PNWD-3240, Rev 1
WTP-RPT-033, Rev 1

Small Column Ion Exchange Testing of SuperLig® 644 for Removing ^{137}Cs from Hanford Waste Tank 241-AN-102 Supernate (Envelope C) Mixed with Tank 241-C-104 Solids (Envelope D) Wash and Permeate Solutions

S. K. Fiskum
S. T. Arm
D. L. Blanchard, Jr

February 2004

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

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SK Fiskum
ST Arm
DL Blanchard, Jr

for W.L. Tamosaitis
4/14/04

ACCEPTED FOR
PROJECT USE

February 2004

Test specification: TSP-W375-00-00028, Rev. 1
Test plan: TP-RPP-WTP-013, Rev. 0
Test exceptions: None
R&T focus area: Pretreatment
Test Scoping Statement(s): B-46

Battelle, Pacific Northwest Division
Richland, Washington, 99352

Completeness of Testing

This report describes the results of work and testing specified by TSP-W375-00-00028, Rev. 1 and TP-RPP-WTP-013, Rev. 0. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

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

2/24/04
Date

History Sheet

Rev	Date	Reason for revision	Revised by
0	January 2003	New Document	SK Fiskum
1	February 2004	Noted higher uncertainty in the total ^{99}Tc values in Tables 2.3 and 3.3 as footnotes.	SK Fiskum
		Replaced the following ICP-MS report with Revision 2 version: multi-element narrative report for ASR 6107.	
		Added Revision 2 ICP-MS report for ASR 6281, Cs and Tc-99 analysis.	

Summary

The River Protection Project-Waste Treatment Plant (RPP-WTP) baseline process for ^{137}Cs removal from Hanford high-level tank waste is ion exchange. The current flowsheet includes the use of Cs-selective, organic ion exchanger SuperLig® 644 (SL-644) material for Cs removal from the aqueous waste fraction. This material has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT. The RPP-WTP Development Requirements Document (DRD)(a) Task 8.2.6 and the RPP-WTP Research and Technology schedule identify Cs and Tc ion exchange process verification tests [WTP Request for Proposal reference Standard 2 Item (a)(3)(ii)].

Battelle, Pacific Northwest Division (PNWD) was contracted to perform Cs ion exchange studies under Contract 24590-101-TSA-W000-0004 and work breakdown structure BN.02.08.05. The Cs ion exchange activities are further defined in Technical Scoping Statement B-46, which is included in Appendix C of the *Research and Technology Plan*.(b) These studies are to verify design and operating parameters for plant-scale ion exchange systems. Test results will also be used to validate ion exchange models.

Objectives

The Cs ion exchange test objectives were to develop load and elution breakthrough profiles using a combination of Hanford tank waste 241-AN-102 supernatant (Envelope C) mixed with wash and permeate solutions from Hanford tank waste 241-C-104 solids (AN-102/C-104); produce and characterize the Cs eluate; remove ^{137}Cs from the AN-102/C-104 to meet low-activity waste (LAW) vitrification criteria; and develop batch-distribution coefficients for AN-102/C-104. The final effluent was to contain <1.75E-5 Ci ^{137}Cs per mole Na, equivalent to < 0.087 μCi ^{137}Cs/mL, based on a 20 wt% waste Na_2O loading in the waste glass. Direction from Bechtel National, Inc. (BNI) for calculating Na_2O loading was later reduced to 10 wt%. All testing objectives were met.

Conduct of Test

This report summarizes testing of the SL-644 in batch-contact studies and in a dual small-column system. The test matrix was AN-102/C-104 evaporated to nominally 4.8 M Na and 161 μCi/mL ^{137}Cs. Batch contacts were performed with the waste at three Cs concentrations at a phase ratio of 100 (liquid volume to exchanger mass) with SL-644. Crystalline silicotitanate (IE-911, an alternative Cs ion exchanger) was also tested similarly to the SL-644; however, the contact solution was AN-102 tank waste supernatant diluted to nominally 6 M Na^+. Ion exchange processing was conducted with two small columns in series with resin bed volumes (BVs) of 10.4 mL (L/D(c) = 4.2) during the conditioning phase with 0.25 M NaOH, and 9.9 mL (L/D = 4.0) during the AN-102/C-104 loading phase. Proper functioning

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(a) PL-W375-TE00002, Rev. 1, River Protection Project Waste Treatment Plant Development Requirements Document, October 31, 2000, M. E., Johnson and T. W. Crawford, CH2MHill Hanford Group, Inc., Richland, WA. DRAFT.

