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**Small Column Ion Exchange Testing
of Superlig 644 for Removal of ^{137}Cs
from Hanford Tank Waste Envelope A
(Tank 241-AW-101)**

D. E. Kurath
D. L. Blanchard
J. R. Bontha

June 2000

Prepared for BNFL, Inc.
under Contract W375-LC-98-4168

PNWD-3001

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Battelle, Pacific Northwest Division
Richland, Washington 99352

SUMMARY

The current BNFL Inc. flow sheet for the pretreatment of the Hanford High-Level tank wastes includes the use of Superlig® materials for the removal of ^{137}Cs from the aqueous fraction of the waste. The Superlig® materials applicable to cesium removal include the cesium selective Superlig®632 and Superlig®644. These materials have been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

The work contained in this report involves testing the Superlig®644 ion exchange material in a small dual column system (15 mL each; L/D = 5.7). The sample processed was approximately 2.5 L of diluted waste ($[\text{Na}^+] = 4.6 \text{ M}$) from Tank 241-AW-101 (Envelope A). This waste had been previously clarified in a single tube cross-flow filtration unit. All ion exchange process steps were tested including resin bed preparation, loading, feed displacement, water rinse, elution and resin regeneration. During the initial run, the lag column did not perform as expected so that the ^{137}Cs concentration in the effluent composite was above the LAW treatment limits. This required a second column run with the partially decontaminated feed that was conducted at a higher flow rate.

A summary of performance measures for both runs is shown in Table S1. The Cs λ values represent a measure of the effective capacity of the SL-644 resin. The Cs λ of 143 for the lead column in run 1 is very similar to the value obtained by the Savannah River Technology Center during Phase 1A testing. The larger Cs λ value for run 2 reflects a general trend for the effective capacity of the SL-644 material to increase as the cesium concentration decreases. The low value for the lag column during the first run indicates that it did not perform as expected. This may have been due to insufficient conditioning of the bed prior to the start of the loading step or to air in the bed that caused channeling. Equilibrium data obtained with batch contacts using the AW-101 Cs IX feed indicates a range for the Cs λ of 80-97. The maximum decontamination factor (DF) for ^{137}Cs is based on analysis of the first samples collected from each column and the concentration in the feed for each run. While the DF's are lower for the second run, this is attributed to the lower ^{137}Cs concentration in the feed and the increased flowrate. The overall composite DF for run 2 was quite good since both columns functioned well. The overall DF for both runs was 3000, which provided an effluent with a ^{137}Cs concentration of $5.89\text{E-}02 \text{ Ci/m}^3$ ($\text{C/C}_0 = 3.31\text{E-}04$). The ^{137}Cs concentration in the effluent composite was 7.3% of the contract limit for ^{137}Cs and also below the basis of design limit.

Table S1. Summary of Performance Measures

	Flow rate (BV/hr)	Cs λ		Composite DF	Maximum ^{137}Cs DF	
		Lead column	Lag column		Lead column	Lag column
Run 1	6.0	143	25	8	11,600	47,100
Run 2	8.4	185	NA	355	99	1,130

The elution of the lead column after run 1 proceeded very well with the majority of the ^{137}Cs contained in the fourth bed volume of eluant (0.5 M nitric acid). The peak ^{137}Cs concentration in this sample was 97 times the ^{137}Cs concentration in the feed.

TERMS AND ABBREVIATIONS

BNFL	BNFL, Inc; subsidiary of British Nuclear Fuels, Ltd.
BV	Bed Volume
C/C ₀	analyte concentration in column effluent divided by analyte concentration in feed
DF	decontamination factor
DI	deionized water
DL	detection limit
E	elution
ER	elution rinse
F	feed
FD	feed displacement
GEA	gamma energy analysis
HLW	high-level waste
HLRF	High Level Radiation Facility
IC	ion chromatography
ICP AES	inductively coupled plasma/atomic emission spectrometry
ICP MS	inductively coupled plasma/mass spectrometry
LAW	low-activity waste
L/D	bed length (height) to diameter ratio
MDL	method detection limit
MRQ	minimum reportable quantity
NMRQ	no minimum reportable quantity
R	regeneration
SAL	Shielded Analytical Laboratory
SRTC	Savannah River Technology Center
TIC	total inorganic carbon
TOC	total organic carbon
TRU	transuranic

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1.0 INTRODUCTION

The current BNFL Inc. flow sheet for the pretreatment of the Hanford High-Level tank wastes includes the use of Superlig® materials in a dual column system for the removal of ¹³⁷Cs from the aqueous fraction of the waste. The Superlig® materials applicable to cesium removal include the cesium selective Superlig®632 and Superlig®644. These materials have been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

The work contained in this report involves the small column testing of the Superlig®644 ion exchange material¹. The sample processed was approximately 2.5 L of diluted waste (@ 4.6 M Na) from Tank 241-AW-101 (the 241 prefix, which is common to all Hanford tanks, will not be used hereafter). This waste had been previously clarified in a single tube cross-flow filtration unit (Brooks et al.). The ion exchange process steps tested include resin bed preparation, loading, feed displacement, water rinse, elution and resin regeneration.

The objectives of this work were to:

- Demonstrate the ¹³⁷Cs decontamination of Envelope A (Tank AW-101) and provide a cesium decontaminated sample for downstream process testing (i.e. ⁹⁹Tc removal, corrosion testing, Low Activity Waste (LAW) melter feed testing and LAW vitrification).
- Demonstrate the effectiveness of all SL-644 ion exchange process steps including loading, feed displacement, DI water washing, elution and resin regeneration.
- Obtain process performance data for SL-644 at conditions different than those previously tested.
- Investigate exchanger/waste chemistry.
- Investigate the potential for exchanger fouling.

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¹ The results presented in this report are based on work conducted under test plan TP-29953-3 Rev 0 Ion Exchange Testing for BNFL Inc, December 1998, and test instructions TI-29953-016, Test Instructions for Cesium Removal Column Testing from Envelope A, TI-29953-042 Test Instructions for Cesium Removal Column Testing from Envelope A: Run 2, and BNFL-TP-29953-048, Preparing a Composite Solution of the acid eluant Samples from AW-101 Cs Ion Exchange Column 1 for Analysis. Some data are recorded in Laboratory Record Book BNW 13687. Conditions for conducting these tests were given in the "Ion Exchange Test Specification Rev 1," TSP-W375-99-00013.

2.0 EXPERIMENTAL

2.1 Ion Exchange Column System

A schematic of the ion exchange column system is shown in Figure 2.1. The system consists of two small columns containing the ion exchange material, a small metering pump, four valves, a pressure gauge and a pressure relief valve. Valve 1 is a five-way valve that allows switching between the waste feed and various process solutions. Valves 2,3 and 4 are three-way valves that can be turned to a flow position, a sample position or a no flow position. Valve 2 is placed at the outlet of the pump and is used to eliminate air from the system, purge the initial volume of the system or isolate the columns from the pump. Valves 3 and 4 are primarily used for obtaining samples and may also be used to isolate the columns from the rest of the system. The columns are Kontes Chromaflex chromatography columns made of glass with adjustable plungers on the bottom and the top. The inside diameter of the columns is 1.5 cm which corresponds to a volume of 1.77 mL/cm of length. The connecting tubing is ¼-in OD (1/8-in ID) polyethylene. The columns are connected in series with the first column referred to as the lead column and the second column referred to as the lag column. The pump is a FMI piston pump with the flow rate controlled from outside of the hot cell with a FMI stroke rate controller. The pump was calibrated with the stroke rate controller and can provide pumping rates from 0-120 mL/hr. The volume actually pumped is determined using the mass of the fluid and the fluid density. The pressure relief valve is set at 40 psi, which is below the maximum operating pressure of 45 psi for the columns. The pressure indicated on the pressure gauge remained below 5 psi during the run. The total holdup volume of the Cs IX system was estimated to be 60 mL (4 BV) with the holdup volume to valve 2 being 30 mL (2 BV).

2.2 SL-644 Resin and Bed Preparation

Some properties of the as-received, hydrogen form of the SL-644 resin (batch # = 644BZ) are shown in Table 2.1. The Battelle values at room temperature and at 85°C are duplicated from Kurath, et al., 1999, while all of the SRTC data is reproduced from (Hassan, et al., 1999). The F factor is the ratio of the dry mass of exchanger to the initial mass of the exchanger. This was obtained by drying approximately 0.5 g in an oven at the temperature of interest until there was no significant change in mass. The as-received bulk density was determined by weighing approximately 10 mL of exchanger in a 50 mL graduated cylinder. The dry bulk densities were obtained by multiplying the as-received bulk density by the appropriate f factor. A particle density of 1.611 g/mL was reported by Hassan, et al., 1999.

Table 2.1. Physical Properties of the As-Received Hydrogen Form of SL-644

	Battelle	SRTC
As-received bulk density (room temperature)	0.86 g/mL	0.88
Dry as received bulk density (85°C)	0.78 g/mL	-
Dry as received bulk density (95°C)	0.69 g/mL	0.793 g/mL
F factor @ 85°C (% water)	0.907 (9.3%)	-
F factor @ 95°C (% water)	0.80 (20%)	0.9 (10%)

The particle size distribution for the as-received SL-644 resin subsample (Kurath, et al., 1999) is compared to the subsample used in column experiments by SRTC (Hassan, et al., 1999) in Table 2.2. It is apparent that the average particle size of the Battelle subsample is smaller than that of the SRTC subsample. The data are not precise enough to allow an accurate estimate of the average particle diameter but the average apparently lies between 210 and 425 microns with a rough estimate of 330 microns.

Table 2.2. Particle Size Distribution of the As-Received, Hydrogen Form of SL-644

Sieve size	Size (μm)	Battelle subsample (wt %)	Size (μm)	SRTC subsample (wt%)
	>600	not measured	>600	14
40	>425	9.9	600-425	44
70	425-210	88	425-210	40
100	210-149	2.1	210-62	0
200	149-74	0		
	<74	0		

The SL-644 resin and bed preparation was performed using a procedure similar to that recommended by Hassan et al. 1999, with the exception that the solution volumes and flow rates were larger. Prior to packing the resin bed, two 7.9 g batches of the as-received SL-644 resin were placed in separate beakers. The resin was soaked in about 70 mL of 1 M NaOH with light agitation for 2.5 hours. The NaOH was decanted and the SL-644 was washed 3 times with 100 mL of DI water per wash. The resin was then slurried into the columns in DI water. The swollen resin volume was about 22 mL and was greater than required. Approximately 32% of the slurried resin volume was removed leaving about 5.4 g of as-received resin per column. The F factor (ratio of the mass of damp resin to dried resin) was determined to be 0.80 at 95°C so the amount of SL-644 in each column was about 4.3 g on a dry basis. Prior to installation of the system into the hot cell, both of the resin beds were cycled through the acid form with the solution amounts shown in Table 2.3. The solution volumes were significantly in excess of those planned for full-scale operation in order to ensure the system was completely flushed and that the resin beds were fully conditioned. The total system hold-up volume was 60 mL.

Table 2.3. Shakedown Testing and Bed Conditioning Parameters

	Solution	Volume	Contact Time
Elution	0.5 M HNO ₃	> 6 BV (>90 mL)	> 1 hr
Eluant rinse	DI water	> 6 BV (>90 mL)	> 1 hr
Regeneration	0.25 M NaOH	> 6 BV (>90 mL)	> 1 hr
DI water wash	DI water	> 6 BV (>90 mL)	> 1 hr

Figure 2.1. Cesium Ion Exchange Column System

A number of the resin bed properties determined during the testing discussed in this report are shown in Table 2.4. The bed volume definition is based on the volume of the SL-644 resin in each column during the loading step. The corresponding bed height and bed length (height) to diameter ratio (L/D) is also given. The expanded particle size diameter was estimated on the basis of the bed volume expansion relative to the as-received bed volume and the initial rough estimate of the average particle size of the resin (330 μm). The average number of resin particles per column diameter is greater than the 20-30 particle diameters commonly assumed necessary to prevent channeling.

Table 2.4. Resin Bed Properties

	Lead and Lag Columns
Bed volume, mL	15
Bed height - cm	8.5
L/D	5.7
Expanded particle size diameter - μm	440
Resin particles per column diameter	34

2.3 Feed Preparation

A sample of the waste from Tank AW-101 (Envelope A) was received in the High Level Radiation Facility (HLRF) during the 4th quarter of 1998. The homogenization, dilution, and subsampling of this sample is described in Urie (1999). The diluted AW-101 sample was then processed in a single tube cross-flow filter to remove entrained solids using a 0.1 micron sintered metal Mott filter (Brooks et al., 1999). The diluted feed added to the filtration unit had a sodium concentration of 6.5 M as determined by ICP-AES (Urie, 1999). Due to dilution from residual water in the filtration unit, the permeate was determined to have a sodium concentration of 6.05 M (Brooks et al. 1999). The clarified AW-101 sample was then transferred in two containers from the HLRF to the shielded analytical laboratory (SAL) hot cells. The total volume transferred was about 2.1 L. The contents of the two transfer containers were combined and homogenized and the sample was diluted with DI water to a target concentration of 4.6 M sodium in preparation for the cesium ion exchange testing. The density of the cesium ion exchange feed was determined with a 25 mL volumetric flask and a 4 place analytical balance.

2.4 Experimental Procedure and Conditions

The experimental conditions for each process step for Run 1 are shown in Table 2.5. In general the flow rates were maintained as close as possible to the recommended values² but in some steps the total volume of the process solutions was increased in an attempt to ensure adequate flushing of the system. In particular, the volume of bed conditioning solutions was increased because the lag column did not appear to be shrinking or swelling as much as the lead column. As will be shown in Section 3.2, the lag column did not perform as expected, possibly because of inadequate bed conditioning. The total lead column eluant volume was also increased in order to achieve the cutoff criteria of $C/C_o = 0.01$ for elution. The lag column was eluted at higher flow rate since the objective was to remove the cesium from the column to prepare for a second run. Samples were not taken during elution of the lag column. The bed conditioning loading, feed displacement and DI water rinse steps were conducted by passing these solutions through both resin beds connected in series. The elution, elution rinse and the regeneration steps were conducted on each column separately.

² Ion Exchange Testing for BNFL Inc, BNFL-TP-22953-003, December 1998, transmitted to BNFL via letter 29953-023, EV Morrey to ME Johnson.

Table 2.5. Experimental Conditions for Run 1

Process Step	Solution	Total Volume, Bed Volumes (mL)	Flow Rate in Bed Volumes Per Hour (mL/hr)	Time hr
Bed conditioning	0.5 M nitric	6 (90)	4.4 (66)	1.4
Bed conditioning	DI water	8 (120)	8 (120)	1
Bed conditioning	0.25 M NaOH	17.9 (270)	7.8 (117)	2.3
Loading	AW-101 Feed	164 (2455)	6 (90)	27.6
Feed displacement	0.1 M NaOH	3 (45)	3 (45)	1
DI water rinse	DI water	6.7 (100)	3.1 (47)	2.1
Elution of lead column				
Elution (lead col)	0.5 M HNO ₃	13 (195)	1.2 (18)	10.8
Eluant rinse (lead)	DI water	7.1 (106)	3 (45)	2.4
Regeneration (lead)	0.25 M NaOH	6.2 (93)	5.9 (89)	1.1
Elution of lag column				
DI water rinse (lag col)	DI water	4 (60)	8 (120)	0.5
Elution (lag col)	0.5 M HNO ₃	12 (180)	3 (45)	4
Eluant rinse (lag col)	DI water	4 (60)	8 (120)	0.5
Regeneration (lag col)	0.25 M NaOH	6 (90)	8 (120)	0.75

Note: The column volume is based on the volume in the columns at the start of the loading step. The bed volume in both columns was 15 mL with a height of 8.5-cm.

Since insufficient cesium decontamination was obtained during Run 1, a second run utilizing the partially decontaminated AW-101 sample from Run 1 was conducted. The experimental conditions for each process step for Run 2 are shown in Table 2.6. In general the flow rates were higher than those in Run 1 and samples were taken only during the loading phase.

The adjustable plungers at the top of each column were used to minimize the volume of solution above each of the resin beds. The bed volumes changed dramatically from a minimum volume in the acid form during elution (9-10 mL) to a maximum volume of about 21 mL in the sodium form in contact with water. The plungers were moved downward at the end of each process step in which the resin beds shrank and were moved upward periodically as the bed expanded. The plungers did not compress or constrain the resin beds. The height of liquid above the beds was kept to less than 1 cm (about 1.8 mL).

Table 2.6. Experimental Conditions for Run 2

Process Step	Solution	Total Volume, Bed Volumes (mL)	Flow Rate in Bed Volumes Per Hour (mL/hr)	Time hr
Bed conditioning	0.25 M NaOH	7.7 (116)	4.7 (71)	1.6
Loading	AW-101 Feed	152 (2280)	8.4 (126)	18.2
Feed displacement	0.1 M NaOH	6.3 (95)	5.8 (87)	1.1
DI water rinse	DI water	5.4 (80)	5.4 (80)	1
Elution	0.5 M HNO ₃	21 (310)	3-4 (45-60)	5.5
Eluant rinse	DI water	7 (105)	6.4 (96)	1.1
Regeneration	0.25 M NaOH	12.3 (185)	6.5 (98)	1.9

Note: The column volume is based on the volume in the columns at the start of the loading step. The bed volume in both columns was 15 mL with a height of 8.5-cm.

The sampling and analysis protocol is shown in Tables 2.7 and 2.8 for Run 1 and Run 2, respectively. The ¹³⁷Cs content was determined in all of the samples using a portable GEA instrument. This allowed near real time analysis of the samples. The response time was limited by the rate at which samples could be removed from the hot cell and was often on the order of hours. Due to the dose rate from ¹³⁷Cs, many of the samples required dilution prior to removal from the hot cell. Samples were diluted in either 0.25 M NaOH or 0.5 M HNO₃. The extent of dilution was determined by mass difference on a 4 place analytical balance. The samples that required dilution included the feed sample, the effluent samples from the last half of the loading phase of Run 1 and most of the eluant samples. The ¹³⁷Cs results from the portable GEA instrument were confirmed by analyzing selected samples with the GEA instrument in the analytical laboratory. The sodium and other metal concentrations were determined with ICP-AES. The OH⁻ concentration was determined by titration with hydrochloric acid.

During the loading phase, the treated effluent was collected in one of two effluent bottles except for the small (2 mL) samples that were taken. A composite sample from each of the two effluent bottles was taken for analysis by GEA. The rest of the samples were collected in approximately 1 BV aliquots. Once the GEA results were confirmed for the eluate, all of the eluate samples were composited and a sample of the composite was submitted for GEA, ICP-AES, TOC and ICP-MS for ⁹⁹Tc.

Table 2.7. Sampling Interval and Analyses for Run 1

Process Step	Lead Column BV	Lag Column BV	Approximate Sample Size (mL)	Analyses
Bed conditioning	-	-	-	-
Bed conditioning	-	-	-	-
Bed conditioning	-	-	-	-
Loading	Every 10 BV	Every 20 BV	2	GEA
Feed displacement	-	Every 1 BV	15	ICP, GEA, OH-
DI water rinse	-	Every 1 BV	15	ICP, GEA, OH-
Elution	Every 1 BV	-	15	GEA
Eluant rinse	Every 1 BV	-	15	GEA
Regeneration	1 composite	-	90	ICP, GEA, OH-
Composite Samples				
Effluent - 1			2	GEA
Effluent - 2			2	GEA
Eluate	1 composite	-	7	ICP-AES, GEA, TOC, ICP-MS (Tc)

Table 2.8. Sampling Interval for Run 2

Process Step	Lead Column	Lag Column	Approximate Sample Size (mL)	Analyses
Bed conditioning	-	-	-	-
Loading	Every 20 BV	Every 20 BV	2	GEA
Feed displacement	-	-	-	-
DI water rinse	-	-	-	-
Elution	-	-	-	-
Eluant rinse	-	-	-	-
Regeneration	-	-	-	-

2.5 Batch Contacts

A number of batch contacts between the SL-644 and subsamples of the AW-101 feed were conducted to obtain equilibrium data. Portions of the subsamples were spiked with 0.1 M CsNO₃ stock solutions to obtain additional concentrations of cesium. The initial cesium concentrations in the AW-101 subsamples are given in Table 2.9. The purpose of these spikes was to ensure that the equilibrium composition of the solutions bracketed the cesium feed concentration.

Table 2.9. Initial Cs Concentrations in the AW-101 Solutions Used for the Batch K_d Tests

Solution	Initial Cs Conc. [M]	Initial Na/Cs (a)	Initial K/Cs (a)
Un-spiked	6.14E-05	74,700	6,350
Cs Spike 1	3.67E-04	12,500	1,060
Cs Spike 2	6.17E-04	7,440	632

(a) Na⁺ and K⁺ are the primary cations that compete with Cs⁺ for ion exchange with SL-644. Concentrations of Na⁺ and K⁺ in the AW-101 solution were 4.59M and 0.39M respectively.

The batch K_d tests were performed at a phase ratio of approximately 100 mL/g (liquid volume to exchanger mass). Typically, 0.05 g of exchanger was contacted with 5 mL of solution. The exchanger mass was determined to an accuracy of 0.0001 g. The waste volume was transferred by pipette, and the actual volume was determined by mass difference with an accuracy of 0.0001 g and the solution density. Agitation was provided by an orbital shaker set at 200 rpm for approximately 96 h. The temperature was not controlled, but was generally constant at 24°C during the 4 days of contact.

All K_d measurements were made in duplicate and blank samples (i.e., without the ion exchange resin) were used to determine the initial concentration of the species of interest. All initial and final solutions were analyzed by GEA to determine the ¹³⁷Cs concentration. The initial sodium and potassium concentrations were determined with ICP-AES.

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

$$K_d = \frac{(C_0 - C_1) * V}{C_1 * M * F} \quad (1)$$

where C₀ and C₁ are the initial and final ¹³⁷Cs concentrations, respectively, V is the volume of the liquid sample (mL), M is the mass of the (as received) ion exchanger (g), and F is the mass of the dried resin divided by the mass of the as-received resin.

3.0 RESULTS AND DISCUSSION

3.1 Feed Composition

The composition of the feed to the cesium ion exchange system is shown in Table 3.1. The concentrations of Na, K, Al, Cr, and P were determined by analysis of a feed sample by ICP-AES. The total cesium concentration is estimated using the GEA results for ^{137}Cs and the isotopic ratios determined with thermal ionization mass spectroscopy and reported in Urie (1999). For the AW-101 sample the ^{137}Cs :total cesium was 0.2465. Some of the anion concentrations are based on an analysis of the Tc IX feed. Some of the component concentrations (generally the anions) are estimated on the basis of the known sample dilution from component concentrations given in the characterization report (Urie, 1999). The feed had a light yellow color that has been attributed to the presence of chromate.

Table 3.1. Composition of Envelope A (AW-101) Cs IX Column Feed ^(c)

Cations, M	
Na^+	4.59
K^+	0.39
Cs^+	6.14E-5
Na/Cs mole ratio	74,700
K/Cs mole ratio	6,350
Anions, M	
AlO_2^- ^(b)	0.411
Cl^-	0.066 ^(a)
CO_3^{2-}	0.13 ^(a)
CrO_4^{2-} ^(b)	8.4E-04
NO_2^-	1.01
NO_3^-	1.50
OH^-	2.17 ^(a)
PO_4^{3-} ^(b)	0.007
SO_4^{2-}	0.0072
Oxalate	<8.6E-3 ^(a)
Radionuclides	
^{134}Cs ($\mu\text{Ci/mL}$)	3.4E-2
^{137}Cs ($\mu\text{Ci/mL}$)	177.5
Solution Density, g/mL	1.228
a) These values have been estimated from the diluted feed characterization data reported in PNWD-2463, BNFL-RPT-003, Rev 0. Unless otherwise noted the results are based on direct analysis of the feed. b) Al, Cr, and P determined by ICP-AES. Anionic form is assumed on the basis of waste chemistry. c) The raw analytical results may be found in the appendix.	

3.2 Loading (^{137}Cs Breakthrough Curves), Feed Displacement and Water Wash

After the resin beds were conditioned, the loading phase was initiated with the diluted AW-101 sample. During the initiation of the loading step, the initial 4 bed volumes^(a) of effluent were not collected. This prevented the estimated holdup of 4 bed volumes of 0.25 M NaOH in the system from being mixed with the AW-101 effluent. Small samples (about 2 mL) were collected from the lead column every 10 BV of feed and from the lag column every 20 BV of feed. Initial samples were collected from both columns soon after loading was initiated in order to obtain a determination of the maximum decontamination factors. The loading phase generally went well except for some leakage of air into the lag column. This was observed by the dropping of the air-liquid interface above the top of the bed. At approximately 45 BV the loading was stopped for slightly over an hour to remove air from the column system. The air did not appear to get into the resin beds and there was no discernable discontinuity in the breakthrough curve.

The cesium effluent concentrations from the columns are shown in Figure 3.1 as C/C_0 vs the bed volumes of feed processed through each column. The C_0 value for ^{137}Cs was determined to be 177.5 $\mu\text{Ci/mL}$. The C/C_0 is plotted on a probability scale (i.e. gaussian distribution) since this tends to provide a straight line breakthrough curve. The C/C_0 values for the feed displacement (3 BV of 0.1 M NaOH) and DI water rinse (6 BV) are also shown in Figure 3.1. These exhibit a steadily decreasing concentration of ^{137}Cs . Most of the C/C_0 values were determined with a portable GEA instrument and selected samples were independently analyzed with a laboratory GEA instrument. The two analyses are generally in good agreement (within 10%). Raw analytical results and calculations may be found in the appendix.

Two ^{137}Cs -removal limits are also shown in Figure 3.1. The higher C/C_0 value of 0.45% corresponds to the contractual limit of 3 Ci/m^3 for ^{137}Cs in the low-activity waste (LAW) glass, and the lower C/C_0 value of 4.5E-02% corresponds to the basis of design target of 0.3 Ci/m^3 for ^{137}Cs in the LAW glass, which is 10 times less than the contractual limit. The C/C_0 values corresponding to these limits are determined using the sodium concentration of 4.59 M in the AW-101 sample, the ^{137}Cs feed concentration of 177.5 $\mu\text{Ci/mL}$, a 20 wt% total Na_2O loading in the glass, and a glass product density of 2.66 g/mL .

Volume corrections have been made in the analysis of the data so that the indicated data points correspond to the bed volumes that have actually passed through each column at the time of sampling. These volumes are different from the amount of feed pumped from the feed container because of the holdup volume. The bed volumes actually pumped from the feed vessel can be estimated by adding 2 bed volumes for the lead column and 4 bed volumes for the lag column.

