD QC Results

Analyte	CAS #	MDL (mg/L)	02-1832 Average Native Amt. (mg/L)	Data Flag	Spiked Conc. (MS/MSD) (mg/L)	Analyzed Conc. (mg/L)	Matrix Spike (MS) % Rec	Analyzed Conc. (mg/L)	Matrix Spike Duplicate(MSD) % Rec.
Acceptance Criteria							*75-125*		*75-125*
Chelators									
EDTA	60-00-4	4.9	570		710/630	1000	**61**	1100	85
HEDTA	150-39-0	8.8	8.8	UX	1400/1300	990	**70**	1300	103
ED3A[(a)]	(b)	4.9	310		(c)		(c)		(c)
NTA	139-13-9	5.6	250		890/790	940	78	890	81
NIDA	25081-31-6	11	880		900[(d)]/800[(d)]	2000	121	1900	**131**
Citric Acid [(e)]	77-92-9	5.8	420		890/790	1200	91	1100	88
Succinic Acid	110-15-6	6.1	91	B	900/800	900	90	820	91
AA (surrogate)	124-04-9	(f)	99%[(f)]				99 [(f)]		106 [(f)]

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid;
ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid;
NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)
Bolded values denote acceptance criteria failures

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A not spiked into MS and MSD samples.
(d) Assumes spiked IDA completely converted to NIDA in the presence of nitrite in tank waste for spike recoveries.
(e) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(f) Value represents percent recovery of the surrogate standard; no MDL calculated.

Process Blank and LCS Results

Analyte	CAS #	MDL (mg/L)	Process Blank (mg/L)	Data Flag	Spiked LCS concentration (mg/L)	Analyzed LCS concentration (mg/L)	Data Flag	Lab Control (LCS/BS) % Rec.
Acceptance Criteria								*80-120*
Chelators								
EDTA	60-00-4	4.9	4.9	U	480	500		103
HEDTA	150-39-0	8.8	8.8	UX	970	1700	X	**170**
ED3A[(a)]	(b)	4.9	4.9	U	(c)	(c)		(c)
NTA	139-13-9	5.6	5.6	U	610	670		111
NIDA	25081-31-6	11	11	U	(c)	(c)		(c)
Citric Acid [(d)]	77-92-9	5.8	5.8	U	610	530		88
Succinic Acid	110-15-6	6.1	18	J	620	600		97
AA (surrogate)	124-04-9	(e)	**73%**[(e)]					**62** [(e)]

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)
Bolded values denote acceptance criteria failures

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A and NIDA not spiked into LCS samples; no nitrite from tank sample to convert IDA into NIDA.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(e) Value represents percent recovery of the surrogate standard; no MDL calculated.

Narrative

The preparation blank results meet the success criteria of <EQL.

For the liquid batch chelator preparation, AP-104 Supernatant (RPL#: 02-1832) was used for the matrix spike and duplicate. Of the seven analytes, EDTA and HEDTA results for the MS failed the spiked recovery criterion; however, the MSD result passed. An additional dilution was performed on the MSD for EDTA only; the MSD sample response was above the calibration curve and was diluted into range using a 1:2 dilution. An assumption was also made for the recovery results obtained for IDA in the MS and MSD. A known amount of IDA was spiked into the MS and MSD; it was assumed all of the IDA is converted to NIDA in the presence of nitrite found in the tank waste. The MSD percent recoveries on NIDA did not pass acceptance criterion; the opposite is true for the MS. The confirmation samples for NIDA, prepared at a later date (results reported below), did pass acceptance criteria. Note that both EDTA and NIDA were spiked at concentration levels that were <2x the native amount for each compound present in the sample. Therefore, recoveries will demonstrate a greater degree of variability for these compounds.

The LCS results passed the recovery acceptance criteria (80-120%) with the exception of HEDTA, which was quite a bit higher at 170% recovery.

Because of the failures observed with the MS and LCS percent recovery data, the QC samples were reprepared from the diluted tank sample obtained from 325 Hot Cell. A different analyst performed the derivatization of the samples to confirm or refute original observed results. The reprepared data confirmed the trends observed in the original results; with the MS EDTA producing a low recovery result. The LCS HEDTA percent recovery result was also confirmed as being higher than the acceptance criteria of 80-120%. One difference between the original and the confirmation results is the HEDTA recovery result for the MS and MSD. Originally, the recoveries for the MS/MSD were 70% and 103% respectively; the confirmation results produced slightly higher results with 135% (MS) and 155% (MSD). The reprepared sample results are shown in the tables below.