(b) Barnes, S., R. Roosa, and R. Peterson. 2002. '*Research and Technology Plan*.', 24590-WTP-PL-RT-01-002, Rev. 1, RPP-WTP project.

(c) L/D, equal to length over diameter, is the resin bed aspect ratio.

of the ion exchange apparatus and resin beds had initially been tested with an AW-101 (Envelope A) simulant. The resin beds had then been used to process 1.2 L of AP-101 diluted feed, an Envelope A waste feed. The AN-102/C-104 waste sample processed was approximately 753 mL, corresponding to 72 BVs. All ion exchange process steps were tested, including resin-bed preparation, loading, feed displacement, water rinse, elution, eluant rinse, and resin regeneration.

Results and Performance Against Objectives

The batch-contact performance data are summarized in Table S.1. The K_d values for SL-644 and IE-911 are shown for the feed condition at the Na/Cs mole ratio of 8E+4. The calculated Cs λ value (column-distribution ratio) is the predicted BVs required to reach 50% Cs breakthrough. It is a function of the equilibrium K_d value and bed density.

Table S.1. Summary of Performance Measures

Flow rate (BV/h)	Extrapolated 50% Cs breakthrough, BV		Composite DF$^{(2)}$	Maximum DF$^{(3)}$	K_d, mL/g (feed condition)		Cs λ, BV (feed condition)	
	Lead column	Lag column			SL-644	IE-911	SL-644	IE-911
2.7	NM$^{(1)}$	NM$^{(1)}$	1.13 E+4	7.7 E+5	950	1100	250	1100

(1) NM = not measured; AN-102/C-104 feed volume was insufficient to establish a breakthrough curve.
(2) The decontamination factor (DF) is calculated by dividing the feed Cs concentration by the composite effluent Cs concentration, based on the total of 67 BVs of feed. This does not necessarily reflect the DF that could be obtained with an estimated loading of 250 BVs to reach 50% C/C_o.
(3) The maximum DF is obtained by dividing the feed Cs concentration by the lowest lead column sample Cs concentration.

The column performance data are also summarized in Table S.1. The 50% Cs breakthrough value from column testing could not be determined; no measurable Cs broke through the lead column. The decontamination factor (DF) for ^{137}Cs was based on ^{137}Cs concentration in the feed divided by the ^{137}Cs concentration in the composite effluent sample. The composite effluent ^{137}Cs concentration was 1.4E-02 µCi/mL and was equivalent to a DF of 1.13 E+4. The maximum DF, 7.7 E+5, measured the best performance that could be expected from this column system. It was calculated relative to the sample containing the lowest ^{137}Cs concentration, i.e., the eighth sample from the lead column. The low-activity vitrified waste form must be no greater than 0.3 Ci/m^3; this limit can be converted to ^{137}Cs concentration in the ion exchange effluent (contract limit). The composite effluent ^{137}Cs concentration was an order of magnitude below the minimum waste loading contract limit (0.168 µCi ^{137}Cs/mL).[(d)]

The lead column was eluted with 0.5 M HNO_3 reaching a C/C_o of 1% in 11 BVs with >99% of the ^{137}Cs contained in 3 BVs of eluate. The peak ^{137}Cs C/C_o value was 53 (based on 1-BV collection increments of nominally 10-mL). The ^{137}Cs concentration in the composite eluate was 6.65E+2 µCi/mL, corresponding to a C/C_o of 4.13.

(d) The conversion requires the following assumptions: Envelope C LAW will contain a minimum of 10 wt% Na_2O, all Na comes from the tank waste, the glass density is 2.66 g/mL, and the waste Na concentration is 4.8 M. For the maximum 14 wt% Na_2O waste loading, the Cs ion exchange effluent must be less than 0.120 µCi/mL.