The $\text{Cs } \lambda$ value is 143 for the lead column and an estimated (by extrapolation) 25 for the lag column. The $\text{Cs } \lambda$ value is the point at which the C/C_0 value is 50% (0.5) and is a direct indicator of the effective capacity of the resin. The $\text{Cs } \lambda$ value of 143 for the lead column is consistent with the estimated $\text{Cs } \lambda$ of 145-150 obtained during Phase IA testing (Hassan and McCabe, 1997). The very low value for the lag column indicates that this resin bed was not functioning properly.

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^(a) Many solution volumes given as bed volumes are based on the 15.0-mL BV for both columns.

Conditions: Temperature = 26°C, Feed Rate = 6 BV/hr, Bed Volume = 15.0 mL, SL-644 Batch Number = 644BZ,
 ^{137}Cs C_0 = 177.5 uCi/mL, Tank AW-101 Na Concentration = 4.6 M.

Figure 3.1. ^{137}Cs Breakthrough Curve (Run 1), Feed Displacement and Water Washing

The cause of the relatively poor performance of the lag column could have been due to channeling due to air in the resin bed or due to incomplete bed conditioning. Air is probably not the cause of the poor bed performance because the initial C/C_0 values for the lag column indicate the bed was not performing well prior to the leakage of air into the column system. Furthermore, air was not observed in the lag column resin bed during subsequent process steps. The cause may be incomplete bed conditioning even though the recommended or greater volumes of conditioning solutions were used. As discussed in section 3.4, the second resin bed did not change volume as much as the lead column during the shakedown testing and the resin bed preconditioning prior to introduction of the feed. This could have been due to partial consumption of the bed conditioning solutions by the resin in the lead column. The SL-644 resin has been shown to be sensitive to the bed conditioning prior to initiating the loading (McCabe et al. 1997).

The maximum decontamination factors measured were 11,600 for the lead column and 47,100 for the lag column. These values are based on ^{137}Cs concentrations of $1.54\text{E-}2 \mu\text{Ci/mL}$ for the lead column and $3.8\text{E-}3 \mu\text{Ci/mL}$ for the lag column. The overall DF was only 8.5 and is low due to the poor performance of the lag column.

The concentrations of sodium, potassium, Al and hydroxide in the feed displacement and DI water rinse solutions from run 1 are shown in Figure 3.2. The concentrations of the sodium, potassium, and Al are indicated on the left hand axis while the hydroxide concentration is shown on the right hand axis. Both Y axes are logarithmic scales in order to clearly show the roughly 100 fold decrease in concentrations. The samples were taken from the effluent line after the lag column, after the solutions had passed sequentially through both columns. Analytical results and calculations may be found in the appendix.

Due to the holdup volume in the system there was a delay between the introduction of the feed displacement and DI water wash solutions and the collection of these samples from the effluent line. In switching from the feed to the feed displacement step, valve 2 (Figure 2.1) was switched to the drain position and the initial part of the system was flushed. The valve was then switched to the flow position and sampling was initiated from the effluent line after the lag column. The holdup in the system between valve 2 and the effluent line was estimated to be about 40 mL so it is likely that the first 2 samples and much of the third sample consisted largely of AW-101 feed. This is supported by the component concentrations that are similar to the feed concentrations in the first two samples and by the visual observation of the sample color. The first two samples had a light yellow color that looked like the AW-101 feed and the third sample had a slight pink tinge to it. The source of the pink color is probably the SL-644 resin. In switching to the DI water rinse step, valve 1 was turned to the water supply container and the initial part of the system was not flushed. The total holdup in the system is estimated at 60 mL (4 BV) so the first four samples in the DI water rinse step, probably contained decreasing amounts of feed and caustic. The component concentrations indicate that washing was nearing completion at the fifth and sixth samples. The first four samples collected during the water rinse step had a distinct pink tinge to them while the last two were nearly clear. The required volume of feed displacement and DI water rinse solutions could be significantly reduced with a smaller holdup volume in the system and may not reflect the performance of a large-scale system.

Figure 3.2. Component Concentrations in Feed Displacement and DI Water Rinse Solutions

Since the lag column did not perform as expected, the overall decontamination factor was only about 8 and the level of ^{137}Cs in the effluent. Consequently a second column run was conducted with the partially treated AW-101 sample. This breakthrough curve is shown in Figure 3.3. A reduced sample set was collected only for the loading step. Two ^{137}Cs -removal limits are also shown in Figure 3.3. The higher C/C_0 value of 3.9% corresponds to the contractual limit for ^{137}Cs in the low-activity waste (LAW) glass, and the lower C/C_0 value of 0.39% corresponds to the basis of design target for ^{137}Cs in the LAW glass, which is 10 times less than the contractual limit. The C/C_0 values corresponding to these limits are determined using the sodium concentration of 4.59 M in the AW-101 sample, the ^{137}Cs feed concentration of 20.9 $\mu\text{Ci/mL}$, a 20 wt% total Na_2O loading in the glass, and a glass product density of 2.66 g/mL.

The $\text{Cs } \lambda$ value for the lead column in run 2 is estimated by extrapolation, to be 185. Insufficient breakthrough of ^{137}Cs prevents the determination of a $\text{Cs } \lambda$ for the lag column. The uniformly low level of ^{137}Cs in the effluent from the lag column indicates that the performance of the resin bed was much improved over the first run. The relatively good performance may be due to the additional cycle of the resin bed through the acid form during the elution of the lag column. The maximum decontamination factor measured was 99 for the lead column and at least 1130 for the lag column. These values are based on the ^{137}Cs concentrations determined by the analytical laboratory in the first sample from each column. While these DF's appear to be lower than those for run 1, it should be noted that roughly 88% of the ^{137}Cs was removed from the waste during the first run. A comparison of the ^{137}Cs concentrations and decontamination factors for both runs is shown in Table 3.2.

Figure 3.3. ^{137}Cs Breakthrough Curve (Run 2)

Table 3.2. Decontamination Factors for ^{137}Cs

	Processed Volume ^(a) , BV (mL)	^{137}Cs Concentration $\mu\text{Ci/mL}$	C/C_0 , %	DF
Run 1				
First sample from Lead Column	8.2 (123)	1.54E-02	8.6E-03	11,600
First sample from lag column	6 (90)	3.8E-03	2.1E-03	47,100
Effluent bottle-1	127 (1905)	10.9	6.2	16.3
Effluent bottle-2	37 (550)	56.1	31.6	
Composite effluent	164 (2455)	20.9	11.8	8.5
Run 2				
First sample from lead column	25 (375)	0.21	1.0	99
First sample from lag column	22 (330)	1.85E-02	0.089	1130
Composite effluent	152 (2280)	5.89E-02	0.28	355
Overall (both runs)	-	5.89E-02	3.3E-02	3000

(a) Processed volume is the volume collected in the effluent bottles adjusted for sampling and column system holdup.

3.3 Elution, Eluant Rinse and Regeneration

At the completion of the DI water wash, elution of the lead column was initiated by pumping 0.5 M nitric acid into the lead column at 1.2 BV/hr. Samples of the eluate and the eluant rinse were collected in approximately 1 BV increments while the regeneration solution was collected as a single composite. The ^{137}Cs content of these samples was determined with the portable GEA instrument and confirmed with analysis in the laboratory instrument. Due to holdup in the system, the first couple of bed volumes probably contained a substantial amount of the DI water rinse that preceded the elution.

The elution generally went well except for two instances when the top portion of the bed started to float due to air bubbles in the resin bed. The source of this air is thought to be in-leakage around the upper column plunger and not due to gas generation in the bed. The air was removed from the bed by tapping the column until the bubble migrated to the top of the resin and into the headspace above the bed and the elution was resumed. Eluate sample #3 was observed to contain solids. Although the solids were not analyzed they were light in color and very fluffy and are suspected of being precipitated NaAlO_2 . The source of this is likely residual waste that was not washed out of the column system.

The C/C_0 ^(a) values for ^{137}Cs are shown in Figure 3.4 for the elution, the eluant rinse and the regeneration steps. The Y axis is a logarithmic scale to clearly show the large range of C/C_0 values. The majority of the ^{137}Cs came off in just one sample (#4) which had a C/C_0 value of 97. The elution was continued until a C/C_0 of 0.01 was reached. The C/C_0 values for the eluant rinse are below the elution

^(a) The C_0 refers to the ^{137}Cs concentration in the AW-101 sample fed to the lead column. For elution, the C/C_0 value is an indication of the extent to which ^{137}Cs is concentrated relative to the feed. It is an indirect measure of the extent to which the resin is actually eluted.

cutoff of $C/C_0 = 0.01$. The ^{137}Cs concentration in the eluant rinse ranges from 0.12 - 5 uCi/mL. The last DI water rinse sample shows an unexpected spike in C/C_0 . The results of an analysis of the regeneration composite sample are shown in Table 3.3. For the ICP-AES results, the component determined is shown along with the assumed form in the regeneration solution.

Table 3.3. Composition of Regeneration Solution

	Concentration, $\mu\text{g/mL}$	BNFL MRQ, $\mu\text{g/mL}$	Concentration, M
Al	29	-	1.1E-03
B	23	-	2.2E-03
Ba	[0.1]	-	8.0E-07
Cr	[0.5]	-	9.8E-06
Fe	[0.8]	-	1.5E-05
Mn	[0.02]	-	4.2E-07
Mo	[0.37]	-	3.9E-06
Na^+	1800	75	7.8E-02
P	[0.65]	-	2.1E-05
Si	45.7	-	1.6E-03
Ti	[0.07]	-	1.6E-03
Zr	0.31	-	3.4E-06
OH^-	1,100	17	0.065
^{137}Cs	6.9E-2 uCi/mL	9	-
Total Cs	-	-	2.4E-08
Density, g/mL	-	-	1.004

Notes: MRQ is minimum reportable quantity. The overall error is estimated to be within $\pm 15\%$. Values in brackets are within 10 times the detection limit, and errors are likely to exceed $\pm 15\%$.

Samples of the eluate composites from runs 1 and 2 were submitted to the analytical laboratory for analysis with ICP-AES, GEA, total alpha, TOC and ICP-MS to determine the ^{99}Tc content. The results are shown in Tables 3.4, 3.5 and 3.6. Sodium was the dominant component detected with ICP-AES, although a number of other metals were also detected. Many of these same metals were also found in the eluate from the AN-107 testing (Kurath et al., 2000) and include Ca, Cr, Cu, Fe, Na Ni, Pb, U and Zn. Many of these same components were also detected during the analysis of the spent resin (Kurath and Wagner 2000) and include Ca, Cr, Cu, Fe, K, Na, Ni, Pb and Zn. A relatively high concentration of Al was found in the lead column eluate (Table 3.3) and this is attributed to the solids observed in sample # 3. As expected, the cesium isotopes are the dominant radionuclides. The only anion detected was nitrate, which is not surprising since the eluant was 0.5 M nitric acid. A small amount of total organic carbon was detected, but it is not known if this is from residual waste in the column system or from organic materials leaching from the resin.

Most of the BNFL-specified minimum reportable quantity (MRQ) levels were met with some exceptions. The large amount of ^{137}Cs prevented the detection limits for ^{241}Am , ^{154}Eu , and ^{155}Eu from meeting the MRQ levels. Relatively high ^{137}Cs activity increases the gamma background level in the detectors due to Compton scattering, thereby making it difficult to detect lower concentrations of other gamma emitters. The large amount of nitrate prevented the detection limit for Cl and F (Table 3.3 only) from meeting the MRQ levels. The large concentration of nitrate requires large sample dilutions and increases the method detection limit (MDL) for the other components. In any case, the anion concentrations other than nitrate are expected to be small. This is somewhat confirmed by the fact that no P was detected with the ICP-AES analysis. The total inorganic carbon (TIC) analysis was not completed because carbonate is known to evolve as CO_2 in acidic solutions.

Figure 3.4. Elution, Eluant Rinse and Regeneration of Lead Column

Table 3.4. Analysis of the Lead Column Eluate Composite from Run 1 and Minimum Reportable Quantities

ICP-AES Components		BNFL MRQ	Anions		BNFL MRQ
	µg/mL	µg/mL		µg/mL	µg/mL
Al	141	7.50E+01	TOC	[119]	1.50E+03
Ba	[0.42]	7.80E+01	TIC	NA	1.5E+02
Ca	[2.1]	1.50E+02	Br	<500	NMRQ
Cd	[0.31]	7.50E+00	Cl	<500	3.0E+00
Co	< 0.27	3.00E+01	F	<500	1.5E+02
Cr	3.48	1.50E+01	NO2	<1000	NMRQ
Cu	50.6	1.70E+01	NO3	33,000	3.0E+03
Fe	12.3	1.50E+02	PO4	[8.9]	2.5E+03
K	382	7.50E+01	SO4	<1000	2.3E+03
La	< 0.27	3.50E+01	oxalate	<1000	NMRQ
Mg	< 1.07	1.50E+02			
Mn	0.67	1.50E+02	Radionuclides		MRQ
Mo	< 0.32	9.00E+01		µCi/mL	µCi/mL
Na	2230	7.50E+01	Cs-134	3.36E-01	NMRQ
Ni	5.89	3.00E+01	Cs-137	1.61E+03	9.00E+00
Pb	15.6	3.00E+02	Sr-90	<5E-2	1.50E-01
Si	51.4	1.70E+02	Tc-99	9.05E-04 (5.3E-2 ug/mL)	1.50E-03
Sn	< 10.7	1.50E+03	Am-241	<1E+0	7.20E-04
Ti	< 0.05	1.70E+01	Eu-154	<6E-2	2.00E-03
U	[48]	6.00E+02	Eu-155	<1E+0	9.00E-02
Zn	12	1.65E+01	Total alpha	<4E-2	2.30E-01
B	72.6	NMRQ	Total volume = 195 mL		
P	[2.9]	NMRQ	Total mass = 198 g		
			Density = 1.015 g/mL		

Notes: MRQ is minimum reportable quantity. NMRQ is no minimum reportable quantity. NA = not analyzed. < indicates detection limit; component not detected. Overall error is estimated to be within +/-15%. Values in brackets are within 10-times the detection limit and errors are likely to exceed +/-15%.

Table 3.5. Analysis of the Lag Column Eluate Composite from Run 1 and Minimum Reportable Quantities

ICP-AES Components		BNFL MRQ	Anions		BNFL MRQ
	µg/mL	µg/mL		µg/mL	µg/mL
Al	[1.3]	7.50E+01	TOC	<18	1.50E+03
Ba	[0.23]	7.80E+01	TIC	NA	1.5E+02
Ca	[3.3]	1.50E+02	Br	<100	NMRQ
Cd	[0.34]	7.50E+00	Cl	<100	3.0E+00
Co	< 0.52	3.00E+01	F	<100	1.5E+02
Cr	6.54	1.50E+01	NO2	<200	NMRQ
Cu	12.7	1.70E+01	NO3	31,500	3.0E+03
Fe	12.8	1.50E+02	PO4	<3	2.5E+03
K	[190]	7.50E+01	SO4	<200	2.3E+03
La	< 0.52	3.50E+01	oxalate	<200	NMRQ
Mg	< 1.1	1.50E+02			
Mn	< 0.52	1.50E+02	Radionuclides		MRQ
Mo	< 0.52	9.00E+01		µCi/mL	µCi/mL
Na	698	7.50E+01	Cs-134	4.05E-02	NMRQ
Ni	[2.8]	3.00E+01	Cs-137	2.42E+02	9.00E+00
Pb	12.8	3.00E+02	Sr-90	2.44E-01	1.50E-01
Si	[6.5]	1.70E+02	Tc-99	4.66E-04 (2.74E-02 ug/mL)	1.50E-03
Sn	< 15.5	1.50E+03	Am-241	<2E-01	7.20E-04
Ti	< 0.26	1.70E+01	Eu-154	<7E-03	2.00E-03
U	[48]	6.00E+02	Eu-155	<2E-01	9.00E-02
Zn	6.2	1.65E+01	Total alpha	<2E-5	2.30E-01
B	7.8	NMRQ	Total volume = 182 mL		
P	< 1.0	NMRQ	Total mass = 185 g		
			Density = 1.015 g/mL		

Notes: MRQ is minimum reportable quantity. NMRQ is no minimum reportable quantity. NA = not analyzed. < indicates detection limit; component not detected. Overall error is estimated to be within +/-15%. Values in brackets are within 10-times the detection limit and errors are likely to exceed +/-15%.

Table 3.6. Analysis of the Eluate Composite for Both Columns, Run 2 and Minimum Reportable Quantities

ICP-AES Components		BNFL MRQ	Anions		BNFL MRQ
	µg/mL	µg/mL		µg/mL	µg/mL
Al	[6.1]	7.50E+01	TOC	< 18	1.50E+03
Ba	< 0.1	7.80E+01	TIC	NA	1.5E+02
Ca	[2.9]	1.50E+02	Br	< 100	NMRQ
Cd	[0.23]	7.50E+00	Cl	< 100	3.0E+00
Co	< 0.52	3.00E+01	F	1500	1.5E+02
Cr	3.25	1.50E+01	NO2	< 200	NMRQ
Cu	7.13	1.70E+01	NO3	26,500	3.0E+03
Fe	8.1	1.50E+02	PO4	< 3	2.5E+03
K	[210]	7.50E+01	SO4	< 200	2.3E+03
La	< 0.52	3.50E+01	oxalate	< 200	NMRQ
Mg	< 1.04	1.50E+02			
Mn	< 0.52	1.50E+02	Radionuclides		MRQ
Mo	< 0.52	9.00E+01		µCi/mL	µCi/mL
Na	1150	7.50E+01	Cs-134	2.43E-02	NMRQ
Ni	5.69	3.00E+01	Cs-137	1.47E+02	9.00E+00
Pb	[9.9]	3.00E+02	Sr-90	8.56E-02	1.50E-01
Si	[6.3]	1.70E+02	Tc-99	2.59E-04 (1.52E-02 ug/mL)	1.50E-03
Sn	< 15.5	1.50E+03	Am-241	<9E-02	7.20E-04
Ti	< 0.26	1.70E+01	Eu-154	<6E-3	2.00E-03
U	< 20.6	6.00E+02	Eu-155	<9E-02	9.00E-02
Zn	< 0.52	1.65E+01	Total alpha	1.64E-05	2.30E-01
B	7.42	NMRQ	Total volume = 305 mL		
P	< 1.03	NMRQ	Total mass = 310 g		
			Density = 1.015 g/mL		

Notes: MRQ is minimum reportable quantity. NMRQ is no minimum reportable quantity. NA = not analyzed. < indicates detection limit; component not detected. Overall error is estimated to be within +/-15%. Values in brackets are within 10-times the detection limit and errors are likely to exceed +/-15%.

3.4 Mass Balance for ¹³⁷Cs

A mass balance for ¹³⁷Cs was completed for runs 1 and 2 in order to compare the ¹³⁷Cs recovered in various process streams to the ¹³⁷Cs present in the feed sample (Table 3.7 and 3.8). As expected, the majority of the ¹³⁷Cs was found in the eluate streams. Most of the ¹³⁷Cs present in the feed sample was accounted for in the samples and process streams, indicating that the integrity of the experiment was fairly good.

Table 3.7. Mass Balance for ^{137}Cs , Run 1

	^{137}Cs , μCi	% of ^{137}Cs in Feed Sample
Feed sample	435,900	100
Effluent-1	20,200	4.63
Effluent-2	29,100	6.67
Load samples	3,100	0.71
Feed displacement	3,500	0.80
DI water rinse	2,500	0.57
Column #1 eluate	313,800	71.98
Column #1 DI water rinse	120	0.03
Column #1 regeneration	6	0.00
Column #2 eluate	44,000	10.10
Total ^{137}Cs recovery	416,300	95.5

Table 3.8. Mass Balance for ^{137}Cs , Run 2

	^{137}Cs , μCi	% of ^{137}Cs in Feed Sample
Feed sample	47,700	100
Effluent	132	0.28
Load samples	47	0.10
Eluate-both columns	45,900	96.26
Total ^{137}Cs recovery	46,100	96.6

3.5 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 (Hassan et al., 1999). The variation in bed volume as a function of the process steps for both columns is shown in Figures 3.5 and 3.6 as a ratio of bed volume to the baseline value of 15 mL. In this case the baseline value was chosen as the volume of the beds shortly after the loading step was initiated. This is the same volume as the bed volume in 0.25 M NaOH when the column system was loaded into the hot cell. The bed volume ratios can be converted to an as-received resin bed volume basis by multiplying the values given in Figures 3.5 and 3.6 by 2.4. This value is the ratio of the 15 mL bed volume used in the figures to the as-received resin volume of 6.2 mL.

Together, the two figures show the bed volume changes of each bed as they are cycled 4 times through the acid and sodium form. The first three cycles are shown in Figure 3.4. The first cycle was performed outside of the hot cell during shakedown testing. A second cycle was performed in order to condition the resin bed just prior to initiating run 1. The third cycle was run 1 with the AW-101 waste sample and the fourth run was the second run with the AW-101 sample. Each process step is denoted with a letter defined as follows: W (DI water), E (elution with 0.5 M nitric acid), ER (elution rinse with DI water), R (regeneration with 0.25 M NaOH), F (feed), and FD (feed displacement with 0.1 M NaOH).

Figure 3.5. Comparison of Bed Volumes of the Lead and Lag Columns for the First Run (SL-644 Batch # 644BZ; Baseline Bed Volume = 15 mL)

Figure 3.6. Comparison of Bed Volumes of the lead and Lag Columns for the Second Run (SL-644 Batch # 644BZ; Baseline Bed Volume = 15 mL)

The following observations are qualitatively similar to the resin volume changes reported in Hassan et al., 1999.

- Both columns reached minimum volumes in the hydrogen form (after elution) and maximum volumes in the sodium form after contact with dilute caustic and water.
- The amplitude of the swelling and shrinking increased for both resin beds as the resin was successively cycled through the hydrogen and sodium forms,

A close comparison of the volume changes in the two beds indicates the following:

- By the end of the 4th cycle, the resin bed in the lead column appears to be about 10% larger than the resin bed in the lag column.
- Both columns had nearly the same volume during the loading step when in contact with the waste sample.
- During the bed conditioning steps prior to the initiation of run 1, the volume change of the lag column did not occur as quickly as the lead column and the lag column may not have been as fully conditioned as the lead column. This could have been due to partial consumption of the bed conditioning chemicals in the lead column. Since adequate conditioning of the resin beds has been found to be important to the SL-644 performance this could explain the relatively poor loading performance of the lag column. In future testing, the bed conditioning solutions will be pumped through the beds until the volume changes of both columns are complete and the columns may be conditioned separately instead of together in series.

3.6 Batch Contact Results

The distribution coefficients (Kds) are shown in Figure 3.7 for the SL-644 resin in contact with the AW-101 column feed. The Kd values were calculated using Equation 1 and are based on the ^{134}Cs and ^{137}Cs concentrations as measured by GEA. The Na/Cs ratio is calculated from the individual sodium and cesium concentrations. The sodium concentrations are based on an analysis of the initial feed as prepared for the column run and is assumed to be constant for the batch contacts. Since the quantity of H^+ added with the resins is small relative to the moles of Na^+ and OH^- (phase ratio of 100 mL of solution: gram of exchanger), this is a reasonable assumption. In these experiments, the waste solutions were estimated to have 10.9 meq of OH^- and 23 meq of Na^+ , while the resin had 0.005 meq of H^+ . The total cesium was based on the ^{137}Cs concentrations, and the ratio of ^{137}Cs :total cesium was determined for the unspiked and spiked solutions. There appears to be considerable scatter in the data since the results are expected to lie on a reasonably smooth curve.

The Kd at the AW-101 feed conditions can be estimated to range from 280-340 mL/g from the point at which the Kd plot crosses the Na/Cs mole ratio (74,700) for the AW-101 sample. The column distribution ratio can be determined from the Kd and the appropriate bed density using the following relationship

$$C_s \lambda = Kd * \rho_b \quad (2)$$

where $C_s \lambda$ is the column distribution ratio, and ρ_b is the dry bed density.

In this case, the dry-bed density is 0.287 g/mL, which is determined from the dry (@ 95°C) mass of exchanger (4.3 g) divided by the expanded BV in the columns of 15 mL. The $C_s \lambda$ is then estimated to range from 80-97. This value should correspond fairly closely to the $C_s \lambda$ of 143 determined from the breakthrough curve for the lead column (see Figure 3.1). The reason for the considerable difference is not known.

Figure 3.7. ¹³⁷Cs Distribution Coefficients (Kd) for AW-101 Feed

4.0 CONCLUSIONS AND RECOMENDATIONS

- Small-column testing with SL-644 indicates that sufficient decontamination of ^{137}Cs can be obtained with waste from Tank 241-AW-101 (Envelope A) to allow the LAW glass product to meet the basis of design limit for ^{137}Cs removal, which is a factor of 10 less than the contractual limit. An overall decontamination factor of 3000 was obtained providing a cesium-decontaminated effluent with a ^{137}Cs concentration of $5.89\text{E-}02 \text{ Ci/m}^3$. This is 7.3% of the contract limit.
- While two separate runs were required to achieve the necessary decontamination, this is not expected to be normally required. During the first run the lag column did not function as expected and an unexpectedly large amount of cesium passed through the system. During the second run both columns functioned well and good decontamination factors were obtained.
- The poor performance of the lag column is thought to have been caused by inadequate bed conditioning prior to the introduction of the waste into the columns. This could have caused channeling or slow uptake of cesium. Channeling could also have resulted from air in the bed due to air leakage into the system. Although air did get into the system it was never observed in the bed in the lag column.
- Batch contacts were conducted to obtain equilibrium data in the form of batch distribution coefficients (Kds). These equilibrium data predict a range for the Cs λ of 80-97.
- The feed displacement (0.1 M NaOH) volume of 3 BV followed by 6 BV of DI water rinse appeared to leave some residual waste in the column system since a small amount of solids were observed in the third eluate sample. An increase in the volume of feed displacement solutions is recommended.
- The cesium loaded columns are efficiently eluted with 0.5 M nitric acid. The majority of the ^{137}Cs elutes from the column in 1 bed volume of eluate although a total of 12 bed volumes of eluant was necessary to reach the elution end point of a $C/C_0 = 0.01$.
- The SL-644 was adequately regenerated with 6 bed volumes of 0.25 M NaOH. The average ^{137}Cs concentration in the regeneration solution was $6.9\text{E-}2 \text{ }\mu\text{Ci/mL}$ with a C/C_0 of $3.89\text{E-}04$.
- Fouling of the resin bed or exchanger was not observed.