MS and MSD Confirmation Results (Reprepared Samples)

Analyte	CAS #	MDL (mg/L)	02-1832 Native Amt. (mg/L)	Data Flag	Spiked Conc. (MS/MSD) (mg/L)	Analyzed Conc. (mg/L)	Matrix Spike (MS) % Rec	Analyzed Conc. (mg/L)	Matrix Spike Duplicate(MSD) % Rec.
Acceptance Criteria							*75-125*		*75-125*
Chelators									
EDTA	60-00-4	4.9	630		710/630	1100	**65**	1100	**74**
HEDTA	150-39-0	8.8	8.8	UX	1400/1300	1900	**135**	2000	**155**
ED3A[(a)]	(b)	4.9	310		(c)		(c)		(c)
NTA	139-13-9	5.6	240		890/790	950	80	930	89
NIDA	142-73-4	11	880		900[(d)]/800[(d)]	1900	110	1800	111
Citric Acid [(e)]	77-92-9	5.8	620		890/790	1300	82	1300	83
Succinic Acid	110-15-6	6.1	91	B	900/800	940	94	860	96
AA (surrogate)	124-04-9	(f)	107%[(f)]				107 [(f)]		110 [(f)]

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid;
ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid;
NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)
Bolded values denote acceptance criteria failures

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A not spiked into MS and MSD samples.
(d) Assumes spiked IDA completely converted to NIDA in the presence of nitrite in tank waste for spike recoveries.
(e) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(f) Value represents percent recovery of the surrogate standard; no MDL calculated.

LCS Confirmation Results (Reprepared Samples)

Analyte	CAS #	MDL (mg/L)	Spiked LCS concentration (mg/L)	Analyzed LCS concentration (mg/L)	Data Flag	Lab Control (LCS/BS) % Rec.
Acceptance Criteria						*80-120*
Chelators						
EDTA	60-00-4	4.9	480	540		111
HEDTA	150-39-0	8.8	970	2200	X	**221**
ED3A[(a)]	(b)	4.9	(c)	(c)		(c)
NTA	139-13-9	5.6	610	720		118
NIDA	25081-31-6	11	(c)	(c)		(c)
Citric Acid [(d)]	77-92-9	5.8	610	560		93
Succinic Acid	110-15-6	6.1	620	640		104
AA (surrogate)	124-04-9	(e)				118 [(e)]

EDTA= ethylenediaminetetraacetic acid; HEDTA= N-(2-hydroxyethyl) ethylenediaminetriacetic acid; ED3A=ethylenediaminetriacetic acid; NTA= nitrilotriacetic acid; IDA=iminodiacetic acid; NIDA = nitrosoiminodiacetic acid; AA = adipic acid (for monitoring derivatization process)
Bolded values denote acceptance criteria failures

(a) ED3A results calculated using EDTA calibration curve.
(b) The CAS number is not available for ED3A.
(c) ED3A and NIDA not spiked into LCS samples; no nitrite from tank sample to convert IDA into NIDA.
(d) Citric acid was measured by using derivatization GC/FID for comparison with the IC method for organic acids. Citric acid mimics succinic acid behavior.
(e) Value represents percent recovery of the surrogate standard; no MDL calculated.

As discussed previously, HEDTA results are "X" flagged and should be considered qualitative only.

3. Recommendations

In the reaction of chelators with BF_3/methanol, methylation occurs at the carboxylic acid ligand sites. However, methylation does not occur at hydroxy sites in the reaction of BF_3/methanol, for example with HEDTA. The hydoxyethyl group of HEDTA resists methylation by BF_3/methanol and, instead, forms a cyclic, or intramolecular lactone with one of the neighboring ligand sites. Depending on the pH, the lactone accounts for only approximately 30% of the species detected. The trimethylated non-cyclic HEDTA with a free hydroxyethyl group accounts for the remaining 70% of the HEDTA. It is speculated that the non-cyclic species is simply too polar to migrate through the GC column (Lokken et al. 1986; Grant, Mong, Lucke, and Campbell 1996) and, hence, is not detected in this analysis.