5.0 REFERENCES

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APPENDIX A

Appendix A: Sample Identification for AW-101 Cs IX Runs

Sample ID	Description
A1-Cs-0	Initial feed sample for run 1 – AW-101
A1-Cs-ICP	Initial feed sample for run 1 – AW-101
A1-Cs-L1 through L17	Loading samples from lead column, run 1 – AW-101
A1-Cs-P1 through P9	Loading samples from lag column, run 1 – AW-101
Eff – 1	Effluent composite container 1, run 1 – AW-101
Eff – 2	Effluent composite container 2, run 1 – AW-101
A1-Cs-PW1 through PW3	Feed displacement samples, run 1 – AW-101
A1-Cs-PR1 through PR6	DI water rinse samples, run 1 – AW-101
A1-Cs-E1-1 through E1-12	Eluate samples from run 1, lead column – AW-101
A1-Cs-E1-R1 through R6	Eluant rinse samples from run 1, lead column – AW-101
Regeneration solution	Regeneration composite, run 1, lead column – AW-101
A1R-Cs-0	Feed sample for run 2 – AW-101
A1R-Cs-L1 through L17	Loading samples from lead column, run 2 – AW-101
A1R-Cs-P1 through P9	Loading samples from lag column, run 2 – AW-101
A1-Cs-E1-composite	Eluate composite, lead column, run 1 – AW-101
A1-Cs-E2-Comp and A1-Cs-E2-CO	Eluate composite, lag column, run 1 – AW-101
A1R-Cs-E-comp	Eluate composite, both columns, run 2 – AW-101
A1-Tc-LC	AW-101 Tc IX Feed which is the same as the Cs IX effluent composite from run 2.
A1-Tc-0	Tc IX Feed which is the same as the Cs IX effluent composite from run 2

Notes: 1) The letter A, B, C or D after the sample ID indicates that the original sample was diluted and/or transferred to a new, uncontaminated vial for loading out from the hot cell.

Sample Identification Continued

The following sample results are included in the appendix since they were reported with some of the AW-101 Cs IX samples. Samples from other work were batched to reduce cost and duration (schedule). These results are discussed in other reports.

C1-Cs-0AR	Initial feed sample – AN-107 Cs IX
C1-Cs-ICP	Additional feed sample – AN-107 Cs IX
C1-Cs-L1 through L12	Loading samples from lead column – AN-107 Cs IX
C1-Cs-P1 through P6	Loading samples from lag column – AN-107 Cs IX
Effluent – 1D	Effluent composite container 1 – AN-107 Cs IX
Effluent – 2D	Effluent composite container 2 – AN-107 Cs IX
AN-107-TcIX	Cs IX effluent which is also the Tc IX feed
C1-Cs-FD1 through FD7	Feed displacement samples - AN-107 Cs IX
C1-Cs-PR1 through PR6	DI water rinse samples - AN-107 Cs IX
C1-Cs-E1-1 through E1-13	Eluate samples from lead column - AN-107 Cs IX
C1-Cs-E1-R1 through R6	Eluant rinse samples from lead column - AN-107 Cs IX
Regeneration	Regeneration composite for lead column - AN-107 Cs IX
C1-Cs-E2-1 through E2-13	Eluate samples from lag column - AN-107 Cs IX
C1-CA	AN-107 feed control for batch contacts
C1-S1A	AN-107 spike 1 feed control for batch contacts
C1-S2A	AN-107 spike 2 feed control for batch contacts
C1-44A, C1-44-D-A	Batch contact with unspiked AN-107 sample
C1-44-S1-A, C1-44-S1-D-A	Batch contact with spike 1 AN-107 sample
C1-44-S2-A, C1-44-S2-D-A	Batch contact with spike 2 AN-107 sample
C1-Cs-E1-composite	Eluate composite from column 1, AN-107 run
C1-Cs-E2-composite	Eluate composite from column 2, AN-107 run
N7-Tc-0	Tc IX feed which is also Cs IX effluent composite
AN-107-TcIX	AN-107 Tc IX feed sample
A1-Tc-EC1	Eluate composite from Tc IX/AW-101
N7-Tc-Elu-Comp	Tc IX eluant composite from elution of lead column loaded with AN-107 sample
N7-Tc-PW1 – PW7	Feed displacement samples from Tc IX, AN-107 run
N7-Tc-PR1 –PR2	Rinse samples from Tc IX, AN-107 run

AW-101 Column Run Analytical Results

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Project: 29953
Client: D. E. Kurath

ACL Number(s): 99-2018, 99-2046 through 99-2054, & 99-2072

Client ID: "A1-Cs-ICP", "A1-Cs-PW1-A" through "A1-Cs-PR6-A", & "regeneration soln"

ASR Number: 5421

Total Samples: 11

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

Analyst: DR Sanders

Analysis Date (Filename): 07-08-99 (A0533)

See Chemical Measurement Center 98620: ICP-325-405-1 File for Calibration and
Maintenance Records.

M&TE Number: ICPAES instrument -- WB73520
Mettler AT400 Balance -- Ser.No. 360-06-01-029

Jerry Wagner 8-2-99
Reviewed by
MW Thur 8-7-99
Concur

8/2/99

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Eleven radioactive liquid samples, A1-Cs-ICP through "regeneration soln" (ACL# 99-2018 through 99-2072), were analyzed by ICPAES after preparation by the Sample Receiving and Preparation Laboratory (SRPL). Samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. Approximately 1ml to 5ml of sample (weighed) was processed and diluted to a final volume of 10ml or 20ml. Several of the liquid samples contained some visible solids before processing. Sample A1-Cs-PW1-A, A1-Cs-PW2-A, and A1-Cs-PR1-A (ACL# 99-2046, 99-2047 and 99-2049 respectively) required an additional 0.5ml of concentrated nitric acid to dissolve the solids. An additional 1ml of concentrated nitric acid was added to sample A1-Cs-PW3-A (ACL# 99-2048) in an attempt to dissolve the solids but ultimately it had to be filtered using 0.45 um filter. After digestion and filtering the one sample, all solutions remained clear. Additional dilution, up to 25-fold, was performed during ICPAES analysis because of high aluminum and sodium concentration levels.

All measurement results reported have been corrected for preparation and analytical dilution. Analytes of interest include Al, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Pb, Si, Sn, Ti, U, and Zn. All results reported are in $\mu\text{g/g}$ including liquid samples as requested by the client. Volumes and weights have been recorded on bench sheets (included with raw data, etc.).

Note: changes in concentration from the preliminary results have been made for the following samples: A1-Cs-PW1-A, A1-Cs-PW2-A, A1-Cs-PW3-A, and A1-Cs-PR1-A (ACL# 99-2046 through 99-2049 respectively).

All quality control checks met tolerance requirements for analytes of interest except as noted below. Following is a list of quality control check measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Aqueous samples)

All results were within tolerance limit of $\leq 10\%$ after correcting for dilution except K (approximately 11%) in sample A1-Cs-ICP (ACL# 99-2018) and Ni (approximately 87%) in sample A1-Cs-PR3-A (ACL# 99-2051) all other analytes above EQL were less than 6% difference after dilution correction. The discrepancy for Ni may have resulted from carry-over in a pipet used to dilute a previous sample or standard having high concentration of nickel. Five other diluted samples prepared similarly were all within the $\leq 10\%$ tolerance limit.

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Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report

Duplicate RPD (Relative Percent Difference):

(Aqueous samples) All analytes of interest were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD).

Post-Spiked Samples (Group A):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

(Aqueous samples) All analytes of interest in the blank spike were recovered within tolerance limit of 80% to 120%.

Matrix Spiked Sample:

(Aqueous samples) All analytes of interest in the matrix-spike were recovered within tolerance limit of 75% to 125%.

Quality Control Check Standards:

Concentration of all analytes of interest within tolerance limit of $\pm 10\%$ accuracy in the standards: QC_MCVA and QC_MCVB. Calibration Blank (ICP98.0) concentration was less than two times IDL. Silicon concentration in the first of two measurements was high by 13%. The second measurement at the end of the ICPAES run was within the tolerance limit of $\pm 10\%$ accuracy.

High Calibration Standard Check:

Verification of the high-end calibration concentration for all analytes of interest was within tolerance of $\pm 5\%$ accuracy.

Process Blank:

(Aqueous samples) All analytes of interest were within tolerance limit of $\leq$ EQL or $< 5\%$ of sample concentration except Si which was equivalent to about 18% or less of that measured in the samples. Boron was also found in the blank which suggests that both silicon and boron was likely from the glass digestion vessel used to prepare the samples.

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**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Laboratory Control Standard (LCS):

(Aqueous samples)   No LCS was prepared for PNL-ALO-128 acid digested samples.

Analytes other than those requested by the client are for information only. Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%. See attached ICPAES data results.

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 $\pm 15\%$ 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 $\mu\text{g/mL}$ (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.

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date=	1.0 BL-2018 Blank (D.I. Water) 7/8/99 (ug/mL)	40.3 99-2018 @5 A1-Cs-ICP 7/8/99 ug/g	17.4 99-2046 @5 A1-Cs-PW1-A 7/8/99 ug/g	17.0 99-2047 @5 A1-Cs-PW2-A 7/8/99 ug/g	18.7 99-2048 @5 A1-Cs-PW3-A 7/8/99 ug/g
0.015	Ag	--	--	--	--	--
0.060	Al	[0.28]	9,030	8,460	8,680	5,480
0.080	As	--	[15]	14.1	13.8	[8.8]
0.050	B	1.97	59.4	37.9	40.2	35.5
0.010	Ba	[0.020]	--	--	--	--
0.010	Be	--	[0.76]	[0.71]	[0.73]	[0.43]
0.100	Bi	--	--	--	--	--
0.100	Ca	--	[4.6]	[6.5]	[7.8]	[4.3]
0.015	Cd	--	[1.2]	[1.2]	[1.3]	[1.3]
0.100	Ce	--	--	--	--	--
0.025	Co	--	--	--	--	--
0.020	Cr	--	35.4	31.2	30.3	16.6
0.015	Cu	--	[4.0]	3.39	3.82	2.97
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[0.034]	11.6	5.75	5.85	[2.8]
2.000	K	--	12,700	11,700	12,200	9,370
0.025	La	--	--	--	--	--
0.005	Li	--	--	[0.13]	[0.089]	--
0.100	Mg	--	--	--	--	--
0.005	Mn	--	[1.3]	--	--	--
0.030	Mo	--	20.1	18.6	19.0	11.5
0.100	Na	2.36	85,900	73,700	78,700	58,800
0.100	Nd	--	--	--	--	--
0.030	Ni	--	[3.6]	[3.0]	[3.1]	[3.5]
0.100	P	--	181	173	179	134
0.060	Pb	--	[22]	20.5	20.6	14.9
0.300	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
0.075	Ru	--	--	[2.1]	[2.0]	--
0.050	Sb	--	[2.6]	[2.1]	[2.0]	[1.4]
0.050	Se	--	[4.7]	[4.2]	[4.1]	[2.7]
0.100	Si	5.19	180	79.5	86.2	70.7
1.000	Sn	--	[43]	[38]	[38]	[23]
0.005	Sr	--	--	--	--	--
0.500	Te	--	--	--	--	--
0.800	Th	--	--	--	--	--
0.005	Ti	[0.015]	--	--	--	--
0.250	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.015	V	--	--	--	--	--
0.500	W	--	[39]	[36]	[37]	[17]
0.010	Y	--	--	--	--	--
0.020	Zn	--	[4.2]	4.29	4.46	[2.3]
0.025	Zr	--	[1.7]	[3.1]	[3.0]	[0.91]

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).

et. Limit	Multiplie=	18.9	3.8	3.9	3.9	4.0
ug/mL	ALO#	99-2049 @5	99-2050	99-2051	99-2052	99-2053
	Client ID	A1-Cs-PR1-A	A1-Cs-PR2-A	A1-Cs-PR3-A	A1-Cs-PR4-A	A1-Cs-PR5-A
	Run Date	7/8/99	7/8/99	7/8/99	7/8/99	7/8/99
	(Analyte)	ug/g	ug/g	ug/g	ug/g	ug/g
0.015	Ag	—	—	—	—	—
0.060	Al	2,650	1,170	389	121	84.7
0.080	As	[4.3]	[1.8]	[0.72]	—	—
0.050	B	20.5	14.9	15.6	12.9	16.4
0.010	Ba	—	[0.086]	[0.092]	[0.094]	[0.091]
0.010	Be	—	[0.047]	—	—	—
0.100	Bi	—	—	—	—	—
0.100	Ca	—	[0.94]	[0.41]	—	—
0.015	Cd	[0.81]	[0.39]	[0.23]	[0.068]	—
0.100	Ce	—	—	—	—	—
0.025	Co	—	—	—	—	—
0.020	Cr	6.20	2.49	[0.71]	[0.31]	[0.25]
0.015	Cu	[1.8]	1.03	0.626	[0.46]	[0.27]
0.050	Dy	—	—	—	—	—
0.100	Eu	—	—	—	—	—
0.025	Fe	[0.90]	[0.51]	[0.55]	[0.24]	[0.17]
2.000	K	5,340	2,300	976	543	345
0.025	La	—	—	—	—	—
0.005	Li	—	—	—	—	—
0.100	Mg	—	—	—	—	—
0.005	Mn	—	—	—	—	—
0.030	Mo	[5.5]	2.46	[0.85]	[0.26]	[0.19]
0.100	Na	29,400	13,200	5,900	2,980	1,920
0.100	Nd	—	—	—	—	—
0.030	Ni	[3.1]	1.82	1.46	1.41	1.21
0.100	P	158	47.4	12.4	[2.8]	[1.8]
0.060	Pb	[6.4]	2.36	[0.95]	[0.41]	—
0.300	Pd	—	—	—	—	—
0.300	Rh	—	—	—	—	—
0.075	Ru	—	—	—	—	—
0.050	Sb	—	[0.21]	—	—	—
0.050	Se	[1.0]	[0.46]	[0.21]	—	—
0.100	Si	36.4	32.6	32.4	33.1	32.6
1.000	Sn	—	[3.9]	[4.4]	—	—
0.005	Sr	—	—	—	—	—
0.500	Te	—	—	—	—	—
0.800	Th	—	—	—	—	—
0.005	Ti	—	[0.053]	[0.063]	[0.062]	[0.053]
0.250	Tl	—	—	—	—	—
2.000	U	—	—	—	—	—
0.015	V	—	—	—	—	—
0.500	W	[11]	[4.8]	—	—	—
0.010	Y	—	—	—	—	—
0.020	Zn	[0.65]	[0.24]	[0.12]	[0.091]	[0.081]
0.025	Zr	—	[0.16]	[0.13]	—	—

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).

Multipler=	4.0	4.0	4.0		
ALO#=	99-2054	99-2054-DUP	99-2072		
Client ID=	A1-Cs-PR6-A	A1-Cs-PR6-A	Regeneration soln		
det. Limit (ug/mL)	Run Date= 7/8/99	7/8/99	7/8/99		
(Analyte)	ug/g	ug/g	ug/g		
0.015	Ag	-	-	-	-
0.060	Al	104	108	29.0	-
0.080	As	-	-	-	-
0.050	B	10.4	11.8	23.1	-
0.010	Ba	[0.081]	[0.087]	[0.11]	-
0.010	Be	-	-	-	-
0.100	Bi	-	-	-	-
0.100	Ca	-	-	-	-
0.015	Cd	-	-	-	-
0.100	Ce	-	-	-	-
0.025	Co	-	-	-	-
0.020	Cr	[0.23]	[0.23]	[0.51]	-
0.015	Cu	[0.14]	[0.15]	-	-
0.050	Dy	-	-	-	-
0.100	Eu	-	-	-	-
0.025	Fe	[0.11]	[0.12]	[0.84]	-
2.000	K	231	238	-	-
0.025	La	-	-	-	-
0.005	Li	-	-	-	-
0.100	Mg	-	-	-	-
0.005	Mn	-	-	[0.023]	-
0.030	Mo	[0.19]	[0.20]	[0.37]	-
0.100	Na	1,270	1,310	1,790	-
0.100	Nd	-	-	-	-
0.030	Ni	[0.59]	[0.61]	-	-
0.100	P	[1.9]	[2.0]	[0.65]	-
0.060	Pb	-	-	-	-
0.300	Pd	-	-	-	-
0.300	Rh	-	-	-	-
0.075	Ru	-	-	-	-
0.050	Sb	-	-	-	-
0.050	Se	-	-	-	-
0.100	Si	28.5	31.2	45.4	-
1.000	Sn	-	-	-	-
0.005	Sr	-	-	-	-
0.500	Te	-	-	-	-
0.800	Th	-	-	-	-
0.005	Ti	[0.048]	[0.056]	[0.065]	-
0.250	Tl	-	-	-	-
2.000	U	-	-	-	-
0.015	V	-	-	-	-
0.500	W	-	-	-	-
0.010	Y	-	-	-	-
0.020	Zn	-	-	-	-
0.025	Zr	-	-	[0.31]	-

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).

proj 29953
Task 28
T 2-8.3.3

PNL-ALO-128

Nitric and Hydrochloric Acid Extraction of Liquids Using a Dry-Block Heater

Client name: <u>Green Kaurath</u>	Work package number: <u>1048409</u>
Work Auth. Doc (WAD): <u>A52 5421</u>	Project number:
Tank/Coro/Project:	PNL QA plan:
Special Instructions:	PNL Impact level:
	Prop. lab (SAL/SRPL/other):
	Preparation batch number:

ACL Sample ID	ACL order number or Client sample ID	Sample Mass (g)	Sample Volume (ml)	Spike added Volume (ml)	Spike added Weight (g)	Final solution Volume (ml)	Process Factor (1)	
1 <u>99-2018</u>	<u>A1-C₅-PCP</u>	<u>1.2400</u>	<u>1 ml.</u>	<u>1.24</u>		<u>10 ml</u>		
2 <u>2046</u>	<u>A1-C₅-PW1-A</u>	<u>3.0178</u>	<u>2.5 ml.</u>	<u>1.207 g/ml</u>		<u>10.5</u>		NOTE: Final dilution volume with extra HNO ₃ additions
3 <u>2047</u>	<u>A1-C₅-PW2-A</u>	<u>3.0874</u>	<u>↓</u>	<u>1.235</u>		<u>10.5</u>		
4 <u>2048</u>	<u>A1-C₅-PW3-A</u>	<u>2.9414</u>	<u>↓</u>	<u>1.179</u>		<u>11.0</u>		
5 <u>2049</u>	<u>A1-C₅-PR1-A</u>	<u>5.4301</u>	<u>5 ml</u>	<u>1.086</u>		<u>20 ml</u>		
6 <u>2050</u>	<u>A1-C₅-PR2-A</u>	<u>5.2053</u>	<u>↓</u>	<u>1.041</u>		<u>↓</u>		
7 <u>2051</u>	<u>A1-C₅-PR3-A</u>	<u>5.1216</u>	<u>↓</u>	<u>1.024</u>		<u>↓</u>		
8 <u>2052</u>	<u>A1-C₅-PR4-A</u>	<u>5.0647</u>	<u>↓</u>	<u>1.013</u>		<u>↓</u>		
9 <u>2053</u>	<u>A1-C₅-PR5-A</u>	<u>5.0494</u>	<u>↓</u>	<u>1.010</u>		<u>↓</u>		
10 <u>2054</u>	<u>A1-C₅-PR6-A</u>	<u>5.0404</u>	<u>↓</u>	<u>1.008</u>		<u>↓</u>		
11 <u>2072</u>	<u>Regen Soln</u>	<u>2.5114</u>	<u>2.5 ml.</u>	<u>1.005</u>		<u>10 ml</u>		
12 <u>MS-2054</u>		<u>5.0595</u>	<u>5 ml.</u>	<u>0.4 ml Part A</u> <u>0.4 ml Part B</u>		<u>20 ml</u>		
13 <u>Bup-2054</u>		<u>5.0452</u>	<u>↓</u>	<u>0.4 ml Part A</u> <u>0.4 ml Part B</u>		<u>↓</u>		
14 <u>BS-2018</u>			<u>↓</u>	<u>0.4 ml Part A</u> <u>0.4 ml Part B</u>		<u>↓</u>		

Analyst's sample preparation comments:

TCLP Spiking Solution Part A - #990305 JTCIP 0.4 ml
" " " Part B - #990629 JTCIP 0.4 ml
Pipette # 288618
(# 288618 @ 0.4 ml) = 0.0426 g (# 130754 @ 2.5 ml) = 2.5152 g
@ 1 ml = 1.0014 g, 1.0023 g, 0.9985 g, 0.0415 g, 0.0424 g, 0.0429 g, 2.5013 g, 2.5016 g, 2.4957 g
1.0018 g, 1.0049 g, 0.0443 g, 2.5011 g

Spike source:

PNL spike ID number:

Anal. balance M&TE: 360-06-01-037

Sample filtered (yes/no): 99-2048 on

0.45 g/ml

(1) Process factor = Final volume (ml) / Sample volume (ml)

X See Back Page #2

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: Lori P. Darnell 6-30-99 Reviewer/Date: _____

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

Project 2111
Task 288
42.8.3.3

ASR # 5421
WP# W48409

File: L:\radchem\hydroxide\asr5421

Analysis Date: 7/1/99

Print Date: 7/8/99

Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator

Equip # WB76843

Lab Loc. 525

Analyst: *[Signature]* 7/8/99

Reviewer: *[Signature]* 7/8/99

Titrant	Molarity
HCl	0.2034

Std. & Spike	Molarity
NaOH	0.1018

RPG #	Sample ID		Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Initial pH reading	OH		Found millimoles base	Molarity base	millimole RPD
							1st Equivalence Point Titrant Vol. (mL)	pH			
99-2046	A1-Cs-PW1-A		0.300	0.3692	1.231	12.000	3.042	10.153	0.619	2.06	
99-2046	A1-Cs-PW1-A	Rep.-1	0.200	0.252	1.260	11.924	2.011	10.179	0.409	2.05	0.84%
99-2046	A1-Cs-PW1-A	Rep.-2	0.200	0.2457	1.229	11.882	2.034	10.196	0.414	2.07	1.14%
99-2046	A1-Cs-PW1-A	Rep.-3	0.100	0.1218	1.218	11.726	1.015	10.004	0.206	2.06	0.20%
99-2047	A1-Cs-PW2-A		0.300	0.3691	1.230	11.907	2.906	10.323	0.591	1.97	
99-2047	A1-Cs-PW2-A	Replicate	0.200	0.2466	1.233	11.925	2.019	10.304	0.411	2.05	4.13%
99-2048	A1-Cs-PW3-A		0.200	0.2311	1.156	11.895	1.610	10.000	0.327	1.64	
99-2048	A1-Cs-PW3-A	Replicate	0.200	0.2345	1.173	11.808	1.634	9.972	0.332	1.66	1.48%
99-2049	A1-Cs-PR1-A		0.200	0.2156	1.078	11.587	0.737	9.639	0.150	0.75	
99-2049	A1-Cs-PR1-A	Rep.-1	0.300	0.3244	1.081	11.686	1.108	9.860	0.225	0.75	0.23%
99-2049	A1-Cs-PR1-A	Rep.-2	0.500	0.5433	1.087	11.964	1.833	10.241	0.373	0.75	0.74%
99-2050	A1-Cs-PR2-A		0.400	0.4101	1.025	11.477	0.689	9.447	0.140	0.35	
99-2050	A1-Cs-PR2-A	Rep.-1	1.000	1.0345	1.035	11.796	1.735	9.862	0.353	0.35	0.72%
99-2050	A1-Cs-PR2-A	Rep.-2	1.000	1.0314	1.031	11.801	1.719	9.967	0.350	0.35	0.93%
99-2051	A1-Cs-PR3-A		1.000	1.018	1.018	11.583	0.933	9.608	0.190	0.19	
99-2051	A1-Cs-PR3-A	Replicate	2.000	2.0274	1.014	11.897	1.849	9.915	0.376	0.19	0.92%
99-2052	A1-Cs-PR4-A		2.000	2.019	1.010	11.655	1.192	9.479	0.242	0.12	
99-2052	A1-Cs-PR4-A	Replicate	3.000	3.0275	1.009	11.663	1.782	9.826	0.362	0.12	0.34%
99-2053	A1-Cs-PR5-A	*	1.000	1.0079	1.008	11.099					
99-2053	A1-Cs-PR5-A	Replicate	1.000	1.0078	1.008	10.901					
99-2054	A1-Cs-PR6-A	*	1.000	1.0064	1.006	11.026					
99-2054	A1-Cs-PR6-A	Replicate	1.000	1.0051	1.005	11.000					
99-2072	Regenerate-A	*	1.000	1.0027	1.003	11.173	0.315	8.563	0.064	0.06	
99-2072	Regenerate-A	Replicate	0.500	0.502	1.004	10.905	0.162	8.213	0.033	0.07	2.82%

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

ASR # **5421**
WP# **W48409**

File: L:\radchem\hydroxide\asr5421

Analysis Date: **7/1/99**

Print Date: 7/8/99

Hydroxide and Alkalinity Determination

Governing Procedures: **PNL-ALO-228**: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
and Operation of Brinkman 636 Auto-Titrator

Equip # **WB76843**

Lab Loc. **525**

Analyst: *R. J. Brown* 7/8/99

Reviewer: *J. J. Deen* 7/8/99

Titrant	Molarity
HCl	0.2034

Std. & Spike	Molarity
NaOH	0.1018

RPG #	Sample ID	Sample Vol. (mL)	Sample Wt. (g)	Density g/mL	Initial pH reading	OH		Found millimoles base	Molarity base	millimole RPD
						1st Equivalence Point Titrant Vol. (mL)	pH			
										% Recovery, Accuracy
Standard 1	0.1018 N NaOH	5.000	5.0371	1.007	11.862	2.504	7.751	0.5093	100.1%	standard
Standard 2	0.1018 N NaOH	5.000	5.0398	1.008	11.814	2.485	7.880	0.5054	99.3%	standard
99-2046MS	PW-1A + 2mL 0.1N NaOH	0.100	0.1228	1.228	11.700	1.967	10.062	0.400	95.3%	matrix spk
99-2047MS	PW-2A + 2mL 0.1N NaOH	0.100	0.1227	1.227	11.759	1.985	9.973	0.404	99.5%	matrix spk
99-2048MS	PW-3A + 2mL 0.1N NaOH	0.100	0.1175	1.175	11.849	1.751	9.950	0.356	93.9%	matrix spk
99-2049MS	PR1A + 2mL 0.1N NaOH	0.500	0.5404	1.081	11.969	2.764	10.097	0.562	92.2%	matrix spk
99-2050MS	PR2A + 2mL 0.1N NaOH	1.000	1.034	1.034	11.884	2.652	9.965	0.539	92.6%	matrix spk

* -- Volume restrictions existed

Performance checks

Balance # 360--01-06-037

Vol.

Wt.

Buffer	VWR Lot #	CMS#	Expire Date
10	981659-24	144109	Jul-00
4	981583-24	144107	Jun-00
7	981894-24	144108	Aug-00

Pipet #		Vol.	Wt.
Pipet #	H30762	5.00	4.9841
Pipet #	288618	0.300	0.2997
Pipet #	120737	0.100	0.1002

Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: *[Signature]* 7/8/99

Reviewer: *[Signature]* 7/8/99

Titrant	Molarity
HCl	0.2034

RPG #	Sample ID	Sample Vol. (mL)	CO3			Found millimoles base	Molarity base	millimole RPD	HCO3			Found millimoles base	Molarity base	millimole RPD
			2nd Equivalence Point Titrant Vol. (mL)	pH					3rd Equivalence Point Titrant Vol. (mL)	pH				
99-2046	A1-Cs-PW1-A	0	0.300	4.027	7.449	0.200	0.668							
99-2046	A1-Cs-PW1-A	Rep.-1	0.200	2.684	7.529	0.137	0.684	2.46%						
99-2046	A1-Cs-PW1-A	Rep.-2	0.200	2.714	7.558	0.138	0.692	1.03%	2.813	5.434	0.020	0.10		
99-2046	A1-Cs-PW1-A	Rep.-3	0.100	1.369	6.461	0.072	0.720	4.03%	1.404	5.477	0.007	0.07	34.32%	
99-2047	A1-Cs-PW2-A	0	0.300	3.983	7.262	0.219	0.730		4.244	4.769	0.053	0.18		
99-2047	A1-Cs-PW2-A	Replicate	0.200	2.682	7.576	0.135	0.674	7.97%	2.787	5.475	0.021	0.11	49.46%	
99-2048	A1-Cs-PW3-A	0	0.200	2.051	6.321	0.090	0.448		2.092	5.370	0.008	0.04		
99-2048	A1-Cs-PW3-A	Replicate	0.200	2.051	7.203	0.085	0.424	5.59%	2.141	4.912	0.018	0.09	74.81%	
99-2049	A1-Cs-PR1-A	0	0.200	0.923	5.560	0.038	0.189							
99-2049	A1-Cs-PR1-A	Rep.-1	0.300	1.374	6.596	0.054	0.180	4.77%	1.412	5.057	0.008	0.03		
99-2049	A1-Cs-PR1-A	Rep.-2	0.500	2.280	7.301	0.091	0.182	0.82%						
99-2050	A1-Cs-PR2-A	0	0.400	0.855	5.683	0.034	0.084							
99-2050	A1-Cs-PR2-A	Rep.-1	1.000	2.131	6.367	0.081	0.081	4.69%	2.227	4.460	0.020	0.020		
99-2050	A1-Cs-PR2-A	Rep.-2	1.000	2.127	6.461	0.083	0.083	2.99%	2.165	5.612	0.008	0.008	86.57%	
99-2051	A1-Cs-PR3-A	0	1.000	1.088	6.209	0.032	0.032		1.123	4.777	0.007	0.007		
99-2051	A1-Cs-PR3-A	Replicate	2.000	2.137	7.163	0.059	0.029	7.36%	2.248	4.457	0.023	0.011	45.30%	
99-2052	A1-Cs-PR4-A	0	2.000	1.300	6.265	0.022	0.011		1.336	4.536	0.007	0.004		
99-2052	A1-Cs-PR4-A	Replicate	3.000	1.947	6.762	0.034	0.011	1.83%	2.006	4.524	0.012	0.004	8.85%	
99-2053	A1-Cs-PR5-A	*	1.000	0.372	7.401	0.076	0.076		0.400	4.648				
99-2053	A1-Cs-PR5-A	Replicate	1.000	0.399	5.253	0.081	0.081	7.00%						
99-2054	A1-Cs-PR6-A	*	1.000	0.243	5.386	0.049	0.049							
99-2054	A1-Cs-PR6-A	Replicate	1.000	0.250	5.250	0.051	0.051	2.84%						
99-2072	Regenerate-A	*	1.000	0.333	7.324	0.004	0.004		0.383	4.266	0.010	0.010	10.43%	
99-2072	Regenerate-A	Replicate	0.500	0.192	4.380	0.006	0.012	192.89%						

Hydroxide and Alkalinity Determination

Governing Procedures: PNL-ALO-228: Determination of Hydroxyl (OH-) and
Alkalinity of Aqueous Solutions, Leachates and Supernates
and
Operation of Brinkman 636 Auto-Titrator
Equip # WB76843
Lab Loc. 525

Analyst: *[Signature]* 7/8/99

Reviewer: *[Signature]* 7/8/99

Titrant	Molarity
HCl	0.2034

CO3		
2nd Equivalence		
Point	Found	
Titrant	millimoles	Molarity
Vol. (mL)	base	base
pH	RPD	

HCO3		
3rd Equivalence		
Point	Found	
Titrant	millimoles	Molarity
Vol. (mL)	base	base
pH	RPD	

RPG #	Sample ID	Sample Vol. (mL)	CO3 Titrant Vol. (mL)	CO3 pH	CO3 millimoles base	CO3 Molarity base	CO3 millimole RPD	HCO3 Titrant Vol. (mL)	HCO3 pH	HCO3 millimoles base	HCO3 Molarity base	HCO3 millimole RPD
Standard 1	0.1018 N NaOH	5.000	2.557	4.121								
Standard 2	0.1018 N NaOH	5.000	2.545	4.246								
99-2046MS	PW-1A + 2mL 0.1N NaOH	0.100	2.355	6.271	0.07892	114.2%	sample	2.399	5.252	0.0089	104.1%	sample
99-2047MS	PW-2A + 2mL 0.1N NaOH	0.100	2.367	6.525	0.0777	110.6%	sample					sample
99-2048MS	PW-3A + 2mL 0.1N NaOH	0.100	2.026	6.573	0.05594	128.2%	sample	2.099	4.540	0.0148	222.9%	sample
99-2049MS	PR1A + 2mL 0.1N NaOH	0.500	3.230	7.517	0.09478	103.1%	sample	3.357	5.006	0.0258	200.5%	sample
99-2050MS	PR2A + 2mL 0.1N NaOH	1.000	3.072	7.48	0.08543	103.4%	sample	3.203	4.824	0.0266	195.5%	sample

Matrix spike recovery is calculated as follows:

Spike = 2.00 mL 0.1018 N NaOH was added to the 0.100-mL of sample for each matrix spilke.

SpikeTitrant vol. (sample @ .1mL + spike) - SampleTitrant vol. (average sample only equated to .1mL) * 0.2034 N (HCl titrant) = meq. OH

meq OH / 2.00 mL added = meq OH/mL found / 0.1018 N OH added * 100 = % recovered.

Prep record on 0.2034 M HCl is on following page.

Chem Rec_51a

Prep date: 4/18/99

Preparation of Standardized 0.2 M HCl

WP# K51300

Standardized 0.1021 M NaOH will be re-checked and then used to standardized the ~ 0.1 M HCl solution. The 0.1021 M NaOH was prepared in Chem Rec_37 (see Chem Rec_37 --prep.date 2-25-98 for original data) and re-verified against NIST SRM84j Potassium Acid Phthalate KHC₈H₄O₄ (KAP) = 204.23 g/mole -- Barcode # 52232 -- (see below verification check).

The re-standardized value of 0.1018 M NaOH was reassigned to this NaOH solution with a revised Expiration Date of Feb. 2000.

Prepared 1- liters of ~0.2 M HCl by diluting 100 mL of 1.029M HCl (Chemrec_10) to 0.5 L with DI. H₂O.

20 mL aliquots of 0.2 M HCl were were neutralized to the phenophthalien endpoint using the re-standardized 0.1018 M NaOH. The volume of NaOH is accurate to +/- 0.02mL and the pipitting error is estimated to be < 1% @ 1s. Thus total error is < 3 % for the measurements

NaOH Molarity veification

Verification Test #	Wt. of KAP	Vol. of 0.1021M NaOH to neutralize	NaOH Molarity =a * 1000 / b * 204.23	Molarity Error +/- @ 1 s
1	0.80894	38.95	0.1017	
2	0.80582	38.84	0.1016	
3	0.96233	46.12	0.1022	
Ave=			0.1018	0.0003
			re-certified value	

Titration Id.	aliquot of sample	Vol. of 0.1018M NaOH to neutralize	Molarity of Acid in Sample	Molarity Error +/- @ 1 s
1	20.00	39.88	0.2030	
2	20.00	39.92	0.2032	
3	20.00	40.04	0.2038	
Ave Molarity HCl =			0.2034	0.00042

Analyst/Date

Battelle, Pacific Northwest National Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

RECORD
COPY

99-2019
6/30/99

Client : Dean Kurath

Cognizant Scientist:

C. Soderjans

Date :

6-30-99

Concur :

T. Trang-b

Date :

6/30/99

Measured Activities ($\mu\text{Ci/g}$)

ALO ID Client ID	Cs-134 $\pm 1s$	Cs-137 $\pm 1s$	Co-60 $\pm 1s$	SnSb-126 $\pm 1s$	RuRh-106 $\pm 1s$	Sb-125 $\pm 1s$	Eu-154 $\pm 1s$	Densities of Samples	
								g	ml
99-2019	1.50E-3	8.17E+0	<2E-4	<2E-3				2.0396	1.992
A1-Cs-OA	21%	2%							
99-2020	<2E-4	1.25E-2	5.82E-4	2.16E-4				2.4625	1.992
A1-Cs-L1A		3%	7%	18%					
99-2024	1.08E-3	4.68E+0	5.25E-4	<2E-3				2.4743	1.992
A1-Cs-L5A	21%	2%	12%						
99-2026	2.82E-3	1.59E+1	6.76E-4					2.4621	1.992
A1-Cs-L7A	11%	2%	9%						
99-2029	7.49E-3	3.56E+1	6.06E-4					2.4671	1.992
A1-Cs-L10A	7%	2%	17%						
99-2032	8.85E-4	3.99E+0						2.0756	1.992
A1-Cs-L13A	17%	2%							
99-2036	1.48E-3	1.07E+1						2.0649	1.992
A1-Cs-L17A	26%	2%							
99-2037	1.27E-5	3.07E-3	4.28E-4	2.16E-4	3.74E-4	1.10E-4	4.58E-5	2.4553	1.992
A1-Cs-P1A	18%	4%	2%	3%	6%	8%	9%		
99-2039	2.96E-4	1.63E+0	5.19E-4	3.93E-4				2.4631	1.992
A1-Cs-P3A	25%	2%	9%	26%					
99-2041	2.49E-3	1.38E+1	6.59E-4					2.4741	1.992
A1-Cs-P5A	11%	2%	9%						
99-2043	6.26E-4	3.77E+0						2.0728	1.992
A1-Cs-P7D	24%	2%							
99-2045	1.38E-3	7.15E+0						2.0632	1.992
A1-Cs-P9A	10%	2%							
99-2046	7.56E-3	5.59E+1						2.4673	1.992
A1-Cs-PW1A	22%	2%							
99-2047	8.46E-3	5.39E+1						2.4729	1.992
A1-Cs-PW2-A	23%	2%							
99-2048	8.69E-3	4.32E+1						2.3377	1.992
A1-CS-PW3-A	17%	2%							
99-2049	6.81E-3	3.29E+1						2.1463	1.992
A1-Cs-PR1-A	7%	2%							
99-2050	3.49E-3	1.78E+1						2.0695	1.992
A1-Cs-PR2-A	9%	2%							
99-2051	2.46E-3	1.40E+1						2.0299	1.992
A1-Cs-PR3-A	10%	2%							

ALO ID Client ID	Measured Activities ($\mu\text{Ci/g}$)							Densities of Samples	
	Cs-134 $\pm 1s$	Cs-137 $\pm 1s$	Co-60 $\pm 1s$	SnSb-126 $\pm 1s$	RuRh-106 $\pm 1s$	Sb-125 $\pm 1s$	Eu-154 $\pm 1s$	g	ml
99-2052 A1-Cs-PR4-A	1.03E-3 23%	5.16E+0 2%						2.014	1.992
99-2053 A1-Cs-PR5-A	4.22E-4 41%	2.31E+0 2%						2.0078	1.992
99-2054 A1-Cs-PR6-A	4.18E-4 26%	1.30E+0 2%						2.002	1.992
99-2055 A1-Cs-E1-2A	<1E-4	4.56E-2 2%						2.0299	1.992
99-2056 A1-Cs-E1-3A	<2E-4	7.17E-2 2%						2.0278	1.992
99-2057 A1-Cs-E1-4A	7.51E-4 22%	3.56E+0 2%						2.0318	1.992
99-2058 A1-Cs-E1-5A	3.28E-2 10%	1.68E+2 2%						2.0336	1.992
99-2059 A1-Cs-E1-6A	2.29E-3 10%	1.19E+1 2%						2.0287	1.992
99-2060 A1-Cs-E1-7A	<2E-4	1.85E-1 2%						2.0303	1.992
99-2062 A1-Cs-E-9A	<2E-4	2.30E-1 2%						2.0305	1.992
99-2065 A1-Cs-E1-12A	4.29E-4 25%	1.91E+0 2%						2.0297	1.992
99-2066 A1-Cs-E1-R1-A	1.19E-3 11%	5.11E+0 2%	4.62E-4 10%					2.026	1.992
99-2068 A1-Cs-E1-R3-A	2.08E-4 29%	4.44E-1 2%	2.74E-4 14%					1.998	1.992
99-2071 A1-Cs-E1-R6-A	2.73E-4 16%	1.11E+0 2%	<9E-5					1.9941	1.992
99-2072 Regeneration soln	<9E-5	6.84E-2 2%	<2E-4					2.003	1.992
99-2079 Eff-1A	1.54E-3 10%	8.89E+0 2%	5.04E-4 10%					2.4646	1.992
99-2080 Eff-2A	8.79E-3 16%	4.57E+1 2%	<2E-3					2.4689	1.992

Radiochemistry Bench Sheet

Client
Work Order #
ASR

D. KURATH

W48409

5421

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+2.8.3.3

RECORD
COPY

Balance 360-06-01-037

Procedure

PNL-ALO-450

Sample ID

TOTAL VIAL
MASS

Dilutions and Aliquot Sizes

GEA Aliquots

Volume (mL)

Mass (g)

Pipet ID Numbers

Pipet Checks

T=22°C

A1-C ₅ -ICP	99-2018	18.41060g			#288618
A1-C ₅ -OA	-2019	26.4155g	2 mL	2.0396g	0.9999g $\bar{x}=0.9947g$
A1-C ₅ -LIA	-2020	21.5918g	2 mL	2.4625g	0.9970g $\pm 0.4\%$
A1-C ₅ -L3A	-2021	20.5912g			0.9919g $\pm 0.9960ml$
-L3A	-2022	19.8026g			0.9915g
-L4A	-2023	21.4377g			0.9932g
A1-C ₅ -L5A	-2024	21.2055g	2 mL	2.4743g	
L6A	-2025	20.8071g			#430754 @ 10ml
A1-C ₅ -L7A	-2026	21.8738g	2 mL	2.4621g	10.0532g
L8A	-2027	20.7730g			10.0188g
L9A	-2028	20.7884g			10.0483g
L10A	-2029	20.8034g	2 mL	2.4671g	10.0337g
L11A	-2030	20.9691g			10.0293g
L12A	-2031	21.8970g			
L13A	-2032	22.0174g	2 mL	2.0756g	
L14A	-2033	21.8437g			
L15A	-2034	21.7822g			
L16A	-2035	22.0348g			
L17A	-2036	21.8065g	2 mL	2.0649g	
PIA	-2037	20.7496g	2 mL	2.4553g	

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date

Lori P. Darnell 6-23-99

PK. Bury

6-23-99

Reviewer/Date

Richard T. R 6/29/99

NOT
USED
6/23/99

not H

Radiochemistry Bench Sheet

Client D. KURATH
 Work Order # W48409
 ASR 5421

Balance 360-06-01-037

Procedure PNL-ALO-450

Sample ID	TOTAL VIAL MASS	Dilutions and Aliquot Sizes	GEA Aliquots		Pipet ID Numbers Pipet Checks
			Volume	Mass	
P2A	99-2038	19.9357g			
P3A	-2039	20.9946g	2 ml	2.4631g	
P4A	-2040	20.6313g			
P5A	-2041	20.4298g	2 ml	2.4741g	See pg. 1 of 4
P6A	-2042	20.8414g			
P7A	-2043	22.1437g ^{pro 4/22/99} 349g	2 ml	2.0728g	
P8A	-2044	22.0841g			
P9A	-2045	21.8967g	2 ml	2.0632g	
PW1-A*	-2046	22.3526g		2.4675g	.1 ml
PW2-A*	-2047	22.3853g		2.4729g	.1 ml
PW3-A*	-2048	22.1076g		2.3377g	.1 ml
PR1-A	-2049	31.7156g		2.1463g	
PR2	-2050	32.3299g		2.0695g	
PR3	-2051	32.4953g		2.0299g	
PR4	-2052	31.9417g		2.0140g	
PR5	-2053	26.3271g		2.0078g	
PR6	-2054	36.7529g		2.0025g	
E1-2	-2055	27.1235g		2.0299g	
E1-3	-2056	26.8506g		2.0278g	
E1-4	-2057	26.8591g		2.0318g	

Det #

Cal #

Spike, Tracer ID
 Isotope
 Concentration
 Ref Date

Analyst/Date

Lori P. Darnell 6-23-99
P.K. Benz 6-23-99

Reviewer/Date

Richard T. R. 6/29/99

Radiochemistry Bench Sheet

Client D. KURATH
 Work Order # W48409
 ASR 5421

Balance 360-06-01-037

Procedure PNL-ALD-450

GEA Aliquots

Pipet ID Numbers
 Pipet Checks

TOTAL VIAL
 mass
 Dilutions and Aliquot Sizes

Sample ID	Volume	Mass	Pipet Checks
81-5 ~~81-5~~ <u>91-2058</u>	2 ml	2.0336g	.1 ml
81-6 <u>-2059</u>	↓	2.0287g	
81-7 <u>-2060</u>	↓	2.0303g	
81-8 <u>-2061</u>			
81-9 <u>-2062</u>	2 mL	2.0305g	
81-10 <u>-2063</u>			
81-11 <u>-2064</u>			
81-12 <u>-2065</u>	2 ml	2.0297g	
R1 ~~81-13~~ <u>-2066</u>	↓	2.0260g	
R2 <u>-2067</u>			
R3 <u>-2068</u>	2 mL	1.9980g	
R4 <u>-2069</u>			
R5 <u>-2070</u>			
R6 <u>-2071</u>	2 ml	1.9941g	
Regen <u>-2072</u>	↓	2.0030g	
#2 eluent - fin <u>-2073</u>		19.9310g	
#2 eluent - comp. <u>-2074</u>		20.7666g	
0.1 M NaOH <u>-2075</u>		21.1962g	
0.25 M NaOH <u>-2076</u>		21.4358g	
0.5 M HNO ₃ <u>-2077</u>		21.2846g	

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date

Lori P. Darnell 6-23-99

Reviewer/Date

Richard T. R. 6/29/99

P.K. Berry 6/23/99

Radiochemistry Bench Sheet

Client D. KURATHI
Work Order # W48409
ASR 5421

Balance 360-06-01-037

Procedure PNL-ALO-450

GEA Aliquots

Pipet ID Numbers
Pipet Checks

Sample ID	TOTAL VIAL MASS Dilutions and Aliquot Sizes

Volume

Mass

Pipet Checks

DI
EFFIA.
EFF2A*

Sample ID
99-2078

TOTAL VIAL
MASS

Dilutions and Aliquot Sizes

20.61619

20.7529g

21. 0023g

3ml

2.4646g

2ml

2.4689

1 ml See pag 1 of 4

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date

Lou P. Darnell 6-23-99

P.K. Berry 6/23/99

Reviewer/Date

Richard T. B. 6/29/99

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

12/6/1999

Client : Kurath

Cognizant Scientist:

L.R. GreenwaldDate : 12/6/99

Concur :

Richard T.R.Date : 12/7/99

proj 29953

+ 2.8.3.3

Task 2.8

Procedure: PNL-ALO-450

1/5

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

ALO ID Client ID	Co-60 Error %	Rurh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Y-88 Error %	Tc-95 Error %	Tc-95M Error %	Co-57 Error %
00-00295 C1-Cs-ICP	4.86E-2 3%	<2.E-1	<2.E-1	<5.E-2	<4.E-3	1.26E+2 2%	3.24E-2 6%	<8.E-2	<7.E-2				
00-00296 C1-Cs-0A	7.10E-4 4%	<3.E-3	<2.E-3	<6.E-4	<6.E-5	1.60E+0 2%	3.39E-4 14%	<2.E-3	<2.E-3				
00-00297 C1-Cs-L1A	4.38E-2 2%	<2.E-2	<7.E-3	<3.E-3	2.93E-4 35%	2.93E+1 2%	2.80E-2 2%	1.67E-2 10%	9.77E-3 17%				
00-00298 C1-Cs-L3A	4.43E-2 2%	<9.E-2	<6.E-2	<3.E-2	<2.E-3	5.29E+1 2%	2.90E-2 5%	<4.E-2	<4.E-2				
00-00299 C1-Cs-L6A	4.69E-2 2%	<5.E-2	<3.E-2	<2.E-2	<1.E-3	8.81E+1 2%	2.85E-2 3%	<2.E-2	<2.E-2				
00-00300 C1-Cs-L9B	5.16E-4 6%	<5.E-3	<3.E-3	<1.E-3	<7.E-5	1.13E+0 2%	4.01E-4 12%	<2.E-3	<2.E-3				
00-00301 C1-Cs-L12B	5.40E-4 6%	<5.E-3	<3.E-3	<1.E-3	<7.E-5	1.16E+0 2%	4.94E-4 12%	<3.E-3	<2.E-3				
00-00302 C1-Cs-P1A	3.52E-2 2%	<8.E-4	2.98E-4 28%	3.20E-4 6%	<2.E-4	1.08E-3 5%	2.52E-2 2%	1.79E-2 3%	9.03E-4 5%				
00-00303 C1-Cs-P2A	4.00E-2 2%	<2.E-3	5.57E-4 25%	3.58E-4 8%	<2.E-4	4.70E-3 3%	2.83E-2 2%	1.96E-2 3%	1.03E-2 7%				

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

ALO ID Client ID	Co-60 Error %	Rurh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Y-88 Error %	Tc-95 Error %	Tc-95M Error %	Co-57 Error %
00-00304 C1-Cs-P3A	4.26E-2 2%	<2.E-3	7.81E-4 20%	2.82E-4 11%	<2.E-4	9.59E-3 2%	2.85E-2 2%	1.95E-2 3%	1.02E-2 7%				
00-00305 C1-Cs-P6A	4.47E-2 2%	<3.E-3	<1.E-3	<4.E-4	<3.E-4	4.34E-1 2%	2.79E-2 2%	2.04E-2 3%	1.04E-2 5%				
00-00306 C1-Cs-FD1-B	4.64E-2 2%	<3.E-3	<2.E-3	<5.E-4	<2.E-4	9.00E-1 2%	2.93E-2 2%	2.09E-2 3%	1.08E-2 5%				
00-00307 C1-Cs-FD2-B	4.50E-2 2%	<3.E-3	<2.E-3	<6.E-4	<2.E-4	7.96E-1 2%	2.72E-2 2%	1.99E-2 3%	1.08E-2 5%				
00-00308 C1-Cs-FD3-B	4.91E-2 2%	<2.E-3	<1.E-3	<4.E-4	<2.E-4	6.03E-1 2%	1.63E-2 2%	1.17E-2 3%	6.56E-3 6%				
00-00309 C1-Cs-FD4-B	4.06E-2 2%	<2.E-3	<1.E-3	<3.E-4	<2.E-4	4.69E-1 2%	8.21E-3 2%	5.97E-3 5%	3.27E-3 13%				
00-00310 C1-Cs-FD5-B	2.80E-2 2%	<2.E-3	<7.E-4	<3.E-4	<2.E-4	3.00E-1 2%	4.95E-3 2%	3.53E-3 4%	2.51E-3 8%				
00-00311 C1-Cs-FD6-B	1.77E-2 2%	<2.E-3	<6.E-4	<3.E-4	<2.E-4	1.65E-1 2%	3.15E-3 3%	2.16E-3 6%	1.24E-3 12%				
00-00312 C1-Cs-FD7-B	1.21E-2 2%	<9.E-4	<5.E-4	<2.E-4	<8.E-5	1.80E-1 2%	2.09E-3 3%	1.34E-3 8%	1.04E-3 17%				
00-00313 C1-Cs-PR1-A	6.71E-3 2%	<8.E-4	<5.E-4	<8.E-5	<6.E-5	1.26E-1 2%	9.12E-4 5%	6.53E-4 16%	6.95E-4 29%				
00-00314 C1-Cs-PR2-A	4.25E-3 2%	<5.E-4	<3.E-4	<3.E-4	<3.E-5	1.14E-1 2%	5.32E-4 4%	3.47E-4 11%	2.38E-4 22%				
00-00315 C1-Cs-PR3-A	4.67E-3 2%	<5.E-4	<3.E-4	<3.E-4	<4.E-5	7.31E-2 2%	1.28E-3 2%	9.27E-4 5%	5.50E-4 11%				
00-00316 C1-Cs-PR4-A	3.46E-3 2%	<3.E-4	<2.E-4	<3.E-5	<3.E-5	3.83E-2 2%	4.81E-4 4%	3.76E-4 9%	1.74E-4 26%				
00-00317 C1-Cs-PR5-A	2.71E-3 2%	<2.E-4	<2.E-4	<3.E-5	<2.E-5	2.51E-2 2%	3.18E-4 4%	2.05E-4 11%	1.32E-4 27%				