Therefore, the recovery of HEDTA is greatly affected by the pH of the solution. A slight change in the final pH may greatly affect the recovery of HEDTA. This variation may be a reason for the high recoveries of HEDTA in the LCS and reprepared samples relative to the calibration standards which are prepared in the same manner.

To increase the reliability of this analytical method, further research is required to understand the variation in recoveries, particularly for HEDTA.

An isotope dilution approach where deuterated analogs of the chelators could be added to the solution prior to sample workup is recommended. Analysis of the samples would then be performed using GC/MS after derivatization. Additional compounds to monitor extraction and sample preparation performance may be advisable.

Existing techniques for EDTA and HEDTA using Cu complexation and ion-pair chromatography (W.R. Grace 1988) could be employed to further validate the performance of this method. This technique could also be used as a confirmatory method for sample matrices although substantial ALARA limitations exist when using liquid chromatographic methods. Additionally, preliminary results using capillary electrophoresis appear promising for the direct analysis of chelators and their associated degradation products without the use of derivatization.

References

Grant KE, GM Mong, RB Lucke, JA Campbell. 1996. Quantitative Determination of Chelators and Their Degradation Products in Mixed Hazardous Wastes from Tank 241-SY-101 Using Derivatization GC/MS. J. of Radioanalytical and Nuclear Chemistry, 211, 383-402.

Lokken RO, RD Scheele, DM Strachan, AP Toste. 1986. Complex Concentrate Pretreatment FY 1986 Progress Report. PNL-7687, Pacific Northwest Laboratory, Richland, Washington.

Organic Chemical Division, General Procedure for the Determination of NTA, HEDTA, EDTA, and DTPA in Chelater and Metal Chelate Mixtures. W.R. Grace and Company, Nashua, New Hampshire, RDSAP Number 0021, 1988, pg. 1.

Pacific Northwest National Laboratory (PNNL) // Battelle Northwest
Advanced Organic Analysis Group (AOAM)

Organic Anions by Ion Chromatography Report

Project / WP#: 42365 / W60656
ASR#: 6403
Client: Sandy Fiskum
Total Samples: 1

RPL #	Client ID
02-2246	AZ-101 CS Eluate

Procedure: TP-RPP-WTP-024, *MDL/EQL Evaluation for Organic Acids by Ion Chromatography in Sand, Water, and Tank Waste.*
(See narrative for adaptation descriptions)

M&TE Number: WD12888 Dionex IC Analyzer System
P37596 Mettler AE50 Balance

Analyst: GM Mong

Analysis Date: 6/19/02

Analysis Files: Data: gm0619E01, gm0620E1, gm0621E1
Standards: BNW14062, p. 47
Spreadsheets: IC Std 4062-47a asr6403
IC Std 14062-47b asr6403

GM Mong 8/7/02
Prepared By

Reviewed By (analysis, report) 8-7-02

Blandina Valenzuela 8-7-02
Reviewed By (QC data)

One sample, AZ-101 CS Eluate, was submitted for organic anion analysis as part of an aggregate of other liquid samples. The Radiological Processing Group (RPG) prepared the samples by dilution with 5 mLs of DI water and elution through a bed of Bio-Rad AG 50W-X8 cation exchange resin (50-100 mesh, sodium form) to relieve the samples of some of their fission product activity. Ion chromatography was performed with 2 separations by using 2 columns, which had different loading characteristics. The first separation, using a Dionex AS-15 column, was for the determination of glycolate and acetate. The second separation, using a Dionex AS-11 column, was for the determination of formate, oxalate, and citrate. The analytical dilutions of the ion exchange eluant (IEX) solution chosen, were those determined to be within the capacity of the columns. The dilutions of the IEX solutions were 400µL/25 mL (62.5x) for the AS-15 separation and 200 µL/25 mL (125x) for the AS-11 separation. The analytical dilutions were sample dependent and were a principle effect on the overall method MDL estimation.

The results were corrected for the density of both the sample (apparent density of 1.0002 g/mL) and the IEX solution (1.024 g/mL). All other dilutions were done on a v/v basis.