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

ALO ID Client ID	Co-60 Error %	Rurh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Y-88 Error %	Tc-95 Error %	Tc-95M Error %	Co-57 Error %
00-00318 C1-Cs-PR6-A	2.11E-3 2%	<2.E-4	<7.E-5	<2.E-5	<2.E-5	1.97E-2 2%	1.94E-4 4%	1.40E-4 10%	<3.E-5				
00-00319 C1-Cs-E1-1B	1.03E-4 6%	<9.E-4	<5.E-4	<5.E-4	<2.E-5	2.13E-1 2%	<5.E-5	<3.E-4	<3.E-4				
00-00320 C1-Cs-E1-3B	<2.E-5	<6.E-4	<4.E-4	<6.E-5	<3.E-5	5.41E-2 2%	<4.E-5	<3.E-4	<3.E-4				
00-00321 C1-Cs-E1-4B ²	<7.E-6	<7.E-4	<4.E-4	<2.E-4	<9.E-6	1.46E-1 3%	<2.E-5	<3.E-4	<3.E-4				
00-00322 C1-Cs-E1-5B ²	<7.E-6	<9.E-4	<6.E-4	<6.E-4	<2.E-5	3.41E-1 3%	<2.E-5	<3.E-4	<3.E-4				
00-00323 C1-Cs-E1-6B ²	<3.E-5	<2.E-3	<9.E-4	<2.E-4	<4.E-5	3.31E-1 2%	<6.E-5	<6.E-4	<7.E-4				
00-00324 C1-Cs-E1-7B1	5.66E-5 8%	<9.E-4	<6.E-4	<9.E-5	<2.E-5	3.21E-1 2%	<4.E-5	<4.E-4	<4.E-4				
00-00325 C1-Cs-E1-9B1	3.34E-4 5%	<2.E-3	<1.E-3	<4.E-4	<3.E-5	4.93E-1 2%	<7.E-5	<6.E-4	<6.E-4				
00-00326 C1-Cs-E1-11B1	2.48E-4 5%	<2.E-3	<7.E-4	<2.E-4	<4.E-5	2.54E-1 2%	<6.E-5	<5.E-4	<6.E-4				
00-00327 C1-Cs-E1-13A	9.09E-4 3%	<3.E-3	<2.E-3	<3.E-4	<9.E-5	8.98E-1 2%	<2.E-4	<2.E-3	<2.E-3				
00-00328 C1-Cs-E1-R1A	1.55E-3 4%	<8.E-3	<5.E-3	<9.E-4	<3.E-4	3.32E+0 2%	<4.E-4	<4.E-3	<5.E-3				
00-00329 C1-Cs-E1-R3A	1.57E-3 2%	<2.E-3	<7.E-4	<3.E-4	<4.E-5	3.63E-1 2%	<6.E-5	<5.E-4	<5.E-4				
00-00330 C1-Cs-E1-R6A	1.78E-4 3%	<3.E-4	<2.E-4	<7.E-5	3.46E-5 12%	5.50E-2 2%	<2.E-5	<1.E-4	<1.E-4				

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

ALO ID Client ID	Co-60 Error %	Rurh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Y-88 Error %	Tc-95 Error %	Tc-95M Error %	Co-57 Error %
00-00331 Regeneration	4.56E-4 2%	<2.E-4	<8.E-5	<3.E-5	2.95E-5 10%	2.29E-2 2%	<2.E-5	<5.E-5	<5.E-5				
00-00332 C1-CA	2.98E-3 3%	<2.E-2	<7.E-3	<3.E-3	<3.E-4	7.41E+0 2%	6.04E-4 23%	<6.E-3	<5.E-3				
00-00333 C1-S1A	2.85E-3 3%	<2.E-2	<7.E-3	<3.E-3	<3.E-4	7.30E+0 2%	2.20E-3 7%	<6.E-3	<5.E-3				
00-00334 C1-S2A	2.86E-3 3%	<2.E-2	<7.E-3	<3.E-3	<3.E-4	7.29E+0 2%	1.78E-3 9%	<6.E-3	<5.E-3				
00-00335 C1-44A	2.45E-3 2%	<2.E-3	<2.E-3	<3.E-4	<7.E-5	7.16E-1 2%	1.65E-3 4%	1.43E-3 20%	<2.E-3				
00-00336 C1-44-D-A	2.50E-3 2%	<2.E-3	<2.E-3	<4.E-4	<6.E-5	5.49E-1 2%	1.67E-3 4%	8.36E-4 19%	7.47E-4 30%				
00-00337 C1-44-S1-A	2.51E-3 2%	<4.E-3	<3.E-3	<8.E-4	<8.E-5	1.40E+0 2%	1.73E-3 5%	<2.E-3	<2.E-3				
00-00338 C1-44-S1-D	2.52E-3 2%	<4.E-3	<3.E-3	<8.E-4	<8.E-5	1.49E+0 2%	1.64E-3 5%	1.12E-3 30%	<2.E-3				
00-00339 C1-44-S2-A	2.53E-3 3%	<7.E-3	<4.E-3	<2.E-3	<2.E-4	3.19E+0 2%	1.80E-3 5%	<3.E-3	<3.E-3				
00-00340 C1-44-S2-D	2.45E-3 3%	<6.E-3	<4.E-4	<2.E-3	<1.E-4	2.46E+0 2%	1.65E-3 6%	<3.E-3	<3.E-3				
00-00341 A1R-Cs-0	5.14E-4 5%	<1.E-2	<6.E-3	<6.E-3	3.45E-3 4%	2.09E+1 2%	<3.E-4	<4.E-3	<4.E-3				
00-00342 A1R-Cs-L1	2.91E-4 2%	<3.E-4	<2.E-4	1.82E-4 10%	4.92E-5 10%	2.11E-1 2%	3.36E-5 20%	<2.E-4	<2.E-4				
00-00343 A1R-Cs-L2	3.03E-4 2%	<4.E-4	<3.E-4	1.61E-4 4%	6.19E-5 6%	3.12E-1 2%	3.23E-5 18%	<2.E-4	<2.E-4				
00-00344 A1R-Cs-P1	2.79E-4 2%	3.40E-4 10%	8.60E-5 19%	2.12E-4 3%	1.62E-5 15%	1.85E-2 2%	1.72E-5 24%	<5.E-5	<5.E-5				

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

ALO ID Client ID	Co-60 Error %	Rurh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %	Y-88 Error %	Tc-95 Error %	Tc-95M Error %	Co-57 Error %
00-00345 A1R-Cs-P2	3.08E-4 2%	2.78E-4 12%	9.85E-5 17%	2.18E-4 2%	4.91E-5 7%	1.97E-2 2%	2.87E-5 15%	<6.E-5	<7.E-5				
00-00346 A1R-Cs-P7	2.77E-4 2%	2.30E-4 14%	6.97E-5 24%	1.77E-4 3%	7.31E-6 37%	2.91E-2 2%	1.78E-5 21%	<5.E-5	<7.E-5				
00-00347 A1-Cs-E2-CO	<1.E-5	<2.E-3	<7.E-4	<2.E-4	6.76E-5 22%	4.54E-1 2%	<5.E-5	<5.E-4	<6.E-4				
00-00348* A1-Tc-EC1	<2.E-6	<2.E-4	<6.E-5	<2.E-5	<2.E-5	6.61E-5 10%	<5.E-6	<7.E-5	<6.E-5	9.93E-6 7%	4.61E-3 2%	9.76E-2 2%	
00-00349** A1-Tc-LC	2.88E-4 2%	2.83E-4 8%	8.48E-5 20%	2.04E-4 4%	9.00E-6 16%	5.89E-2 2%	2.33E-5 8%	<4.E-5	<6.E-5	2.14E-5 4%	8.52E-5 4%	1.80E-3 2%	6.84E-5 38%
00-00365 C1-Cs-OAR	7.29E-3 3%	<3.E-2	<2.E-2	<6.E-3	<6.E-4	1.81E+1 2%	4.65E-3 8%	<1.E-2	<9.E-3				

*Sample activity of Tc-95m as of 11/17/99 at 15:00

**Sample activity of Tc-95m as of 11/18/99 at 14:30

CLIENT D. Kurath
ASR 5571

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Proj 29953
T2-8.3.3
Tash 2-8
D1/3

ANALYST MJ Stile DATE 11/5/99
REVIEWER _____ DATE _____

Dilutions should be done by mass.		VIAL	VIAL+ SAMPLE	MASS	VIAL + SAMPLE + DDI	DILUTION	GEA GEOMETRY	Minimum reportable quantities (MRQ)	Notes
Sample ID									
C1-Cs-ICP	00-0295	16.7602	17.9922	1.232	18.9798	1.80	2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL)	
C1-Cs-0A	00-0296	16.7755	17.7844	1.0089	18.7732	1.98	2	Cs-137; 9 uCi/mL	extra long count for minor radionuclides requested
C1-Cs-L1A	00-0297	16.7903	18.0211	1.2308	19.0117	1.80	2	Cs-137; 9 uCi/mL	extra long count for minor radionuclides requested
C1-Cs-L3A	00-0298	16.6708	19.143	2.4722			2	Cs-137; 9 uCi/mL	
C1-Cs-L6A	00-0299	16.658	19.1424	2.4844			2	Cs-137; 9 uCi/mL	
C1-Cs-L9B	00-0300	16.9888	22.0289	5.0401	26.9407	1.97	10	Cs-137; 9 uCi/mL	
C1-Cs-L12B	00-0301	16.9657	22.0073	5.0416	26.9156	1.97	10	Cs-137; 9 uCi/mL	
C1-Cs-P1A	00-0302	16.7541	19.2027	2.4486			2	Cs-137; 9 uCi/mL	extra long count for minor radionuclides requested
C1-Cs-P2A	00-0303	16.7488	17.9864	1.2376	18.9755	1.80	2	Cs-137; 9 uCi/mL	extra long count for minor radionuclides requested
C1-Cs-P3A	00-0304	16.782	18.0306	1.2486	19.021	1.79	2	Cs-137; 9 uCi/mL	extra long count for minor radionuclides requested
C1-Cs-P6A	00-0305	16.9608	18.2027	1.2419	19.1875	1.79	2	Cs-137; 9 uCi/mL	
C1-Cs-FD1-B	00-0306	16.808	19.2878	2.4798			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample
C1-Cs-FD2-B	00-0307	16.948	19.4158	2.4678			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample
C1-Cs-FD3-B	00-0308	16.9552	19.3009	2.3457			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample
C1-Cs-FD4-B	00-0309	16.7455	18.9407	2.1952			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample
C1-Cs-FD5-B	00-0310	16.683	18.7853	2.1023			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample
C1-Cs-FD6-B	00-0311	16.9792	19.0251	2.0459			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample
C1-Cs-FD7-B	00-0312	16.9417	18.9551	2.0134			2	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	Need to load out undiluted sample

GEA PREP SHEET

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Kurath

ANALYST

MJ Steele

DATE

11/5/99

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DATE

dilutions should be done by mass.		VIAL	VIAL + SAMPLE	MASS	VIAL + SAMPLE + DDI	DILUTION	GEA GEOMETRY	Minimum reportable quantities (MRQ)	Notes
Sample ID									
C1-Cs-PR1-A	00-0313	16.6851	26.7142	10.0291			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-Cs-PR2-A	00-0314	17.0297	27.0518	10.0221			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-Cs-PR3-A	00-0315	16.7783	26.8425	10.0642			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-Cs-PR4-A	00-0316	16.9674	26.9676	10.0002			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-Cs-PR5-A	00-0317	16.7893	26.7803	9.991			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-Cs-PR6-A	00-0318	16.7316	26.6941	9.9625			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-Cs-E1-1B	00-0319	16.7099	18.7458	2.0359			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-3B	00-0320	16.7883	18.8182	2.0299			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-4B2	00-0321	16.6441	21.7182	5.0741			10	Cs-137; 9 uCi/mL	
C1-Cs-E1-5B2	00-0322	16.8426	21.878	5.0354			10	Cs-137; 9 uCi/mL	
C1-Cs-E1-6B2	00-0323	16.7339	18.7569	2.023			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-7B1	00-0324	16.7446	18.7761	2.0315			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-9B1	00-0325	16.6514	18.6798	2.0284			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-11B1	00-0326	16.7359	18.7691	2.0332			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-13A	00-0327	16.7129	26.8081	10.0952			10	Cs-137; 9 uCi/mL	
C1-Cs-E1-R1-A	00-0328	16.8581	18.8788	2.0207			2	Cs-137; 9 uCi/mL	
C1-Cs-E1-R3-A	00-0329	16.8183	26.9791	10.1608			10	Cs-137; 9 uCi/mL	
C1-Cs-E1-R6-A	00-0330	16.727	26.7102	9.9832			10	Cs-137; 9 uCi/mL	
regeneration soln	00-0331	16.7578	26.7397	9.9819			10	Cs-137; 9 uCi/mL, Na, K (75 ug/mL), OH (17 ug/mL)	
C1-CA	00-0332	16.7378	21.794	5.0562	26.6989	1.97	10		
C1-S1A	00-0333	16.6398	21.6825	5.0427	26.571	1.97	10		
C1-S2A	00-0334	16.6644	21.7125	5.0481	26.6167	1.97	10		
C1-44A	00-0335	16.6225	21.6696	5.0471	26.5551	1.97	10		
C1-44-D-A	00-0336	16.8092	21.8543	5.0451	26.7674	1.97	10		
C1-44-S1-A	00-0337	16.7603	21.7962	5.0359	26.7005	1.97	10		

CLIENT

Kwatha
5571

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ANALYST

Mg Steach

DATE

11/5/99

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DATE

itions should be done by mass.	VIAL	VIAL+ SAMPLE	MASS	VIAL + SAMPLE + DDI	DILUTION	GEA GEOMETRY	Minimum reportable quantities (MRQ)	Notes
Sample ID								
C1-44-S1-D-A	16.6993	21.7415	5.0422	26.6497	1.97	10		
C1-44-S2-A	16.7008	21.749	5.0482	26.6393	1.97	10		
C1-44-S2-D-A	16.81	21.8646	5.0546	26.7551	1.97	10		
A1R-Cs-0	16.7654	18.0063	1.2409	18.9992	1.80	2		extra long count for minor radionuclides requested (stored in RMA in 410), some solids in vial, mix up a bit before using
A1R-Cs-L1	16.7184	19.1763	2.4579			2		extra long count for minor radionuclides requested (stored in RMA in 410), some solids in vial, mix up a bit before using
A1R-Cs-L2	16.8217	19.2722	2.4505			2		extra long count for minor radionuclides requested (stored in RMA in 410), some solids in vial, mix up a bit before using
A1R-Cs-P1	16.8819	19.3285	2.4466			2		extra long count for minor radionuclides requested (stored in RMA in 410), some solids in vial, mix up a bit before using
A1R-Cs-P2	16.7088	19.1687	2.4599			2		extra long count for minor radionuclides requested (stored in RMA in 410), some solids in vial, mix up a bit before using
A1R-Cs-P7	16.1667	19.0743	2.9076			2		extra long count for minor radionuclides requested (stored in RMA in 410), some solids in vial, mix up a bit before using
A1-Cs-E2-CO	16.7207	18.7355	2.0148			2		Cs is main rad of interest but report others if detected (RMA in 410)
A1-Tc-EC1	16.7414	26.8372	10.0958			10	see table 6-1	Extra long count for minor radionuclides requested
A1-Tc-LC	16.6691	28.8807	12.2116			10		Extra long count for minor radionuclides requested
C1-CS-0AR	16.7944	18.8635	2.0691			2		

Radiochemical Bench Sheet

Client
Work Order #
ASR

Procedure

Sample ID

Dilutions and Aliquot Sizes

Balance

Pipet ID Numbers
Pipet Checks

 $T = 19^{\circ}\text{C}$

#07544 0.5ml

0.4993

0.4967

0.4972

0.4987

ave = 0.4920g

V = 0.4989ml

#105722

10ml 9.9691

9.9576

9.9510

9.9462

9.9560g

V = 9.9719ml

5ml 4.9507

4.9302

4.9317

4.9251

ave = 4.9344

V = 4.9423ml

#126686

1ml 0.9921

0.9918

0.9907

0.9897

ave = 0.9911g

V = 0.9927

Spike, Tracer ID
Isotope
Concentration
Ref Date

Analyst/Date

Reviewer/Date

mg steel 11/4/99

GEA + ICP
Prep

ID Kurath

5571

Prep

 $\rho_{\text{water}} @ 19^{\circ}\text{C} = 0.99840 \text{ g/ml}$

Eluate Composite Analytical Results

Battelle

Pacific Northwest Laboratories

Project Number

Internal Distribution

Date December 21, 1999

329/4 File

Mike Urie

To Dave Blanchard

From Tom Farmer *Quill Thomas Farmer*

Subject ICP/MS Analysis of Submitted Samples
(ACL #00-0615 through 00-0619)

Pursuant to your request, the 7 samples that you submitted for analysis were analyzed by ICPMS for ^{99}Tc . The results of this analysis are reported on the attached page.

An Amersham ^{99}Tc standard was used to generate the calibration curve and an independent Amersham ^{99}Tc standard was used as the continuing calibration verification (CCV) standard. The 1% high-purity nitric acid solution used to dilute the standards and samples was used as a reagent blank. The samples were diluted an extra 10x from the dilutions received. The results include your dilutions and are reported in ng analyte/ g (ppb) of original sample $\pm$ one standard deviation.

The ^{99}Tc values reported assume that the Ru present is exclusively fission-product Ru, and therefore does not have an isotope at m/z 99; i.e., everything observed at m/z 99 is due to ^{99}Tc . The fingerprint we're seeing for Ru is obviously not natural, and is consistent with that observed in previous tank waste analyses. Semiquantitative Ru concentrations, corrected for sample dilution, are provided for your information.

If you have any questions regarding this analysis, feel free to call me at 372-0700 or James Bramson at 372-0624.

4SR 5626

project 29953

Tech 2.8

T2.8.3.3

elute composites

Kurath/Blanchard Tc-99 Analysis

December 21, 1999

Results are reported in ng/g (ppb) of original sample.

Sample ID	Client Number	ICP/MS Number	Tc-99 ng/g $\pm$ 1SD	Ru-101/ Ru-102	$\dagger$ Ru-101 ng/g
1%HNO3		9c17a7	0.035 $\pm$ 0.005		
1%HNO3		9c17a1	<0.024		
1%HNO3		9c17a18	<0.024		
00-00615DB	Dilution Blank	9c17a8	<1.0	1.0303	0.6
00-00615	N7-Tc-Elu-Comp	9c17a15	3600 $\pm$ 170	0.7692	4
00-00615DUP	N7-Tc-Elu-Comp	9c17a16	3510 $\pm$ 160	3.2727	8
00-00616	A1-Cs-E2-Comp	9c17a11	27 $\pm$ 6	0.0242	0.8
00-00617	A1R-Cs-E-Comp	9c17a12	15 $\pm$ 4	0.9706	1
00-00618	C1-Cs-E1-Comp	9c17a13	22 $\pm$ 4	0.7927	6
00-00619	C1-Cs-E2-Comp	9c17a14	15 $\pm$ 4	1.2948	16
00-00619 + spike	C1-Cs-E2-Comp	9c17a17	547 $\pm$ 21		
Spike Recovery			103%		

CCV results are reported in ng/ml (ppb)

5ppb Tc-99	9c17a5	4.72 $\pm$ 0.15
5ppb Tc-99	9c17a19	5.13 $\pm$ 0.03

$\dagger$ Based on response from indium

DATA REVIEW

Reviewed by: *O.J. Farnsworth*

Date: 30 Dec 99 Page: 1 of 1

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

Client: D Blanchard

Charge Code/Project: W48409/29953-75%
W48414/29953-25%

ACL Numbers: 00-0615 to 00-0619, 00-0626

ASR Number: 5626 and 5628

Analyst: MJ Steele

Analysis Date: December 18-20, 1999

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"

M&TE: IC system (WD25214); Balance (360-06-01-031) --- See Chemical Measurement Center 98620 RIDS IC File for Calibration, Standards Preparations, and Maintenance Records.

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Final Results:

Lab ID	Sample ID	(F) ug/ml	CL ug/ml	NO2 ug/ml	Br ug/ml	NO3 ug/ml	PO4 ug/ml	SO4 ug/ml	C2O4 ug/ml
00-00615	N7-Tc-Elu-Comp	< 0.3	15	8	< 0.3	19	< 0.5	1	< 0.5
00-00615 MS	N7-Tc-Elu-Comp MS Rec	94%	OvrRng	101%	103%	103%	103%	103%	101%
00-00616	A1-Cs-E2-Comp	< 100	< 100	< 200	< 100	31,400	< 200	< 200	< 200
00-00616	A1-Cs-E2-Comp Dup	< 100	< 100	< 200	< 100	31,600	< 200	< 200	< 200
	RSD	n/a	n/a	n/a	n/a	1%	n/a	n/a	n/a
00-00617	A1R-Cs-E-Comp	< 100	< 100	< 200	< 100	26,500	< 200	< 200	< 200
00-00618	C1-Cs-E1-Composite	< 100	< 100	< 200	< 100	24,500	< 200	< 200	< 200
00-00619	C1-Cs-E2-Composite	< 100	< 100	< 200	< 100	26,300	< 200	< 200	< 200
00-00626	N7-Tc-0	3,200	480	27,900	< 250	110,000	1,100	3,800	1,400
00-00626 MS	N7-Tc-0 MS Rec	112%	105%	122%	108%	OvrRng	111%	114%	111%

RPD = Relative Percent Difference (between sample and duplicate/replicate)

MS Rec = Matrix Spike Standard % recovery

(1) Fluoride is most likely formate or acetate due to slight peak shift and broadening

The samples were analyzed by ion chromatography (IC) for inorganic anions as specified in the governing ASR. The liquid samples were diluted at the IC workstation up to 4,000-fold to ensure that all anions were within the calibration range.

Q.C. Comments:

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, the sample, A1-Cs-E2-Comp (00-0616), was split at the IC workstation and analyzed as two samples (replicate). For anions above the estimated quantitation level (i.e., the lowest calibration standard), the Relative Percent Difference (RPD) is will within the acceptance criteria of 20%.

Matrix Spike: Two matrix spikes were prepared from samples submitted. The recoveries for all anions were within the 75% to 125% recovery acceptance criteria for those anions measured within the calibration range.

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

Blank Spike: No blank spikes were analyzed within the analytical runs.

System Blank/Processing Blanks: Twelve system blanks were process during the analysis of the samples. No anions were detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards: Seven mid-range verification standards were analyzed throughout the analysis runs. Except for only two oxalate values, the reported results for all anions of interest were recovered within the acceptance criteria of $\pm 10\%$ for the verification standard.

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 $\pm 15\%$ or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

Analyst:

Date

Approval:

Date

Archive Information:

Files: ASR 5626 5628 Blanchard.doc

ASR 5606 5626 5642.xls

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

12/22/1999

Client: Blanchard/Kurath

Cognizant Scientist:

LR Greenwood

Date:

12/22/99

Concur:

T Trang-b

Date:

12/28/99

Procedure: PNL-ALO-450 Gamma Energy Analysis

Measured Activities (uCi/g) with 1- σ error

ALO ID Client ID	Tc-95m* Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
00-0615 N7-Tc-Elu-Comp	4.44E-1 2%	<2.E-4	<3.E-4	<9.E-5	<7.E-4	<7.E-4
00-0615 Dup N7-Tc-Elu-Comp	4.55E-1 2%	<2.E-4	<3.E-4	<1.E-4	<7.E-4	<7.E-4
00-0616 A1-Cs-E2-Comp	<6.E-2	3.99E-2 7%	2.38E+2 2%	<7.E-3	<2.E-1	<2.E-1
00-0617 A1R-Cs-E-Comp	<5.E-2	2.39E-2 8%	1.45E+2 2%	<6.E-3	<9.E-2	<9.E-2
00-0618 C1-Cs-E1-Composite	<3.E-1	<2.E-2	4.67E+2 2%	<4.E-2	<6.E-1	<6.E-1
00-0619 C1-Cs-E2-Composite	<4.E-1	<3.E-2	7.05E+2 2%	<4.E-2	<7.E-1	<7.E-1

* Tc-95m values are reported as of December 17, 1999 at 8:00 am PST.

proj 29953

Task 2.8

T2.8.3.3

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building

1/18/2000

Client : Blanchard/Kurath

Cognizant Scientist: L R HernandezDate: 1/18/00Concur: T Trang - 6Date: 1/18/2000

Procedure: PNL-ALO-420/421

Measured Activities (uCi/g) with 1- σ error

ALO ID Client ID	Alpha Error +/-	Sr-90 Error +/-
00-0615 N7-Tc-Elu-Comp	5.73E-6 43%	8.70E-4 5%
00-0616 A1-Cs-E2-Comp	<2.E-5	2.40E-1 4%
00-0617 A1R-Cs-E-Comp	1.62E-5 38%	8.43E-2 3%
00-0618 C1-Cs-E1-Composite	1.20E-4 10%	4.10E-2 8%
00-0619 C1-Cs-E2-Composite	9.86E-5 11%	5.60E-2 9%
00-0619 DUP C1-Cs-E2-Composite		1.41E-1 6%
RPD		86%
Matrix Spike	98%	105%
Blank Spike	108%	103%
Blank	<2.E-5	<7.E-5

proj 29953
Task 2.8
T2.8.3.3

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ACL Number(s): 00-0615 to 00-0619
&
00-0626 to 00-0664

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Task 2.8
T2.8.3.3

Client ID: "N7-Tc-Elu-Comp" to "C1-Cs-E2-Composite"
&
"N7-Tc-0" to "N7-Tc-R01"

~~12-053~~
~~12~~. Eluate
 Composite

Total Samples: 13

Jerry Wagner 12-29-99
Reviewed by
MW Zhu 1-4-00
Concur

Page 1

Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ... ICPAES Data Report

Five radioactive liquid samples, N7-Tc-Elu-Comp to C1-Cs-E2-Composite (ACL# 00-0615 to 00-0619), were analyzed by ICPAES after each sample was diluted using dilute nitric acid by the Sample Receiving and Preparation Laboratory (SRPL). Final sample volume was 25ml (weighed). Concentration was adjusted for dilution using the weight of final solution divided by sample weight.

Eight radioactive liquid samples, N7-Tc-0 to N7-Tc-R01 (ACL# 00-0626 to 00-0664), were analyzed by ICPAES after samples were prepared by SRPL using PNL-ALO-128 acid digestion procedure. Approximately 3ml of sample (weighed) was digested and diluted to a final volume of approximately 10ml (weighed). Concentration was adjusted for dilution from processing using the weight of final solution divided by sample weight.