This work utilizes the QC acceptance criteria developed in test plan TP-RPP-WTP-024 MDL/EQL Evaluation for Organic Acids by Ion Chromatography in Sand, Water, and Tank Waste. The analyte list for this work differs substantially from that developed for the test plan; consequently, the analytical method has to be adapted to meet these new requirements. As part of batch QC, the MS/MSD (post spike) data is obtained from a similar sample from ASR-6378.

One of the requested analytes (gluconate) cannot be reliably determined by the analysis developed here. Two critical analytical hurdles prevent direct analysis for gluconate: (1) Gluconate suffers from low sensitivity to conductivity detection. (2) Gluconate co-elutes with other anions native to tank waste materials in the analytical systems used. For the AS-11 column, gluconate and fluoride are both nearly non-retained and co-elute. For the AS-15 separation, there is great selectivity for the weakly retained analytes fluoride, glycolate, and acetate; however, gluconate was found to co-elute with glycolate. This point will be addressed in the comments below

1. Analysis

The results from the analysis of the AZ-101 CS Eluate sample are provided in Table 1 below. The concentration values are given in mg/L and are corrected for the density of the liquid sample and IEX solution. The data reported are the average of duplicate injections. All analytes were found to be below the MDL.

Table 1. Organic Anion Results for AZ-101 CS Eluate

RPD success criteria: < 20% when > 10x MDL
RSD success criteria: < 15% when > 10x MDL

Sample ID	Glycolate[1] mg/L	Acetate mg/L	Formate mg/L	Oxalate mg/L	Citrate mg/L
AZ-101 CS Eluate	< 140 U	<110 U	< 190 U	< 250 U	< 480 U
MDL (est)[2]	140	110	190	250	480
EQL (est)[3]	420	330	570	740	1500
Preparation DF	24.5	24.5	24.5	24.5	24.5
Analysis DF	62.5	62.5	125	125	125
Total DF	1500	1500	3100	3100	3100
MDL (at the instrument)	0.087	0.069	0.059	0.076	0.15

DF = dilution factor
Explanation of flag: U: Analyte is not observed or the determination was below the MDL level
(1) In the AS-15 separation, glycolate and gluconate co-elute. Separate analysis found that the signal response for gluconate is approximately 25% - 38% of the signal response for glycolate. Thus, the results in this column could be interpreted as glycolate/gluconate. Quantitation was based on the calibration curve of glycolate.
(2) The estimated MDL is based on the MDL at the instrument (taken to be one-third of the LLS) multiplied by the total dilution factor.
(3) The estimated EQL is taken to be the LLS concentration multiplied by the total dilution factor.

The results reported for acetate and glycolate in Table 1 were determined by the AS-15 separation. Formate, oxalate, and citrate were determined by the AS-11 separation. The formate and oxalate results were < MDL for both separations.

2. Quality Control Sample Comments

Duplicate (DUP) and Triplicate (TRIP). No duplicate sample was created for this sample set. Batch quality control samples used for this analysis can be derived from ASR 6378 samples, which were analyzed at the same time. The ASR 6378 sample was prepared in triplicate, and the batch met the RSD success criteria of <15% and RPD criteria of <20% for all analytes. The ASR 6378 samples had reportable amounts of glycolate, acetate, formate, oxalate, and citrate.

Post Spike (PS) and Post Spike Duplicate (PSD). These samples cannot be successfully spiked in the matrix so that the included volume of spike would be <10% of the sample. Several of the analytes would exceed their respective solubilities in water if this exercise was undertaken. Therefore, a spike was added to the IEX solution delivered for analyses. The only QC available is therefore a **Post Spike**. The spike recoveries as well as the RPD between the PS and PSD meet the QC success criteria.

Post spike data was acquired as part of a larger batch of samples. The samples used for post spiking were from ASR 6378. The estimated MDL levels are those pertinent to the ASR 6378 samples.