Measurement results reported have been corrected for preparation and analytical dilution. All results reported are in $\mu\text{g/ml}$ for liquid samples. Weights have been recorded on bench sheets and included with this report.

Liquid samples contained low to high concentrations of sodium ($<0.1\%$ to about 10%). All other analytes measured were typically much lower in concentration.

Quality control check-standard results met tolerance requirements except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements under MCS-033.

Five fold serial dilution:

(Aqueous samples) All results for analytes of interest were within tolerance limit of $\leq 10\%$ after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

(Aqueous samples) All analytes of interest were recovered within tolerance limit of $\leq 20\%$ relative percent difference (RPD).

Post-Spiked Samples (Group A):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125% .

Post-Spiked Samples (Group B):

(Aqueous samples) All analytes of interest were recovered within tolerance of 75% to 125% .

12/29/99

**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

Blank Spike:

(Aqueous samples)   None required or prepared.

Matrix Spiked Sample:

(Aqueous samples)   None required or prepared.

Quality Control Check Standards (aqueous samples):

Concentration of all analytes was within tolerance limit of $\pm 10\%$ accuracy in the standards: QC_MCVA, QC_MCVB, ICP98.0 and QC_SSTMV except as follows. Tin and palladium was low by as much as 23% in QC_MCVB check standard measurements. Single element standards of tin at 2 $\mu\text{g/ml}$ and palladium 2 $\mu\text{g/ml}$ measured were well within the tolerance limit thus confirming calibration check for these two analytes.

High Calibration Standard Check (aqueous samples):

Verification of the high-end calibration concentration for all analytes is within tolerance of $\pm 5\%$ accuracy.

Process Blank:

(Aqueous samples)   All analytes are within tolerance limit of $\leq \text{EQL}$ or $< 5\%$ of sample concentration.

Laboratory Control Standard (LCS):

(Aqueous samples)   No LCS was prepared for PNL-ALO-128 acid digested samples.

Please note bracketed values listed in the data report are within ten times instrument detection limit and have a potential uncertainty much greater than 15%.

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**Battelle PNNL/325 Bldg/RPG/Inorganic Analysis ...
ICPAES Data Report**

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 $\pm 15\%$ 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 $\mu\text{g/mL}$ (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.

12/29/99

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

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Multiplier=		1.0	5.2	5.2	10.3	10.3
ALO#=		00-0615-DB	00-0615	00-0615-DUP	00-0616	00-0617
Client ID=		Dilution Blank	N7-Tc-Elu-Comp	N7-Tc-Elu-Comp	A1-Cs-E2-Comp	A1R-Cs-E-Comp
Det. Limit	Run Date=	12/16/99	12/16/99	12/16/99	12/16/99	12/16/99
(ug/mL)	(Analyte)	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL
0.025	Ag	--	--	--	--	--
0.060	Al	--	[0.47]	[0.44]	[1.3]	[6.1]
0.250	As	--	--	--	--	--
0.050	B	--	10.5	10.5	7.78	7.42
0.010	Ba	--	[0.11]	[0.096]	[0.23]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[1.9]	--	[3.3]	[2.9]
0.015	Cd	--	--	--	[0.34]	[0.23]
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	6.54	3.25
0.025	Cu	--	--	--	12.7	7.13
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	12.8	8.09
2.000	K	--	--	--	[190]	[210]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	--	105	105	698	1,150
0.100	Nd	--	--	--	--	--
0.030	Ni	[0.044]	[0.18]	--	[2.8]	5.69
0.100	P	--	--	--	--	--
0.100	Pb	--	--	--	12.8	[9.9]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	--	[23]	[22]	[6.5]	[6.3]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	6.20	--
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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

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Multiplier=	10.3	10.4	1.0	15.1	14.6
ALO#=	00-0618	00-0619	00-0626-BLK1	00-0626 @5	00-0641 @5
Client ID=	C1-Cs-E1-Composite	C1-Cs-E2-Composite	Process Blank-1	N7-Tc-0	N7-Tc-PW1
Run Date=	12/16/99	12/16/99	12/16/99	12/16/99	12/16/99
Det. Limit (ug/mL)	Run Date= (Analyte)	Run Date= (Analyte)	Run Date= (Analyte)	Run Date= (Analyte)	Run Date= (Analyte)
0.025	Ag	—	—	—	—
0.060	Al	[3.6]	—	2,070	1,920
0.250	As	—	—	—	—
0.050	B	12.5	—	25.4	33.5
0.010	Ba	—	—	—	—
0.010	Be	—	—	—	—
0.100	Bi	—	—	—	—
0.250	Ca	—	[0.34]	146	141
0.015	Cd	[0.50]	—	24.2	22.8
0.200	Ce	—	—	—	—
0.050	Co	—	—	[1.8]	[1.8]
0.020	Cr	5.25	5.29	37.2	35.3
0.025	Cu	20.3	39.3	11.9	11.3
0.050	Dy	—	—	—	—
0.100	Eu	—	—	—	—
0.025	Fe	7.23	7.63	7.35	7.00
2.000	K	—	—	641	620
0.050	La	—	—	—	—
0.030	Li	—	—	—	—
0.100	Mg	—	—	—	—
0.050	Mn	—	—	[1.1]	[1.0]
0.050	Mo	—	—	14.1	13.5
0.150	Na	920	1,630	99,600	90,500
0.100	Nd	—	—	[2.1]	[2.7]
0.030	Ni	67.5	36.7	181	174
0.100	P	—	—	266	245
0.100	Pb	[7.7]	[10]	52.2	50.7
0.750	Pd	—	—	—	—
0.300	Rh	—	—	—	—
1.100	Ru	—	—	—	—
0.500	Sb	—	—	—	—
0.250	Se	—	—	—	—
0.500	Si	[15]	[39]	[65]	85.7
1.500	Sn	—	—	—	—
0.015	Sr	[0.84]	[0.49]	115	110
1.500	Te	—	—	—	—
1.000	Th	—	—	—	—
0.025	Ti	—	—	—	—
0.500	Tl	—	—	—	—
2.000	U	[87]	[170]	—	—
0.050	V	—	—	—	—
2.000	W	—	—	[66]	[63]
0.050	Y	—	—	[1.0]	[0.98]
0.050	Zn	[0.84]	5.40	[5.7]	[5.8]
0.050	Zr	—	—	[2.1]	[1.1]

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).

Det. Limit (ug/mL)	Multiplier= ALO#= Client ID= Run Date= (Analyte)	14.7 00-0642 @5 N7-Tc-PW3 12/16/99 ug/mL	16.4 00-0643 @5 N7-Tc-PW5 12/16/99 ug/mL	3.4 00-0644 N7-Tc-PW7 12/16/99 ug/mL	3.4 00-0645 N7-Tc-PR1 12/16/99 ug/mL	3.8 00-0646 N7-Tc-PR2 12/16/99 ug/mL
0.025	Ag	--	--	--	--	--
0.060	Al	1,870	398	21.1	13.5	8.37
0.250	As	--	--	--	--	--
0.050	B	34.1	26.8	19.3	18.7	22.4
0.010	Ba	--	--	[0.041]	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	134	[31]	[3.0]	[2.2]	[1.4]
0.015	Cd	22.1	4.72	[0.24]	[0.14]	--
0.200	Ce	--	--	--	--	--
0.050	Co	[1.8]	--	--	--	--
0.020	Cr	34.5	7.59	[0.39]	1.17	[0.69]
0.025	Cu	10.9	[2.3]	[0.16]	[0.14]	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	6.81	[1.6]	[0.44]	[0.25]	[0.81]
2.000	K	613	[100]	[14]	[9.9]	[7.6]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	[0.95]	--	--	[0.24]	--
0.050	Mo	13.2	[2.7]	--	--	--
0.150	Na	91,500	31,100	3,420	2,920	2,740
0.100	Nd	[2.7]	--	--	--	--
0.030	Ni	168	38.2	2.22	1.63	[0.66]
0.100	P	245	73.5	5.49	4.63	[1.8]
0.100	Pb	49.1	[11]	[0.71]	--	--
0.750	Pd	--	--	[2.9]	[2.9]	[3.0]
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	90.1	142	107	123	155
1.500	Sn	--	--	--	--	--
0.015	Sr	106	24.0	1.31	[0.34]	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	[61]	--	--	--	--
0.050	Y	[0.93]	--	--	--	--
0.050	Zn	[5.2]	[1.6]	[0.45]	[0.43]	[0.35]
0.050	Zr	[2.1]	--	--	--	--

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).

Battelle PNNL/RPG/Inorganic Analysis ... ICPAES Data Report

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Multiplier=		16.9					
ALO#=		00-0664 @5					
Client ID=		N7-Tc-R01					
Run Date=		12/16/99					
Det. Limit (ug/mL)	(Analyte)	ug/mL					
0.025	Ag	--	--	--	--	--	--
0.060	Al	11.2	--	--	--	--	--
0.250	As	--	--	--	--	--	--
0.050	B	34.7	--	--	--	--	--
0.010	Ba	--	--	--	--	--	--
0.010	Be	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--
0.250	Ca	--	--	--	--	--	--
0.015	Cd	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--
0.050	Co	--	--	--	--	--	--
0.020	Cr	--	--	--	--	--	--
0.025	Cu	--	--	--	--	--	--
0.050	Dy	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--
0.025	Fe	[0.44]	--	--	--	--	--
2.000	K	--	--	--	--	--	--
0.050	La	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--
0.050	Mn	--	--	--	--	--	--
0.050	Mo	--	--	--	--	--	--
0.150	Na	19,200	--	--	--	--	--
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	310	--	--	--	--	--
1.500	Sn	--	--	--	--	--	--
0.015	Sr	[0.38]	--	--	--	--	--
1.500	Te	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--
2.000	U	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--
2.000	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).

project 2-9953, + cell 2-8, T2.83.3

Multiplier=	10.7								
ALO#=	99-2289 @1								
Client ID=	A1-Cs-E1-Composite								
Det. Limit	Run Date=	8/3/99							
(ug/mL)	(Analyte)	ug/g							
0.015	Ag	--	--	--	--	--	--	--	--
0.060	Al	141	--	--	--	--	--	--	--
0.080	As	--	--	--	--	--	--	--	--
0.050	B	72.6	--	--	--	--	--	--	--
0.010	Ba	[0.42]	--	--	--	--	--	--	--
0.010	Be	--	--	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--	--	--
0.100	Ca	[2.1]	--	--	--	--	--	--	--
0.015	Cd	[0.31]	--	--	--	--	--	--	--
0.100	Ce	--	--	--	--	--	--	--	--
0.025	Co	--	--	--	--	--	--	--	--
0.020	Cr	3.48	--	--	--	--	--	--	--
0.015	Cu	50.6	--	--	--	--	--	--	--
0.050	Dy	--	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--	--
0.025	Fe	12.3	--	--	--	--	--	--	--
2.000	K	382	--	--	--	--	--	--	--
0.025	La	--	--	--	--	--	--	--	--
0.005	Li	--	--	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--	--	--
0.005	Mn	0.67	--	--	--	--	--	--	--
0.030	Mo	--	--	--	--	--	--	--	--
0.100	Na	2,230	--	--	--	--	--	--	--
0.100	Nd	--	--	--	--	--	--	--	--
0.030	Ni	5.89	--	--	--	--	--	--	--
0.100	P	[2.9]	--	--	--	--	--	--	--
0.060	Pb	15.6	--	--	--	--	--	--	--
0.300	Pd	--	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--	--
0.075	Ru	--	--	--	--	--	--	--	--
0.050	Sb	--	--	--	--	--	--	--	--
0.050	Se	--	--	--	--	--	--	--	--
0.100	Si	51.4	--	--	--	--	--	--	--
1.000	Sn	--	--	--	--	--	--	--	--
0.005	Sr	--	--	--	--	--	--	--	--
0.500	Te	--	--	--	--	--	--	--	--
0.800	Th	--	--	--	--	--	--	--	--
0.005	Ti	--	--	--	--	--	--	--	--
0.250	Tl	--	--	--	--	--	--	--	--
2.000	U	[48]	--	--	--	--	--	--	--
0.015	V	--	--	--	--	--	--	--	--
0.500	W	--	--	--	--	--	--	--	--
0.010	Y	--	--	--	--	--	--	--	--
0.020	Zn	12.0	--	--	--	--	--	--	--
0.025	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).

Radiochemistry Bench Sheet

Client Kurath DE
 Work Order # CMS
 ASR 5463

ICP

Balance 360-06-01-026Procedure DilutionPipet ID Numbers T = 21.5 °CPipet Checks P/K 7/30/99

Sample ID Dilutions and Aliquot Sizes

99-2289 1 ml $\rightarrow$ 10 EV = 11 ml

#205722N @ 10 mL = 10.0434 g
 10.0319 g
 10.0066 g
 9.9969 g

(estimated Sample wt. = 1.025 g/mL * 0.99852 g = 1.0260 g sample)
 or 0.9975 g/mL

Joe Wagner 8-9-99

126686 = 1.0008 g
 0.9994 g
 0.9995 g
 0.9983 g
 0.9946 g

47286 = 0.0998 g
 0.0999 g
 0.1001 g
 0.0999 g
 0.1000 g

49170 = 0.0501 g
 0.0496 g
 0.0499 g
 0.0500 g
 0.0499 g

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date

M. Steele

7/30/99

Reviewer/Date

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

Client: D. Blanchard

Charge Code: 75%/25% W48409/W48414

Project: 29953

ACL Numbers: 00-0615 to 00-0619

ASR Number: 5626

Analyst: MJ Steele

Analysis Date: January 7, 2000

Procedure: PNL-ALO-381, "Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method"

M&TE: Carbon System (WA92040); Balance (360-06-01-023).

Final Results:

Lab Number	Sample ID	Vol ml	TOC µg C/ml	TOC RPD %
00-0615	N7-Tc-Elu-Comp	0.3000	<60	
00-0615 Dup	N7-Tc-Elu-Comp Rep	1.0000	<20	n/a
00-0615 Spike	N7-Tc-Elu-Comp MS	1.0000	104%	
00-0616	A1-Cs-E2-Comp	0.3000	80	
00-0616 Dup	A1-Cs-E2-Comp Rep	1.0000	<20	n/a
00-0617	A1R-Cs-E-Comp	1.0000	<20	
00-0617 Dup	A1R-Cs-E-Comp Rep	1.0000	<20	n/a
00-0618	C1-Cs-E1-Comp	1.0000	150	
00-0618 Dup	C1-Cs-E1-Comp Rep	1.0000	150	0
00-0619	C1-Cs-E2-Comp	1.0000	200	
00-0619 Dup	C1-Cs-E2-Comp Rep	1.0000	190	5

RPD = Relative Percent Difference (between sample and duplicate/replicate)

n/a = Not applicable; either sample or duplicate result is <5 times MDL

proj 29953

Task 2.8

T2.8.3.3

P1/2

The analysis of the subject samples submitted under ASR 5626 were performed by the hot persulfate wet oxidation method. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95°C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. Per the ASR and since the samples were acidic, only TIC analyses were performed on these samples.

The table above shows the results, rounded to one to two significant figures. The result for A1-Cs-E2-Comp (i.e., 80 µg/ml) is only marginally above the method detection limit for the 0.3 ml sample size analyzed (i.e., 60 µg/ml) and should be considered qualitative. 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

Q.C. Comments:

The TIC standard is calcium carbonate and TOC standard is α-Glucose (the certificates of purity are attached). The standard materials were used in solid form for system calibration standards as well as matrix spikes. TIC and TOC percent recovery are determined using the appropriate standard (i.e., calcium carbonate for TIC or glucose for TOC).

Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per matrix type.

Calibration Standards: The QC system calibration standards were all within acceptance criteria, with the average recovery being 98.4% for TIC and 98.1% for TOC.

Calibration Blanks: The three calibration blanks run at the beginning and middle of the analysis run were acceptable, averaging 13.9 µgC TIC and 454.7 µgC TOC.

Duplicates: No actual sample duplicates were provided to the laboratory for analysis. However, each sample was analyzed in replicate and the relative percent differences (RPD) between replicates are within the acceptance criteria of 20% for all values measured above the EQL (i.e., 5 times the method detection limit).

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. A matrix spike was prepared from sample 00-0615 (N7-Tc-Elu-Comp) with the TOC spike recovery being 103.8%, well within the 75% to 125% recovery acceptance criteria.

General Comments:

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically $\pm 15\%$ or better for non-complex samples that are free of interferences.
- The estimated quantitation limit (EQL) is defined as 5 times the MDL. Results less than 5 times the MDL have higher uncertainties, and RPDs are not calculated for any results less than 5 times the MDL.
- Some results may be reported as less than (" $<$ ") values. These less than values represent the sample MDL (method detection limit), which is the system MDL adjusted for the volume of sample used for the analysis. The system MDL is based on the attached pooled historical blank data. The evaluation and calculation of the system MDL is included in the data package.

Report Prepared by: MW Thru

Date 1-11-00

Review/Approval by: D. B. Blum

Date 1-17-00

Archive Information:

Files: ASR 5626 Blanchard.doc

ASR 5478 5626 Liq+Solids.xls

Multiplier=		10.7						
ALO#=		99-2289 @1						
Client ID=		A1-Cs-E1-Composite						
Det. Limit	Run Date=	8/3/99						
(ug/mL)	(Analyte)	ug/g						
0.015	Ag	--	--	--	--	--	--	--
0.060	Al	141	--	--	--	--	--	--
0.080	As	--	--	--	--	--	--	--
0.050	B	72.6	--	--	--	--	--	--
0.010	Ba	[0.42]	--	--	--	--	--	--
0.010	Be	--	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--	--
0.100	Ca	[2.1]	--	--	--	--	--	--
0.015	Cd	[0.31]	--	--	--	--	--	--
0.100	Ce	--	--	--	--	--	--	--
0.025	Co	--	--	--	--	--	--	--
0.020	Cr	3.48	--	--	--	--	--	--
0.015	Cu	50.6	--	--	--	--	--	--
0.050	Dy	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--
0.025	Fe	12.3	--	--	--	--	--	--
2.000	K	382	--	--	--	--	--	--
0.025	La	--	--	--	--	--	--	--
0.005	Li	--	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--	--
0.005	Mn	0.67	--	--	--	--	--	--
0.030	Mo	--	--	--	--	--	--	--
0.100	Na	2,230	--	--	--	--	--	--
0.100	Nd	--	--	--	--	--	--	--
0.030	Ni	5.89	--	--	--	--	--	--
0.100	P	[2.9]	--	--	--	--	--	--
0.060	Pb	15.6	--	--	--	--	--	--
0.300	Pd	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--
0.075	Ru	--	--	--	--	--	--	--
0.050	Sb	--	--	--	--	--	--	--
0.050	Se	--	--	--	--	--	--	--
0.100	Si	51.4	--	--	--	--	--	--
1.000	Sn	--	--	--	--	--	--	--
0.005	Sr	--	--	--	--	--	--	--
0.500	Te	--	--	--	--	--	--	--
0.800	Th	--	--	--	--	--	--	--
0.005	Ti	--	--	--	--	--	--	--
0.250	Tl	--	--	--	--	--	--	--
2.000	U	[48]	--	--	--	--	--	--
0.015	V	--	--	--	--	--	--	--
0.500	W	--	--	--	--	--	--	--
0.010	Y	--	--	--	--	--	--	--
0.020	Zn	12.0	--	--	--	--	--	--
0.025	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).

Radiochemistry Bench Sheet

Client Kurath DE
Work Order # CMS
ASR 5463

Procedure

Dilution

ICP

Balance 360-06-01-026

Pipet ID Numbers

T = 21.5 °C

Pipet Checks PKD 7/30/99

Sample ID Dilutions and Aliquot Sizes

99-2289 1 ml $\rightarrow$ 10 EV = 11 ml

#105722N @ 10 mL = 10.0434 g

10.0319 g

10.0066 g

9.9969 g

10.0164 g

(Estimated Sample wt. = 1.025 g/mL * 0.99852 g = 1.0260 g sample)
0.9975 g/mL

J. Wayne 8-9-99

#126086 = 1.0008 g

0.9994 g

0.9995 g

0.9983 g

0.9946 g

#47286 = 0.0998 g

0.0999 g

0.1001 g

0.0999 g

0.1000 g

#49170 = 0.0501 g

0.0496 g

0.0499 g

0.0500 g

0.0499 g

Spike, Tracer ID

Isotope

Concentration

Ref Date

Analyst/Date

M. Steele

7/30/99

Reviewer/Date

Battelle Pacific Northwest Laboratory
Radiochemical Processing Group-325 Building
Radioanalytical Applications Team

99-2289
8/12/1999

Client : DE Kurath

Cognizant Scientist:

R. Greenwood

Date :

8/12/99

Concur :

J.K. Hickum

Date :

8/12/99

Total Alpha (PNL-ALO-4001)
Sr-90 (PNL-ALO-476, 431)
Gamma (PNL-ALO-450)

Measured Activities ($\mu\text{Ci/ml}$)

ALO ID Client ID	Alpha Error %	Sr-90 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
99-2289 A1-CS-E1-Composite	<5.E-2	<5.E-2	3.36E-1 10%	1.61E+3 2%	<6.E-2	<1.E+0	<1.E+0
99-2289 DUP A1-CS-E1-Composite	<4.E-2						
Matrix Spike	109%	88%					
Reagent Spike	101%	102%					
Blank	<4.E-2	<3.E-1					

Note: Due to the high level of Cs-137, it was not possible to achieve the requested detection limits for Eu-154, Eu-155, and Am-241.

Date September 1, 1999

File/LB

To D. Kurath

 From M. Urie *M. Urie*

 Subject Carbon analysis of Cx-IX Sample

The analysis of the subject sample submitted under ASR 5463 was performed by the hot persulfate wet oxidation method, PNL-ALO-381, rev. 1. The hot persulfate method uses acid decomposition for TIC and acidic potassium persulfate oxidation at 92-95 °C for TOC, all on the same sample, with TC being the sum of the TIC and TOC. However, since the sample was highly acidic only the TOC portion of the analysis was performed to obtain total carbon (as TOC).

The samples were analyzed on August 27, 1999 and Table 1 below shows the results, rounded to two 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.

All samples were analyzed directly (i.e., no preparative or analytical dilution), and are reported in microgram of carbon per milliliter of original sample. The masses of the sample aliquots used for analysis are provided.

Table 1: TC Results (as TOC)

ALO Number	Sample ID	Sample Quantity		TOC/TC μg/mL	TOC/TC RPD (%)
		mL	g		
99-02289	Cs-IX	0.300	0.296	130	
99-02289 Replicate	CS-IX Rep	0.300	0.304	110	n/a

RPD = Relative Percent Difference

n/a = not calculated; sample and/or replicate result less than five times MDL

QC Narrative

The TOC standard is α-Glucose (the certificates of purity are attached). The standard material was used in solid form for system calibration standards as well as matrix spikes.

The QC for the methods involves calibration blanks, system calibration standards, sample duplicates, and one matrix spike per batch of samples analyzed. The QC system calibration standards were all within acceptance criteria, with the average recovery being 97.2% for TOC. The calibration blanks were acceptable, averaging 68 μgC for TOC. The standard deviation of the calibration blanks was significantly higher than normal, and has moderate impact on the reported results since the reported results are less than five times the method detection limit.

The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. No spike was performed on the Cs-IX sample; however, the TOC spike recovery for another acidic sample in the same analytical batch had a recovery of 116%. This is within the acceptance criteria of 75% to 125%. The precision (as Relative Percent Difference [RPD]) could not be determined since the carbon concentration in the sample is less than 5 times the method detection limit.

Review/Approve:

Marilyn J. Steele 9-1-99

Archive Information:

Files: ASR 5463 Kurath.doc, ASR 5463 Kurath -- Liquids.xls

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Page 1 of 1

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

WO/Project: W48412/29953
Client: D. Kurath

ACL Numbers: 99-02276
ASR Number 5461

Results:

Lab ID	Sample ID	F ug/ml	Cl ug/ml	NO ₂ ug/ml	Br ug/ml	NO ₃ ug/ml	PO ₄ ug/ml	SO ₄ ug/ml	C ₂ O ₄ ug/ml
99-02276	A1-Tc-0	1,020	2,670	46,500	< 200	92,800	< 400	690	< 400
99-02276 MS	A1-Tc-0 Spike	3.6	3.7	25.0	3.3	37.1	13.6	13.4	13.5
	Spike Recovery	111%	102%	112%	110%	116%	113%	111%	112%

Comments:

All sample and analytical QC was within acceptance criteria; however, no duplicate was provided to assess precision. The sample was analyzed at various analytical dilutions providing RPDs for F, Cl, and NO₂ of 47%, 9%, and 3%, respectively. The NO₃ was measured at only one dilution that provided results within the calibration range. Significant coeluting interferences were prominent at the F and Cl retention times; most likely from organic anions. These interferences make the quantitation of F and Cl difficult, and the reported values should be considered upper bounds for the F and Cl concentrations.

Procedure: PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography"

Analyst: MJ Steele

Analysis Date: August 5, 1999

M&TE: IC system (WD25214); Mettler AT400 Balance (360-06-01-031) See Chemical Measurement Center 98620 RIDS for IC File for Calibration, Standards Preparations, and Maintenance Records.

Analyst: *MJ Steele* 8/26/99

Approval: *Mike* 8/27/99

Notes:

- 1) "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- 2) 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.
- 3) Routine precision and bias is typically $\pm 15\%$ or better for non-complex aqueous samples that are free of interference and have similar concentrations as the measured anions.