Table 2. Organic Anion Results for the AP-104 Supernatant PS and PSD samples (ASR 6378)

Post Spike Success Criteria: 75% to 125% of expected value **PS and PSD batch data from ASR 6378, samples 02-01832**						
Analyte/Sample	**MDL* mg/L**	**Spike mg/L**	**Sample mg/L**	**Measured mg/L**	**Rec %**	**RPD %**
Glycolate PS	5.3	50	48	99	102	
Glycolate PSD		50	48	100	104	2
	mg/L	**mg/L**	**mg/L**	**mg/L**	**%**	**%**
Acetate PS	4.2	42	26	65	93	
Acetate PSD		42	26	69	102	9
	mg/L	**mg/L**	**mg/L**	**mg/L**	**%**	**%**
Formate PS	7.2	35	120	160	111	
Formate PSD		35	120	160	99	12
	mg/L	**mg/L**	**mg/L**	**mg/L**	**%**	**%**
Oxalate PS	9.3	43	24	70	108	
Oxalate PSD		43	24	67	100	7
	mg/L	**mg/L**	**mg/L**	**mg/L**	**%**	**%**
Citrate PS	18	86	28	106	91	
Citrate PSD		86	28	104	88	4

*MDL's are derived from the instrument MDL multiplied by the dilution factor applied to the post spike

Preparation Blank (PB) and Laboratory Control Standard (LCS).
Two LCS samples were prepared. LCS-1 contained glycolate, acetate, formate, oxalate, citrate, and gluconate. LCS-2 contained glycolate, acetate, formate, oxalate, and citrate only. These samples were constructed to demonstrate the co-elution problems associated with gluconate; the LCS-1 sample contained 8.7 times more gluconate than glycolate. In the AS-15 analysis gluconate and glycolate co-elute. There is no discernable retention time difference between fluoride and gluconate in the AS-11 analysis.
The LCS yield data (for LCS-1 below) indicates the co-elution of glycolate and gluconate .

LCS data is reported as the concentration delivered to the hot cell. LCS data is assembled from the AS-15 and AS-11 data in the same fashion that PS, PSD, analytical samples, and other QC are obtained. The LCS data clearly demonstrates the overlap between gluconate and glycolate. With this exception, the PB and LCS data all meet the success criteria.

Table 3. Organic Anion Results for the PB and LCS

PB success criteria: < EQL LCS success criteria: 80% to 120% Recovery					
Sample ID	**Glycolate mg/L (Rec)**	**Acetate mg/L (Rec)**	**Formate mg/L (Rec)**	**Oxalate mg/L (Rec)**	**Citrate mg/L (Rec)**
PB	<130 U	< 110 U	< 180 U	< 230 U	< 460 U
LCS-1 measured	**21000 (420%)**	3900 (90%)	4900 (106%)	3500 (101%)	10000 (99%)
LCS-1 expected	4900	4400	4600	3500	10000
LCS-2 measured	7200 (98%)	4500 (103%)	5000 (95%)	3600 (108%)	11000 (99%)
LCS-2 expected	7300	4400	5300	3400	11000

Explanation of flag: U: analyte is either not observed or the determination was below the included MDL level.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Standards.
ICB and CCB standards met the success criteria. All analytes were below the MDL levels shown below. Since the analysis was done using two column separations, two sets of ICB and CCB data were compiled for the table below.

Table 4. Organic Anion Results for the ICB and CCB Standards

ICB/CCB success criteria: < MDL (at the instrument)					
Sample ID	**Glycolate mg/L**	**Acetate mg/L**	**Formate mg/L**	**Oxalate mg/L**	**Citrate mg/L**
MDL	0.087	0.069	0.059	0.076	0.15
ICB	< 0.087 U	< 0.069 U	< 0.059 U	< 0.076 U	< 0.15 U
CCB	< 0.087 U	< 0.069 U	< 0.059 U	< 0.076 U	< 0.15 U

Explanation of flag: U: analyte is either not observed or the determination was below the included MDL level.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) Standards.

The ICB/CCV analysis met the success criteria, with the exception of the AS-11 formate, which fell just outside of the acceptable criteria. This failure has no impact on overall data quality. Since the analysis was done using two column separations, two sets of ICV and CCV data were examined.

ICV/CCV success criteria: 90% to 110% Recovery (at the instrument)					
Sample ID	**Glycolate mg/L (Rec)**	**Acetate mg/L (Rec)**	**Formate mg/L (Rec)**	**Oxalate mg/L (Rec)**	**Citrate mg/L (Rec)**
ICV-AS11 measured	n.a.	n.a.	0.49 (101%)	0.53 (104%)	1.30 (109%)
ICV-AS11 expected			0.48	0.50	1.20
CCV-AS11 measured	n.a.	n.a.	**0.48 (111%)**	0.55 (103%)	1.10 (104%)
CCV-AS11 expected			0.43	0.54	1.10
ICV-AS15 measured	0.58 (96%)	0.64 (103%)	0.51 (105%)	0.50 (99%)	n.a.
ICV-AS15 expected	0.61	0.62	0.48	0.50	
CCV-AS15 measured	0.70 (105%)	0.56 (96%)	0.47 (109%)	0.54 (100%)	n.a.
CCV-AS15 expected	0.66	0.58	0.43	0.54	
n.a. = not applicable					

Low-Level Standard (LLS).

The LLS met the success criteria. Since the analysis was done using two separations, two sets of data were examined. The LLS level is comparable to the EQL level. By examination of the integrated area of the LLS analytes, the MDL level is approximated by 1/3 of the LLS or EQL level. This criteria is applied to all data released by this laboratory.

Table 6. Organic Anion Results for the LLS

LLS success criteria: 75% to 125% Recovery (at the instrument)					
Sample ID	**Glycolate mg/L (Rec)**	**Acetate mg/L (Rec)**	**Formate mg/L (Rec)**	**Oxalate mg/L (Rec)**	**Citrate mg/L (Rec)**
LLS-AS11 measured	n.a.	n.a.	0.18 (103%)	0.23 (106%)	0.45 (104%)
LLS-AS11 expected			0.17	0.22	0.43
LLS-AS15 measured	0.26 (98%)	0.21 (88%)	0.15 (85%)	0.20 (92%)	n.a.
LLS-AS15 expected	0.27	0.23	0.17	0.22	
n.a. = not applicable					

ASR 6566

^{238}Pu and $^{239+240}Pu$ Re-analysis

Battelle Pacific Northwest National Laboratory
Radiochemical Chemical Science & Engineering -325 Building

10/11/2002

Client : Fiskum
ASR: 6566

Cognizant Scientist: L R Greenwood Date : 10/11/02

Concur : T Trang-Le Date : 10/11/02

Procedure: PNL-ALO-417& 496 for Pu & Am

Measured Activities (uCi/g) with 1-sigma error

RPL ID Client ID	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %
02-3342 PB Process Blank	2.04E-6 24%	8.47E-6 11%	<6.E-7
02-3342 AZ102C (feed)	7.91E-3 3%	8.96E-4 5%	<2.E-5
02-3342 DUP AZ102C (feed)	6.47E-3 2%	6.28E-4 5%	<9.E-6
RPD	20%	35%	

Measured Activities (uCi/ml) with 1-sigma error

RPL ID Client ID	Pu-239+ Pu-240 Error %	Pu-238 Error %	Pu-236 Error %
02-3343 AZ102C-EC-D	3.16E-3 4%	3.19E-4 5%	<3.E-6
02-3344 PB Process Blank	<6.E-6	<7.E-6	<7.E-6
02-3344 AZ102C-CsE-Comp	1.56E-2 3%	1.85E-3 6%	<4.E-5
02-3345 AZ101-CsEluate-Comp	1.61E-2 3%	1.74E-3 7%	<5.E-5
02-3346 RZ101-CsEluate-Comp	2.47E-3 4%	3.20E-4 12%	<4.E-5
02-3347 AZ101-CsEluate-Comp	8.82E-3 4%	1.02E-3 10%	<6.E-5
02-3347 Lab DUP AZ101-CsEluate-Comp	8.56E-3 3%	8.14E-4 8%	<3.E-5
RPD	3%	22%	
Matrix Spike 3347 Matrix Spike 3250	108% 111%		
Reagent Spike	112%		
Lab Blank #1 Lab Blank #2	<5.E-8 <6.E-8	<5.E-8 <5.E-8	<5.E-8 <4.E-8

Appendix E

Cesium Ion Exchange and Batch Contacts Testing Personnel

Appendix E: Cesium Ion Exchange and Batch Contacts Testing Personnel

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