APPENDIX B

Appendix B: Spreadsheet Calculations

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	
1	Loading of SL-644 with AW-101 feed																		
2	Loading																		
3			Start Date and Time:		6/8/99	15:59													
4			Bed volume col 1 =		15.0	mL													
5			Bed volume col 2 =		15.0	mL													
6			Empty Eff Btl - 1 w/ cap(w / holes) + plastic wra				386.7	g											
7			Empty Eff Btl -2 w/ cap(w / holes) + plastic wra				260.3	g											
8			Bed volumes diverted to waste =				3.75												
9			Supernate Density =				1.228	(g/mL)											
10			Co (ave) =		716384	cnts/g of sample =		238795	cnts/g of waste/minute =		293239.7	cnts/mL of waste/minute							
11			regeneration and diluent (0.25 M NaOH) density @ 25 °C =				1.008	g/mL											
12			density of 0.1 M NaOH @ 25 °C =																
13			eluant density (0.5 M nitric acid) @ 25 °C =				1.014	g/mL											
14			Feed analyses: Cs-137, uCi/mL																
15			Date	uCi/mL															
16			Mar-99	179.3	Batch contacts with AW-101 feed and SL-632; sample # RD1AW-C														
17			May-99	174.7	Batch contacts with AW-101 feed and SL-644; sample # AW-101-C														
18			May-99	176.0	Batch contacts with AW-101 feed and SL-644; sample # AW-101-S1														
19			May-99	177.7	Batch contacts with AW-101 feed and SL-644; sample # AW-101-S2														
20			Jun-99	179.6	Column run feed analysis														
21			average	177.5															
22																			
23			The effluent volume on which the bed volumes is based was determined by estimating the total mass and volume of waste pumped through the columns at the time of sampling. For column #1 this includes the mass of the waste in the effluent bottle + the samples taken from column # 1 and # 2 prior to the sample of interest (the mass of the effluent bottle was recorded during each sample event). For column # 2 the effluent volume includes the mass of the waste in the effluent bottle + the samples taken from column #2 prior to the the sample of interest (samples taken from column # 1 did not go through column # 2. A hold up volume of 2 bed volumes (30 mL) is added for column # 1. This is the system volume between the end of the effluent line and valve 3. This is actually added in the bed volume column in the spreadsheet. The holdup between the bottom of column # 2 and the effluent bottle was not accounted for. The 80.77 g sub sample was added to the effluent mass starting at sample A1-Cs-L5 and A1-Cs-P3. Since the run started with 0.25 M caustic in the system, approximately 3.75 bed volumes of solution were diverted to the waste jug. Effluent collection therefore started about the time the waste began exiting the system.																
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25																			
26																			
27																			
28																			
29																			
30																			
31																			
32																			
33																			

	A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ
1	Loading of SL-644																		
2	Loading																		
3																			
4																			
5																			
6																			
7																			
8																			
9																			
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	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R
		ACL number	sample time (hr:mm:ss)	elapsed time (hr)	sample mass taken g	mass of diluent g	volume of diluent mL	mass of sample pipetted for dilution g	mass of sample + diluent g	mass of sample + diluent transferred (4) g	sample mass counted (4) g	sample volume counted mL	count time, min	Tot Cts	back ground	Tot cts-bck	counts/g of sample	¹³⁷ Cs analytical value uCi/g
34	Vial																	
35																		
36	A1-Cs-0 (cnt 1)				0.5685	9.6080	9.5317	0.5685	10.1765	9.3740	0.5237	0.4264	3	382375	4649	377726	721307	
37	A1-Cs-0 (cnt 2)				0.5685	9.6080	9.5317	0.5685	10.1765	9.3740	0.5237	0.4264	3	372589	19	372570	711461	
38	A1-Cs-0	99-2019	15:59:00	0.000	0.5685	9.6080	9.5317	0.5685	10.1765	9.3740	0.5237	0.4264	3				716384	8.17
39	A1-Cs-L1 ⁽¹⁾	99-2020	17:41:00	1.700	5.2291	na	na	na	na	na	4.6635	3.7976	3	4739	4649	90	19	1.25E-02
40	A1-Cs-L2 ⁽¹⁾	99-2021	19:21:00	3.367	4.3819	na	na	na	na	na	3.7768	3.0756	3	6360	4649	1711	453	-
41	A1-Cs-L3 ⁽¹⁾	99-2022	21:00:00	5.017	3.3300	na	na	na	na	na	2.8705	2.3375	3	10769	4649	6120	2132	-
42	A1-Cs-L4 ⁽¹⁾	99-2023	22:40:00	6.683	4.7865	na	na	na	na	na	4.6507	3.7872	3	40492	4649	35843	7707	-
43	A1-Cs-L5 ⁽¹⁾	99-2024	0:20:00	8.350	4.5398	na	na	na	na	na	4.3417	3.5356	3	98935	4649	94286	21716	4.68
44	A1-Cs-L6 ⁽¹⁾	99-2025	3:20:00	10.270	4.0571	na	na	na	na	na	3.8629	3.1457	3	183112	4649	178463	46199	-
45	A1-Cs-L7 ⁽¹⁾	99-2026	5:00:00	11.937	5.2023	na	na	na	na	na	5.0288	4.0951	3	364967	4649	360318	71651	15.90
46	A1-Cs-L8 ⁽¹⁾⁽³⁾	99-2027	6:40:00	13.603	4.0347	na	na	na	na	na	3.8652	3.1476	3	370325	4649	365676	94607	-
47	A1-Cs-L9 ⁽¹⁾	99-2028	8:20:00	15.270	4.2058	na	na	na	na	na	3.8643	3.1468	3	497614	4649	492965	127569	-
48	A1-Cs-L10 ⁽¹⁾	99-2029	10:00:00	16.937	4.3877	na	na	na	na	na	3.9165	3.1893	3	-	-	-	-	35.60
49	A1-Cs-L11 ⁽¹⁾	99-2030	11:40:00	18.603	4.4231	na	na	na	na	na	4.0788	3.3215	3	838406	4649	833757	204412	-
50	A1-Cs-L12	99-2031	13:20:00	20.270	3.8758	4.6604	4.6234	0.6049	5.2653	4.9420	0.5678	0.4623	3	151016	5732	145284	255891	-
51	A1-Cs-L13 ⁽²⁾	99-2032	15:00:00	21.937	3.6942	4.6772	4.6401	0.6049	5.2821	5.0516	0.5785	0.4711	3	98546	9763	98546	170346	3.99
52	A1-Cs-L14	99-2033	16:40:00	23.603	5.0048	4.6908	4.6536	0.6000	5.2908	4.9755	0.5642	0.4595	3	199728	5732	193996	343816	-
53	A1-Cs-L15	99-2034	18:20:00	25.270	3.6147	4.6822	4.6450	0.6021	5.2843	5.0183	0.5718	0.4656	3	228749	5732	223017	390032	-
54	A1-Cs-L16	99-2035	20:00:00	26.937	4.0148	4.7007	4.6634	0.5823	5.2830	4.9983	0.5509	0.4486	3	254327	5732	248595	451236	-
55	A1-Cs-L17	99-2036	20:43:00	27.653	3.0418	4.6684	4.6313	0.5689	5.2373	4.9493	0.5376	0.4378	3	260281	253	260028	483669	10.70
56			(1) Initial samples were removed from hot cell without dilution. Since the Cs-137 concentration was relatively low, the dose was also low.								(4) samples A1-Cs-0 and A1-Cs-P2A have extra values in formula to account for new caps; the original caps were dropped in the hot cell.							
57			(2) vial L13 had P7 lid and vial P7 had L13 lid, assumed that vial label was correct, mass of sample transferred may be incorrect since caps have slightly different masses.															
58			(3) Portable counter not used, vial contaminated.															
59																		
60																		
61	Vial	ACL number	sample time (hr:mm:ss)	elapsed time (hr)	sample mass taken g	mass of diluent g	volume of diluent mL	mass of sample pipetted for dilution g	mass of sample + diluent g	mass of sample + diluent transferred (5) g	sample mass counted g	sample volume counted mL	count time, min	Tot Cts	back ground	Tot cts-bck	counts/g of sample counted	¹³⁷ Cs analytical value uCi/g
62																		
63	A1-Cs-0	99-2019	15:59:00	0.000	0.5685	9.6080	9.5317	0.5685	10.1765	9.3740	0.5237	0.4264					716384	8.17
64	A1-Cs-P1	99-2037	17:45:00	1.767	4.3369	na	na	na	na	na	3.9024	3.1779	3	156	56	100	23	3.07E-03
65	A1-Cs-P2	99-2038	21:03:00	5.067	3.9472	na	na	na	na	na	2.9995	2.4426	3	1985	56	1929	489	-
66	A1-Cs-P3	99-2039	0:26:00	8.450	4.4894	na	na	na	na	na	4.2999	3.5015	3	37587	4649	32938	7660	1.63E+00
67	A1-Cs-P4	99-2040	5:05:00	12.020	3.9565	na	na	na	na	na	3.7704	3.0704	3	125433	4649	120784	32035	-
68	A1-Cs-P5 ⁽⁵⁾	99-2041	8:24:00	15.337	4.0298	na	na	na	na	na	3.5785	2.9141	3	-	-	-	-	1.38E+01
69	A1-Cs-P6	99-2042	11:44:00	18.670	4.3199	na	na	na	na	na	3.9237	3.1952	3	417304	4649	412655	105170	-
70	A1-Cs-P7 ⁽²⁾	99-2043	15:03	21.987	3.5656	4.6737	4.6366	0.562	5.2357	5.2885	0.562	0.4577	3	95810	9763	86047	153109	3.77E+00
71	A1-Cs-P8	99-2044	18:24:00	25.337	3.6544	4.6436	4.6067	0.5626	5.2062	5.1709	0.5588	0.4550	3	137001	5732	131269	234918	-
72	A1-Cs-P9	99-2045	20:00:00	26.937	3.4425	4.6688	4.6317	0.6019	5.2707	5.0023	0.5712	0.4652	3	169554	253	169301	296370	7.15E+00
73											(5) samples A1-Cs-0 and A1-Cs-P2A have extra values in formula to account for new caps; the original caps were dropped in the hot cell.							
74																		
75																		
76																		

	A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ
34	Vial	mass of sample counted	mass of waste counted	¹³⁷ Cs in sample	¹³⁷ Cs per g of waste	¹³⁷ Cs per mL of waste	C/Co, portable GEA	C/Co, analytical (5)	analytical/ portable	Eff Btl Mas (g)	Effluent Mass+ samples (6)	Eff Vol (6)	Bed volumes (6)	Flow Rate BV/hr				Cs-137 in samples	
35		g	g	uCi	uCi/g	uCi/mL					(g)	(g)	(mL)					uCi	
36	A1-Cs-0 (cnt 1)																		
37	A1-Cs-0 (cnt 2)																		
38	A1-Cs-0	2.0396	0.1139	83.14	146	179.6	-	-	-	386.7	0	0.0	0.0						
39	A1-Cs-L1 ⁽¹⁾			0.07	0.0125	1.54E-02	0.003%	0.0086%	3.21	492.3	115.2	93.8	8.2	4.84				0.020	
40	A1-Cs-L2 ⁽¹⁾	-	-	0.40	-	-	0.063%	-	-	664.4	291.6	237.5	17.8	5.73				0.400	
41	A1-Cs-L3 ⁽¹⁾	-	-	1.4	-	-	0.298%	-	-	848.2	478.8	389.9	27.9	6.14				1.43	
42	A1-Cs-L4 ⁽¹⁾	-	-	7.4	-	-	1.08%	-	-	1035.1	674.4	549.2	38.5	6.35				7.4	
43	A1-Cs-L5 ⁽¹⁾			21	4.7	5.75E+00	3.03%	3.24%	1.07	1155.9	880.5	717.0	49.7	6.69				19.9	
44	A1-Cs-L6 ⁽¹⁾	-	-	38	-	-	6.45%			1372.5	1105.7	900.4	61.8	6.35				37.8	
45	A1-Cs-L7 ⁽¹⁾			83	15.9	1.95E+01	10.00%	11.00%	1.10	1564.2	1302.6	1060.7	72.5	6.39				75.2	
46	A1-Cs-L8 ⁽¹⁾⁽⁹⁾	-	-	77	-	-	13.21%	-	-	1727.6	1474.0	1200.3	81.8	5.57				77.0	
47	A1-Cs-L9 ⁽¹⁾	-	-	108	-	-	17.81%	-	-	1902	1652.6	1345.7	91.4	5.80				108.2	
48	A1-Cs-L10 ⁽¹⁾	-	-	156	35.6	4.37E+01	-	24.63%	-	2070.9	1829.9	1490.1	101.0	5.76				156.2	
49	A1-Cs-L11 ⁽¹⁾	-	-	182	-	-	28.53%	-	-	2248	2011.4	1638.0	110.9	5.90				182.4	
50	A1-Cs-L12	-	-	200	-	-	35.72%	-	-	2427.6	2199.2	1790.9	121.0	6.10				200.1	
51	A1-Cs-L13 ⁽²⁾	2.0756	0.2377	129	34.8	42.8	23.78%	24.11%	1.01	288.6	2380.2	1938.3	130.8	5.88				126.9	
52	A1-Cs-L14	-	-	347	-	-	47.99%	-	-	465.1	2565.3	2089.0	140.8	6.01				347.1	
53	A1-Cs-L15	-	-	284	-	-	54.44%	-	-	645.9	2749.7	2239.2	150.8	5.99				284.4	
54	A1-Cs-L16	-	-	365	-	-	62.99%	-	-	821.1	2932.6	2388.1	160.7	5.94				365.5	
55	A1-Cs-L17	2.0649	0.2243	300	98.5	121.0	67.52%	68.16%	1.01	896.8	3014.7	2455.0	165.2	6.21				296.8	
56													ave=	5.97				total Cs-137 in samples	2287
57																			
58																			
59																			
60																			
61	Vial	mass of sample counted	mass of waste counted	¹³⁷ Cs in samples	¹³⁷ Cs per g of waste	¹³⁷ Cs per mL of waste	C/Co	C/Co, analytical	analytical/ portable	Eff Btl Mass	Effluent Mass+ sample (7)	Eff Vol (7)	Bed volumes (7)					Cs-137 in samples	
62		g	g	uCi	uCi/g	uCi/mL				(g)	(g)	(mL)	BV					uCi	
63	A1-Cs-0	2.0396	0.1139		146.2480	179.6	-	-	-	386.7	-	-	-						
64	A1-Cs-P1	-	-	1.33E-02	3.07E-03	3.8E-03	0.0032%	0.0021%	0.66	492.3	109.9	89.5	6.0					0.02	
65	A1-Cs-P2	-	-	3.89E-01	-	-	0.0682%	-	-	848.2	469.8	382.6	25.4					0.39	
66	A1-Cs-P3	-	-	7.32E+00	1.63E+00	2.0E+00	1.07%	1.13%	1.05	1155.9	862.7	702.6	46.7					6.9	
67	A1-Cs-P4	-	-	2.56E+01	-	-	4.47%	-	-	1564.6	1275.4	1038.6	69.0					25.6	
68	A1-Cs-P5 ⁽¹⁾	-	-	5.56E+01	1.38E+01	1.7E+01	-	9.55%	-	1902	1616.8	1316.6	87.5					55.6	
69	A1-Cs-P6	-	-	9.17E+01	-	-	14.68%	-	-	2247.7	1966.8	1601.7	106.5					91.7	
70	A1-Cs-P7 ⁽²⁾	2.0728	0.2203	1.26E+02	35.4762	43.6	21.37%	24.55%	1.15	288.6	2327.6	1895.5	126.0					110.1	
71	A1-Cs-P8	-	-	1.73E+02	-	-	32.79%	-	-	645.9	2688.6	2189.4	145.5					173.2	
72	A1-Cs-P9	2.0632	0.2356	2.16E+02	62.6109	76.9	41.37%	43.32%	1.05	896.8	2942.9	2396.5	159.3					205.8	
73																		total Cs-137 in sample	669.3
74																			
75																			
76																			

(5) The analytical value from cell U36 is used for Co.

(6) Bed volumes are the volume of feed that has passed through the column(s). For column #1, this requires that the mass of samples from both columns be added to the mass of effluent in the effluent bottle. The volume is determined by dividing by the density. Two (2) bed volumes are added to account for the 30 mL holdup between the bottom of column 1 and the effluent bottle.

(7) Bed volumes are the volume of feed that has passed through the column(s). For column #2, this requires that the mass of sample from column #2 be added to the mass of effluent in the effluent bottle. The volume is determined by dividing by the density. Holdup between the bottom of column 2 and the effluent bottle is small and not accounted for.

80.771 g taken in separate poly bottle starting at 11:30. Starting with sample A1-Cs-L5, 80.77 g is added to the effluent bottle mass.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R
	Bottle	ACL number	mass of bottle	final mass of bottle + waste	mass of waste *	volume of waste	mass of vial + cap	mass of vial + cap + sample	mass of sample	mass of analytical vial + cap	mass of analytical vial + cap + sample	mass of sample	count time	Tot Cts	back ground	Tot cts-bck	counts/g of sample counted	C/Co
77			g	g	g	mL	g	g	g	g	g	g	min	cnts	cnts	cnts	cnts/g	
78	Effluent bottle 1	99-2079	386.7	2576.6	2270.7	1849	16.8388	20.8866	4.0478	17.0246	20.7529	3.7283	3	155008	253	154755	41508	5.79%
80	Effluent bottle 2	99-2080	260.3	896.8	636.5	518	17.0686	21.3081	4.2395	16.9795	21.0023	4.0228	3	884541	253	884288	219819	30.68%
81	total				2907.17	2367												
82	* includes 80.77 g from a subsample added back to effluent - 1																	
83																		
84				initial DF column 1 =		11561												
85				initial DF column 2 =		47073												
86				Composite DF =		8.49												
87																		
88																		
89																		
90	Feed Displacement: 0.1 M NaOH																	
91																		
92			Start Date and Time:		6/9/99	21:39												
93			Density of 0.1 M NaO		1.001	g/mL												
94	Vial/Bottle	ACL number	sample start time	sample finish time	Elapsed time	mass of vial + cap	mass of vial + cap + sample	mass of sample collected	sample volume	mass of analytical vial + cap (tare)	mass of analytical vial + cap + sample (tare)	mass of sample counted	count time, min	background	Total counts	total cts - bckg	net cts per g of sample per min	net cts per mL of sample per min
95			hr:min	hr:min	hr	g	g	g	mL	g	g	g	min	cnts	cnts	cnts	cnts/g/min	cnts/mL/min
96	A1-Cs-PW1	99-2046	21:39	22:01	0.367	16.875	37.2728	20.398	16.533	16.198	22.353	6.155	1	57	526504	526447	85534	105528
97	A1-Cs-PW2	99-2047	22:02	22:21	0.700	16.865	34.6309	17.766	14.368	16.369	22.385	6.017	1	57	495412	495355	82333	101800
98	A1-Cs-PW3	99-2048	22:22	22:40	1.017	16.827	34.156	17.329	14.826	16.275	22.108	5.833	1	57	382026	381969	65485	76542
99																		
100	DI water rinse																	
101																		
102	Down from 10:40 to 11:08 to remove air from above column 1 and to switch to DI water rinse																	
103			start date and time		6/9/99	23:08												
104			system shut down and restarted in the morning															
105	Vial/Bottle	ACL number	sample start time (hr:mm:ss)	sample finish time	Elapsed time	cumulative time	mass of vial + cap	mass of vial + cap + sample	mass of sample collected	sample volume	mass of analytical vial + cap (tare)	mass of analytical vial + cap + waste (tare)	mass of waste counted	count time, min	background	Total counts	total cts - bckg	net cts per g of sample per min
106			hr:mm:ss	hr:min	hr	hr	g	g	mL	mL	g	g	g	min	cnts	cnts	cnts	cnts/g
107	A1-Cs-PR1	99-2049	23:08	23:27	0.317	0.317	17.008	34	16.992	15.834	16.7031	31.7156	15.013	3	57	2260081	2260024	50181
108	A1-Cs-PR2	99-2050	23:28	23:48	0.333	0.650	16.910	34	17.090	16.516	16.6897	32.3299	15.640	1	57	424503	424446	27138
109	A1-Cs-PR3	99-2051	6:23	6:47	0.400	1.050	16.8011	34.2579	17.457	17.200	16.7045	32.4953	15.791	1	57	337434	337377	21365
110	A1-Cs-PR4	99-2052	6:49	7:09	0.333	1.383	16.948	32.6103	15.662	15.553	16.7809	31.9417	15.161	1	57	121934	121877	8039
111	A1-Cs-PR5	99-2053	7:12	7:32	0.333	1.717	16.839	32.4708	15.632	15.571	16.7359	26.3271	9.591	1	57	34917	34860	3635
112	A1-Cs-PR6	99-2054	7:35	8:02	0.450	2.167	16.823	37.1737	20.351	20.330	16.5282	36.7529	20.225	1	57	41116	41059	2030
113																		
114																		

A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ
77 Bottle	mass of waste counted	¹³⁷ Cs per g of waste	¹³⁷ Cs per mL of waste	C/Co based on analytical	analytical C/Co			Cs-137 in effluent										
78	g	uCi/g	uCi/mL					uCi										
79 Effluent bottle 1	2.4646	8.9	10.9	6.15%	1.06			20186										
80 Effluent bottle 2	2.4689	45.7	56.1	31.62%	1.03			29088										
81 total								49274										
82																		
83																		
84																		
85																		
86																		
87																		
88																		
89																		
Feed																		
Displacement: 0.1																		
90 M NaOH																		
91																		
92																		
93																		
94 Vial/Bottle	C/Co	mass of waste counted	volume	density	¹³⁷ Cs in samples	¹³⁷ Cs per g of sample	¹³⁷ Cs per mL of sample	C/Co based on analytical	analytical C/Co	Bed Volumes	cumulative e BV's	Flow rate			Cs-137 in wash			
95		g	mL	g/mL	uCi	uCi/g	uCi/mL			BV	BV	BV/hr			uCi			
96 A1-C3-PW1	35.99%	2.4675	2	1.234	1412	69.2	85.4	48.11%	1.34	1.10	1.10	3.0			1412			
97 A1-C3-PW2	34.72%	2.4729	2	1.236	1189	66.9	82.7	46.61%	1.34	0.96	2.05	2.9			1189			
98 A1-C3-PW3	26.10%	2.3377	2	1.169	879	50.7	59.3	33.39%	1.28	0.99	3.04	3.1			879			
99										AVE =	2.99	total Cs-137 in wash			3479			
100 DI water rinse																		
101																		
102																		
103																		
104																		
105 Vial/Bottle	net cnts per mL of sample	C/Co	mass of waste counted	volume	density	¹³⁷ Cs per sample	¹³⁷ Cs per g of sample	¹³⁷ Cs per mL of sample	C/Co based on analytical	analytical C/Co	Bed Volumes	cumulative e BV's	Flow rate		Cs-137 in wash			
106	cnts/mL/min		g	mL	g/mL	uCi	uCi/g	uCi/mL			BV	BV	BV/hr		uCi			
107 A1-C3-PR1	53852	18.36%	2.1463	2	1.073	1118.6	32.9	35.4	19.95%	1.09	1.05	1.05	3.3		561			
108 A1-C3-PR2	28081	9.58%	2.0695	2	1.035	605.2	17.8	18.5	10.42%	1.09	1.10	2.15	3.3		306			
109 A1-C3-PR3	21685	7.39%	2.0299	2	1.015	479.6	14	14.3	8.06%	1.09	1.14	3.29	2.9		246			
110 A1-C3-PR4	8095	2.76%	2.014	2	1.007	168.3	5.16	5.22	2.94%	1.07	1.03	4.33	3.1		81			
111 A1-C3-PR5	3649	1.24%	2.0078	2	1.004	75.0	2.31	2.33	1.31%	1.06	1.03	5.36	3.1		36			
112 A1-C3-PR6	2032	0.69%	2.002	2	1.001	48.3	1.3	1.31	0.74%	1.07	1.35	6.71	3.0		27			
113												ave =	3.10		total Cs-137 in wash			
114																		

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R
115	Elution - 1st Column																	
116																		
117			Start Date and Time:		6/10/99	8:21												
118																		
119			diluent and eluant (0.5 M nitric) density @ 25 °C				1.014	g/mL										
120	Vial/Bottle	ACL number	sample start time	sample finish time	Elapsed time	cumulative time	mass of vial + cap	mass of vial + cap + sample	mass of sample	volume of eluant	mass of dilution vial + cap	mass of diluent vial + cap + diluent	mass of diluent vial + cap + diluent + eluant	mass of sample + diluent	volume of diluent	mass of sample transferred to dilution vial	mass of sample + diluent transferred to Analytical vial	mass of eluant counted
121			hr:min	hr:min	hr	hr	g	g	g	mL	g	g	g	g	mL	g	g	g
122	A1-Cs-E1-1		8:21	8:39	0.300	0.300	16.964	19.1003	2.136	2.107	16.795	26.505						
123	A1-Cs-E1-2	99-2055	8:50	9:50	1.000	1.300	16.843	33.3892	16.546	16.318	16.889	27.010	27.471	10.582	10.040	0.461	10.152	0.442
124	A1-Cs-E1-3	99-2056	9:53	11:32	0.700	2.000	16.864	33.8635	17.000	16.765	16.902	27.023	27.086	10.184	10.040	0.063	9.932	0.062
125	A1-Cs-E1-4	99-2057	11:35	12:32	0.950	2.950	16.882	34.7913	17.909	17.662	16.925	27.055	27.149	10.224	10.049	0.095	9.868	0.091
126	A1-Cs-E1-5	99-2058	12:38	1:35	0.950	3.900	16.924	35.2461	18.322	18.069	16.984	27.116	27.217	10.234	10.052	0.101	9.926	0.098
127	A1-Cs-E1-6	99-2059	1:39	2:33	0.900	4.800	16.780	34.264	17.485	17.243	16.941	27.066	27.156	10.215	10.045	0.090	9.922	0.088
128	A1-Cs-E1-7	99-2060	2:36	3:36	1.000	5.800	16.870	36.6603	19.790	19.517	16.926	27.068	27.164	10.238	10.062	0.095	9.875	0.092
129	A1-Cs-E1-8	99-2061	3:38	4:30	0.867	6.667	16.760	33.6586	16.899	16.666	16.750	26.474	26.957	10.207	9.647	0.483	8.861	0.419
130	A1-Cs-E1-9	99-2062	4:35	5:26	0.850	7.517	16.916	32.8171	15.902	15.682	16.803	26.528	27.010	10.207	9.648	0.482	8.755	0.413
131	A1-Cs-E1-10	99-2063	5:29	6:29	1.000	8.517	16.900	35.0337	18.134	17.884	16.793	21.903	-	na	na	na	17.855	17.855
132	A1-Cs-E1-11	99-2064	6:31	7:31	1.000	9.517	17.036	35.4636	18.428	18.173	16.812	21.930	-	na	na	na	18.064	18.064
133	A1-Cs-E1-12	99-2065	7:33	8:29	0.933	10.450	16.912	33.7219	16.810	16.578	16.664	21.773	-	na	na	na	16.335	16.335
134	A1-Cs-E1-comp	99-2289																
135	Elution rinse DI water																	
136																		
137			Start Date and Time		6/11/99	8:39												
138	Vial/Bottle	ACL number	sample start time	sample finish time	Elapsed time	cumulative time	mass of vial + cap	mass of vial + cap + sample	mass of sample	volume of sample	mass of sample transferred to analytical vial	mass of sample counted	count time	background	Total counts	total cnts - bckg	net cnts per g of sample	net cnts/mL of sample/minute
139			hr:min	hr:min	hr	hr	g	g	g	mL	g	g	min	cnts	cnts	cnts	cnts/g	cnts/mL/min
140	A1-Cs-E1-R1	99-2066	8:55	9:10	0.250	0.250	16.935	33.3453	16.410	16.410	15.344	15.344	3	56	119012	118956	7753	2618
141	A1-Cs-E1-R2	99-2067	9:12	10:00	0.800	1.050	16.911	35.4564	18.545	18.545	18.047	18.047	3	56	27700	27644	1532	511
142	A1-Cs-E1-R3	99-2068	10:15	10:35	0.333	1.383	17.024	34.6139	17.590	17.590	17.036	17.036	1	56	11385	11329	665	664
143	A1-Cs-E1-R4	99-2069	10:36	10:58	0.367	1.750	17.025	36.5217	19.497	19.497	18.980	18.980	1	56	5052	4996	263	263
144	A1-Cs-E1-R5	99-2070	11:00	11:19	0.317	2.067	16.876	33.1322	16.256	16.256	15.841	15.841	1	56	3110	3054	193	193
145	A1-Cs-E1-R6	99-2071	11:20	11:40	0.333	2.400	16.811	35.6997	18.889	18.889	18.373	18.373	1	56	31664	31608	1720	1715
146																		

	A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ
115	Elution - 1st Column																		
116																			
117																			
118																			
119																			
120	Vial/Bottle	count time	background	Total counts	total cnts - bckg	net cnts per g of sample	net counts/ mL of sample/ minute	¹³⁷ Cs analytical value	mass of sample counted	mass of eluant counted	¹³⁷ Cs per sample	¹³⁷ Cs per g of eluant	¹³⁷ Cs per mL of eluant	C/Co based on portable GEA	C/Co based on analytical data	analytical C/Co/ portable GEA C/Co	Bed volumes of eluant (8)	Cumulative bed volumes (8)	Flow rate
121		min	cnts	cnts	cnts	cnts/g	cnts/mL/min	uCi/g	g	g	uCi	uCi/g	uCi/mL				BV	BV	BV/hr
122	A1-Cs-E1-1																0.14	0.1	
123	A1-Cs-E1-2	3	146	2299	2153	4866	1645	4.56E-02	2.0299	0.0885	17	1.0	1.1	0.0056	0.006	1.07	1.08	1.2	1.1
124	A1-Cs-E1-3	3	146	3436	3290	53461	18070	7.17E-02	2.0278	0.0126	197	12	11.7	0.0616	0.066	1.07	1.11	2.3	1.6
125	A1-Cs-E1-4	3	146	166173	166027	1816472	613967	3.56E+00	2.0318	0.0188	6884	384	389.7	2.0937	2.196	1.05	1.17	3.5	1.2
126	A1-Cs-E1-5	0.115	146	307913	307767	3129393	27593085	1.68E+02	2.0336	0.0201	310653	16955	17192.4	94.0974	96.878	1.03	1.20	4.7	1.3
127	A1-Cs-E1-6	3	146	539794	539648	6166551	2084294	1.19E+01	2.0287	0.0179	23589	1349	1368.1	7.1078	7.709	1.08	1.15	5.9	1.3
128	A1-Cs-E1-7	3	146	8730	8584	93577	31629	1.85E-01	2.0303	0.0189	394	20	20.2	0.1079	0.114	1.05	1.30	7.2	1.3
129	A1-Cs-E1-8	3	146	20495	20349	48579	16420	-	-	-	166	-	-	0.0560	-	-	1.11	8.3	1.3
130	A1-Cs-E1-9	3	146	10497	10351	25057	8469	2.30E-01	2.0305	0.0958	78	4.9	4.9	0.0289	0.028	0.96	1.04	9.3	1.2
131	A1-Cs-E1-10	3	146	296578	296432	16602	5612	-	-	-	61	-	-	0.0191	-	-	1.19	10.5	1.2
132	A1-Cs-E1-11	1	146	69434	69288	3836	3889	-	-	-	43	-	-	0.0133	-	-	1.21	11.7	1.2
133	A1-Cs-E1-12	1	146	48113	47967	2937	2978	1.91E+00	2.0297	2.0297	32	1.9	1.9	0.0102	0.011	1.07	1.10	12.8	1.2
134	A1-Cs-E1-comp																	ave =	1.23
135	Elution rinse DI water																		
136																			
137																			
138	Vial/Bottle	sample density	¹³⁷ Cs in sample	¹³⁷ Cs analytical value	¹³⁷ Cs analytical value	C/Co based on portable GEA	C/Co based on analytical data	analytical C/Co/ portable GEA C/Co	Bed volumes of sample	cumulative BV's of elution rinse	cumulative BV's of elution rinse + eluant	Flow rate							
139		g/mL	uCi	uCi/g	uCi/mL				BV	BV	BV	BV/hr							
140	A1-Cs-E1-R1	1.013	84	5.110	5.110	8.93E-03	2.88E-02	3.23	1.09	1.09	13.90	4.4							
141	A1-Cs-E1-R2	-	6	-	-	1.74E-03	-	-	1.23	2.32	15.13	1.5							
142	A1-Cs-E1-R3	0.999	8	0.444	0.444	2.27E-03	2.50E-03	1.10	1.17	3.49	16.30	3.5							
143	A1-Cs-E1-R4	-	3	-	-	8.98E-04	-	-	1.30	4.79	17.59	3.5							
144	A1-Cs-E1-R5	-	2	-	-	6.57E-04	-	-	1.08	5.87	18.67	3.4							
145	A1-Cs-E1-R6	0.997	21	1.110	1.110	5.85E-03	6.25E-03	1.07	1.26	7.12	19.93	3.8							
146									ave +	2.97									

(8) sampling initiated immediately after starting to pump nitric acid. The first 1 or 2 column volumes were likely water. Density measurements indicate sample (column volume) # 2 was nitric acid.

[illegible]

	A	S	T	U	V	W	X	Y	Z	AA	AB	AC	AD	AE	AF	AG	AH	AI	AJ
147	Regeneration (0.25 M NaOH)																		
148	Vial/Bottle	¹³⁷ Cs analytical value uCi/g	¹³⁷ Cs analytical value uCi/mL	C/Co based on analytical data	analytical C/Co/ portable GEA C/Co	Bed volumes of regeneration n soln BV	cumulative BV's of elution rinse + eluant + regen soln BV	Flow rate BV/hr											
149																			
150	Regeneration	6.84E-02	6.88E-02	3.88E-04	0.97	6.2	26.11	5.9											
151																			
152	Chemical supply samples																		
153	vial																		
154																			
155	0.1 M NaOH																		
156	0.25 M NaOH																		
157	0.5 M nitric																		
158	DI water																		
159																			
160	Elution - 2nd Column																		
161																			
162																			
163																			
164	Vial	net cnts/mL of sample/ minute	portable GEA C/Co	¹³⁷ Cs															
165		cnts/mL/min		uCi															
166	Al-Cs-E2-CO, Eluate column 2 composite	405989	1.38	44709.90															
167	final eluate sample from column # 2	10106	0.034																
168																			

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R
169	Loading of non cesium radionuclides																	
170	Run 1 ----->																	
171				Co-60	RuRh-106	Sb-125	SnSb-126	Eu-154				Co-60	RuRh-106	Sb-125	SnSb-126	Eu-154		
172		ACL number		uCi/mL	uCi/g	uCi/mL	uCi/mL	uCi/mL				uCi/g	uCi/g	uCi/g	uCi/g	uCi/g		
173	A1-Cs-L1	99-2020	lead #1	7.15E-04			2.65E-04			A1-Cs-L1	lead col #1	5.82E-04			2.16E-04			
174	A1-Cs-L5	99-2024	lead #5	6.45E-04						A1-Cs-L5	lead col #5	5.25E-04						
175	A1-Cs-L7	99-2026	lead #7	8.30E-04						A1-Cs-L7	lead col #7	6.76E-04						
176	A1-Cs-L10	99-2029	lead #10	7.44E-04						A1-Cs-L10	lead col #10	6.06E-04						
177																		
178	A1-Cs-P1	99-2037	lag col #1	5.21E-04	4.59E-04	1.35E-04	2.65E-04	5.62E-05		A1-Cs-P1	lag col #1	4.24E-04	3.74E-04	1.10E-04	2.16E-04	4.58E-05		
179	A1-Cs-P3	99-2039	lag col #3	6.37E-04			4.83E-04			A1-Cs-P3	lag col #3	5.19E-04			3.93E-04			
180	A1-Cs-P5	99-2041	lag col #5	8.09E-04						A1-Cs-P5	lag col #5	6.59E-04						
181	Run 2 ----->																	
182	A1R-Cs-0	00-0341	Cs IX feed sample	6.31E-04	<1.2E-2	<7E-3	<7E-3	<3.6E-4		A1R-Cs-0	Cs IX feed sample	5.14E-04	<1E-2	<6E-03	<6E-03	<3E-4		
183	A1R-Cs-L1	00-0342	lead #1	3.57E-04			2.23E-04	4.13E-05				2.91E-04			1.82E-04	3.36E-05		
184	A1R-Cs-L2	00-0343	lead #2	3.72E-04			1.98E-04	3.97E-05				3.03E-04			1.61E-04	3.23E-05		
185																		
186	A1R-Cs-P1	00-0344	lag col #1	3.43E-04	4.18E-04	1.06E-04	2.60E-04	2.11E-05		A1R-Cs-P1	lag col #1	2.79E-04	3.40E-04	8.60E-05	2.12E-04	1.72E-05		
187	A1R-Cs-P2	00-0345	lag col #2	3.78E-04	3.41E-04	1.21E-04	2.68E-04	3.52E-05		A1R-Cs-P2	lag col #2	3.08E-04	2.78E-04	9.85E-05	2.18E-04	2.87E-05		
188	A1R-Cs-P7	00-0346	lag col #7	3.40E-04	2.82E-04	8.56E-05	2.17E-04	2.19E-05		A1R-Cs-P7	lag col #7	2.77E-04	2.30E-04	6.97E-05	1.77E-04	1.78E-05		
189	A1-Tc-LC	00-0349	c IX efflu	3.54E-04	3.48E-04	1.04E-04	2.51E-04	2.86E-05		A1-Tc-LC	c IX effluen	2.88E-04	2.83E-04	8.48E-05	2.04E-04	2.33E-05		
190																		
191																		
192	Cs-137 Mass Balance																	
193																		
194	Cs-137 at start		mass of feed, g	volume of feed, mL	Cs-137, uCi/mL	Cs-137, uCi												
195			3016	2456	177.5	435857												
196																		
197	Cs-137 recovered		mass	volume	Cs-137 uCi/mL	Cs-137 ⁽⁷⁾ uCi	% of original ⁽⁷⁾	Cs-137 ⁽⁸⁾ uCi	% of original ⁽⁸⁾									
198			g	mL	uCi/mL	uCi		uCi										
199	Effluent # 1		2271	1849	10.917	20186	4.63%	20186	4.63%									
200	Effluent # 2		637	518	56.120	29088	6.67%	29088	6.67%									
201	load samples # 1		72	59		2383	0.55%	2383	0.55%									
202	load samples # 2		36	29		696	0.16%	696	0.16%									
203	Feed displacement		55.5	45.7		3479	0.80%	3479	0.80%									
204	DI water rinse		204.5	103.2		2495	0.57%	2495	0.57%									
205	Elution, 1st Col:		197.8	194.9	1610.0	313752	71.98%	342113	78.49%									
206	DI water rinse col # 1		107.2	107.2		123	0.03%	123	0.03%									
207	regeneration solution #1		93.2	93.0	0.069	6	0.00%	6	0.00%									
208	Elution, 2nd Col:		184.7	182.0	242.0	44037	10.10%	44037	10.10%									
209	DI water rinse #2		data not taken ----->															
210	regeneration solution #2		data not taken ----->															
211	Total:					416246	95.50%	444606	102.01%									

(7) Cesium content in eluants is based on concentration in composite samples.
(8) Cesium content in eluants is based on summation of individual samples.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	Loading of SL-644 with AW-101 feed													
2														
3	Loading													
4			Start Date and Time:		6/16/99	11:21								
5			Bed volume col 1 =		15.0	mL								
6			Bed volume col 2 =		15.0	mL								
7			Empty Eff Btl - 2 w/ cap(w / holes) + plastic wrap =				262.7	g						
8			Empty Eff Btl -3 w/ cap(w / holes) + plastic wrap =				265.1	g						
9			Bed volumes diverted to waste =				3.75							
10			Supernate Density =				1.228	(g/ml)						
11			Co =	716384	cnts/g of sample =		238795	cnts/g of sample/minute		run 1				
12			Co =	91052	cnts/g of sample =		30351	cnts/g of sample/minute		run 2				
13			regeneration and diluent (0.25 M NaOH) density @ 25 °C =						1.008	g/mL				
14			Feed analyses: Cs-137, uCi/mL											
15			Date	uCi/mL										
16			Mar-99	179.3	Batch contacts with AW-101 feed and SL-632; sample # RD1AW-C									
17			May-99	174.7	Batch contacts with AW-101 feed and SL-644; sample # AW-101-C									
18			May-99	176.0	Batch contacts with AW-101 feed and SL-644; sample # AW-101-S1									
19			May-99	177.7	Batch contacts with AW-101 feed and SL-644; sample # AW-101-S2									
20			Jun-99	179.6	Column run feed analysis									
21			average	177.5	Estimated Cs-137 concentration run 2 feed =				22.6	uCi/mL				
22					ratio of run 2 feed:run 1 feed =				0.127					
23					Cs-137 concentration ACL feed analysis =				20.9	uCi/mL				
24														
25	The effluent volume on which the bed volumes is based was determined by estimating the total mass and volume of waste pumped through the columns at the time of sampling. For column #1 this includes the mass of the waste in the effluent bottle + the samples taken from column # 1 and # 2 prior to the sample of interest (the mass of the effluent bottle was recorded during each sample event). For column # 2 the effluent volume includes the mass of the waste in the effluent bottle + the samples taken from column #2 prior to the the sample of interest (samples taken from column # 1 did not go through column # 2. A hold up volume of 2 bed volumes (30 mL) is added for column # 1. This is the system volume between the end of the effluent line and valve 3. This is actually added in the bed volume column in the spreadsheet. The holdup between the bottom of column # 2 and the effluent bottle was not accounted for.													
26														
27														
28														
29														
30														
31														
32														

	A	O	P	Q	R	S	T	U
1	Loading of SL-644 w							
2								
3	Loading							
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	A	B	C	D	E	F	G	H	I	J	K	L	M	N
33	Vial		sample time	pumping time	sample mass taken	sample mass counted	sample volume counted	count time, min	Tot Cts	back ground	Tot cts-bck	counts/g of sample	Cs-137 concentration (ACL)	C/Co portable GEA
34			(hr:mm:ss)	(hr)	g								uCi/mL	
35	A1R-Cs-0 (1)	00-0341	11:53:00	0.000	2.3976	2.0906	1.7024	3	190379	25	190354	91052	20.9	
36	A1R-Cs-L1 (2)	00-0342	16:11:00	2.720	3.3419	2.9013	2.3626	3	2889	25	2864	987	2.11E-01	1.084%
37	A1R-Cs-L2 (3)	00-0343	18:56:00	5.200	4.2685	3.8955	3.1722	3	5429	25	5404	1387	3.12E-01	1.524%
38	A1R-Cs-L3 (4)	--	10:44:00	7.970	4.1405	3.8129	3.1050	3	10801	25	10776	2826		3.104%
39	A1R-Cs-L4	--	13:18:00	10.537	3.2783	2.9950	2.4389	3	18559	25	18534	6188		6.796%
40	A1R-Cs-L5	--	15:50:00	13.070	3.8069	3.4629	2.8200	3	45932	25	45907	13257		14.560%
41	A1R-Cs-L6 (5)	--	18:30:00	15.537	2.7523	2.0593	1.6770	3	40936	25	40911	19866		21.819%
42	A1R-Cs-L7	--	21:08:00	18.170	3.4386	3.1119	2.5341	3	90377	25	90352	29034		31.888%
43			21:50:00	18.870										
44														
45			(1) feed started at 11:21 on 6/16/99 and effluent diverted to waste bottle until 11:53 at which time effluent collection was initiated. (2) pump off from 1:20-2:55 to remove air from columns, settle floating resin, fix a loose connection. (3) pump off from 17:58-16:14 to switch feed bottles. (4) pump shut down at 19:09 for the night until 8:11 AM on 6/17/99. (5) pump off from 16:35 to 16:47 on 6/17/99											
46														
47														
48														
49														
50														
51														
52														
53	Vial		sample time	elapsed time	sample mass taken	sample mass counted	count time, min	Tot Cts	back ground	Tot cts-bck	counts/g of sample counted	Cs-137 concentration (ACL)	C/Co (portable GEA)	C/Co (ACL)
54			(hr:mm:ss)	(hr)	g							uCi/mL		
55	A1R-Cs-0	00-0341	11:53:00	0.000	2.3976	2.091	3	190379	25	190354	30351	20.9		
56	A1R-Cs-P1	00-0344	16:11:00	2.720	3.0742	2.7006	3	290	25	265	33	1.85E-02	0.108%	0.0885%
57	A1R-Cs-P2	00-0345	18:56:00	5.200	3.5324	3.2633	3	356	25	331	34	1.97E-02	0.111%	0.0943%
58	A1R-Cs-P3	--	10:44:00	7.970	4.3019	3.8995	3	376	25	351	30	--	0.099%	--
59	A1R-Cs-P4	--	13:18:00	10.537	3.132	2.8337	5	562	25	537	38	--	0.125%	--
60	A1R-Cs-P5	--	15:50:00	13.070	4.2113	3.9199	5	763	25	738	38	--	0.124%	--
61	A1R-Cs-P6	--	18:30:00	15.537	3.2822	2.9355	5	615	25	590	40	--	0.132%	--
62	A1R-Cs-P7	00-0346	21:08:00	18.170	5.8958	5.5628	5	1671	25	1646	59	2.91E-02	0.195%	0.14%
63														

	A	O	P	Q	R	S	T	U
33	Vial	C/Co (ACL)	Cs-137	Eff Btl Mass	Effluent Mass + samples	Eff Vol	Bed volumes	Flow rate
34			uCi	(g)	(g)	(mL)	BV	BV/hr
35	A1R-Cs-0 (1)			265.1	0	0.0	0.0	
36	A1R-Cs-L1 (2)	1.010%	0.57	673.7	415.0	338.0	24.5	8.99
37	A1R-Cs-L2 (3)	1.493%	1.08	1059.4	808.5	658.4	45.8	8.59
38	A1R-Cs-L3 (4)		2.19	1449.5	1207.1	982.9	67.3	7.79
39	A1R-Cs-L4		3.79	1844.1	1608.1	1309.5	89.0	8.46
40	A1R-Cs-L5		9.43	2226	1998.0	1627.0	110.1	8.33
41	A1R-Cs-L6 (5)		10.22	412	2375.4	1934.4	130.6	8.28
42	A1R-Cs-L7		18.66	797.3	2770.1	2255.7	151.9	8.11
43				828.7	2801.5	2281.3	153.6	
44							ave =	8.36
45								
46								
47		Initial DF for lead column =			99			
48								
49								
50								
51								
52								
53	Vial	analytical/ portable C/Co	Cs-137	Eff Btl Mass	Effluent Mass + sample	Eff Vol	Bed volumes	
54			uCi	(g)	(g)	(mL)	BV	
55	A1R-Cs-0			265.1	0			
56	A1R-Cs-P1	0.82	0.05	673.7	411.7	335.2	22.3	
57	A1R-Cs-P2	0.85	0.06	1059.4	800.9	652.2	43.4	
58	A1R-Cs-P3	--	0.07	1449.5	1195.3	973.4	64.7	
59	A1R-Cs-P4	--	0.07	1844.1	1593.0	1297.3	86.2	
60	A1R-Cs-P5	--	0.09	2226	1979.2	1611.7	107.1	
61	A1R-Cs-P6	--	0.07	412	2353.8	1916.8	127.4	
62	A1R-Cs-P7	0.71	0.20	797.3	2745.0	2235.4	148.6	
63				828.7	2776.4	2260.9	150.3	

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
64	Bottle		mass of bottle	final mass of bottle + waste	mass of waste	volume of waste	Cs-137 (ACL)	C/Co						
65			g	g	g	mL	uCi/mL							
66	Effluent bottle 2	--	262.7	828.7	566.0	461	--	--						
67	Effluent bottle 3	--	265.1	2448.1	2183	1778	--	--						
68	A1-Tc-LC	00-349			2749	2239	5.89E-02	2.82E-03						
69				initial run 2 DF, column #1 =		99								
70				initial run 2 DF column #2 =		1130								
71				composite run 2 DF =		355		composite DF (run #1 + run #2) =			3014			
72														
73	Feed Displacement: 0.1 M NaOH													
74														
75	Data not taken													
76														
77	DI water rinse													
78														
79	Data not taken													
80														
81	Elution - both columns													
82				diluent and eluant (0.5 M nitric) density @ 25 °C =				1.014	g/mL					
83			mass	Cs-137	volume	Cs-137								
84			g	uCi/mL	mL	uCi								
85	A1R-Cs-E-comp	00-0617	316.6	147	312	45898								
86														
87	Elution rinse DI water													
88														
89	Data not taken													
90														
91	Regeneration (0.25 M NaOH)													
92														
93	Data not taken													
94														

	A	O	P	Q	R	S	T	U
64	Bottle	Initial DF for lag column	1130					
65								
66	Effluent bottle 2							
67	Effluent bottle 3							
68	A1-Tc-LC	Average DF	3.55E+02					
69								
70								
71								
72								
	Feed							
	Displacement: 0.1							
73	M NaOH							
74								
75	Data not taken							
76								
77	DI water rinse							
78								
79	Data not taken							
80								
	Elution - both							
81	columns							
82								
83								
84								
85	A1R-Cs-E-comp							
86								
	Elution rinse DI							
87	water							
88								
89	Data not taken							
90								
	Regeneration (0.25							
91	M NaOH)							
92								
93	Data not taken							
94								

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
95	Cs-137 Mass													
96	Balance													
97	Cs-137 at start			mass of feed, g	volume of feed, mL	Cs-137, uCi/mL	Cs-137, uCi							
98				2801	2281	20.9	47680							
99														
100	Cs-137 recovered			mass	volume	Cs-137	Cs-137	% of total						
101				g	mL	uCi/mL	uCi							
102	Effluent comp			2749	2239	0.059	132	0.28%						
103	load samples # 1			25	20		46	0.10%						
104	load samples # 2			27	22		1	0.00%						
105	Elution, 1st Col:			316.6	312.2		45898	96.26%						
106	Total:						46076	96.64%						

SL-644 Kd's with AW-101 Column Feed

Density of AW-101 waste diluted for IX				Resin batch number: 644BZ, std Kd= 570 mL/g						
pipette check	T= 23 C	density of water	0.99753 g/mL	As received resin density =		0.86 g/mL				
mass delivered, g	volume, mL	waste mass delivered, g	waste density, g/mL							
5.0099	5.0223	6.1707	1.2347	f factor @ = 95 °C		0.8008				
4.9529	4.9652	6.2034	1.2412	dry density =		0.689 g/mL				
4.9755	4.9878	6.1863	1.2378							
4.9841	4.9964	6.1674	1.2340							
5.0047	5.0171	6.1872	1.2380							
ave	4.9978	6.1798	1.2365							
		ave waste density =	1.2370							
waste density by vol flask (use this value) =			1.228 g/mL							
pipett check for analytical dilutions				Density of 0.25 M NaOH @ 25 C		1.0078 g/mL				
	0.4809 mL									
	0.4795 mL									
	0.4787 mL			Average Cs-137 concentration =		177.50 uCi/mL				
	0.4775 mL			Average Cs-134 concentration =		3.44E-02 uCi/mL				
	0.4788 mL									
average	0.47908 mL									

SL-644 Kd's with AW-101 Column Feed

Analytical Sample ID	AW101-644	AW101-644D	AW101-S1-644	AW101-S1-644D	AW101-S ² ₁ -644	AW101-S ² ₁ -644D	AW-101-C	AW-101-S1	AW-101-S2		
analytical Cs-137, uCi/mL	1.82	2.25	3.9	3	3.59	3.47	8.41	8.65	10.6		
analytical Cs-134, uCi/mL	3.56E-04	4.08E-04	8.08E-04	5.33E-04	6.74E-04	6.91E-04	1.72E-03	1.51E-03	2.20E-03		
net waste added to analytical vial, g	0.5816	0.5685	0.5891	0.5916	0.594	0.594	0.5931	0.6037	0.7446		
analytical waste volume, mL	0.4736	0.4629	0.4797	0.4818	0.4837	0.4837	0.4830	0.4916	0.6064		
mass of vial + 0.25 M NaOH	26.4968	26.5595	26.5409	26.4009	26.7023	26.5952	26.4303	26.4880	26.4995		
Tare, g	16.8542	16.9284	16.9000	16.7618	17.0955	16.9990	16.8037	16.9043	16.8651		
diluent mass, g	9.6426	9.6311	9.6409	9.6391	9.6068	9.5962	9.6266	9.5837	9.6344		
diluent volume, mL	9.57	9.56	9.57	9.56	9.53	9.52	9.55	9.51	9.56		
dilution factor	21.20	21.64	20.94	20.85	20.71	20.69	20.78	20.34	16.77		
Cs-137, sample concentration, uCi/mL	38.59	48.70	81.67	62.56	74.34	71.78	174.74	175.97	177.72		
Cs-137, sample concentration, M	3.25E-06	4.10E-06	6.88E-06	5.27E-06	6.27E-06	6.050E-06	1.473E-05	1.483E-05	1.498E-05		
total cesium	1.32E-05	1.67E-05	1.70E-04	1.31E-04	2.58E-04	2.49E-04	5.97E-05	3.67E-04	6.17E-04		
Cs-134, sample concentration, uCi/mL	7.55E-03	8.83E-03	1.69E-02	1.11E-02	1.40E-02	1.43E-02	3.57E-02	3.07E-02	3.69E-02		

Sodium concentration is based on the ICP results for the Cs IX column run conducted 6/99. See ASR 5421. Density measured in 25 mL volumetric flask at 1.228 g/mL.

Note: total cesium in waste is based on Cs-137 concentration and the TIMS results which indicate that the Cs-137 mole fraction is 0.2465. The spikes are adjusted for the addition of 0.1 M cesium nitrate. See Cs spike prep sheet.

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