Quality Requirements

This work was designated as QL-3 per the RPP-WTP Quality Assurance (QA) Program, BNFL-5193-QAP-01, Rev. 6. PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the QA plan, CHG-QAPjP, Rev. 0. The Cs eluate was analyzed at the time in accordance with PNWD's *Nuclear Quality Assurance Requirements and Description (NQARD)* manual, which implemented the requirements of DOE/RW-0333P, *Quality Assurance Requirements and Description (QARD)*, and to the approved Test Plan, TP-RPP-WTP-013.

PNWD addressed verification activities by conducting an Independent Technical Review of the final data report in accordance with Procedure QA-RPP-WTP-604. This review verified that the reported results were traceable, that inferences and conclusions were soundly based, and that the reported work satisfied the Test Plan objectives.

Issue

The lag column effluent ^{137}Cs concentration was higher than the lead column ^{137}Cs effluent concentration. The lag column had previously been in the lead column position for processing AP-101 diluted feed. The ^{137}Cs coming off the lag column was attributed to inadequacy of the baseline elution (i.e., elution to C/C_0=1%) to sufficiently elute Cs from the resin during AP-101 processing.

Terms and Abbreviations

AP-101DF	Hanford tank waste AP-101 diluted feed to 5 M Na
AN-102/C-104	Hanford tank waste AN-102 supernatant mixed with wash and permeate solutions from Hanford tank waste C-104 solids
AV	apparatus volume
BNI	Bechtel National, Incorporated
BV	bed volume
C/C_o	analyte concentration in column effluent divided by analyte concentration in feed
CMC	chemical measurement center
CST	crystalline silicotitanate
DF	decontamination factor
DI	deionized
DOE	U.S. Department of Energy
DRD	Development Requirements Document
EQL	estimated quantitation level
F-factor	mass of dry ion exchanger divided by mass of wet exchanger
FMI	Fluid Metering, Inc., Syosset, NY
GEA	gamma energy analysis
HLW	high level waste
IBC	IBC Advanced Technologies, Inc., American Fork, Utah
IC	ion chromatography
ICP AES	inductively coupled plasma/atomic emission spectrometry
ICP MS	inductively coupled plasma/mass spectrometry
IDL	instrument detection limit
KPA	kinetic phosphorescence analysis
λ	column distribution ratio
LAW	low activity waste
L/D	length over diameter ratio
M	molarity, moles/liter
Meq	milli-equivalent
MRQ	minimum reportable quantity

NA	not analyzed
NMRQ	no minimum reportable quantity
NPT	National Pipe Thread
NQARD	Nuclear Quality Assurance Requirements and Description (manual)
OH	hydroxide
PNWD	Battelle, Pacific Northwest Division
QA	quality assurance
QARD	Quality Assurance Requirements and Description
RPL	Radiochemical Processing Laboratory
RPP-WTP	River Protection Project-Waste Treatment Plant
SL-644	SuperLig® 644
SRTC	Savannah River Technology Center
TC	total carbon
TIC	total inorganic carbon
TIMS	thermal ionization mass spectrometry
TOC	total organic carbon
TRU	transuranic
wt%	weight percent
WTP	Waste Treatment Plant

Contents

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Tables

1.0 Introduction

The U. S. Department of Energy plans to vitrify tank wastes at the Hanford Site in preparation for permanent disposal. Before vitrification, tank wastes will be divided into low-activity and high-activity fractions through specific pretreatment processes. The Waste Treatment Plant (WTP) pretreatment flowsheet for the Hanford high-level tank wastes includes the use of SuperLig® 644 (SL-644) material for ^{137}Cs removal from the aqueous waste fraction. The SL-644 is a cesium-selective ion exchanger and has been shown to be effective in removing Cs from a variety of Hanford tank wastes (Hassan, McCabe, and King 2000; Hassan et al. 2000; Hassan et al. 2001; King, Hassan, and McCabe 2001; Kurath, Blanchard, and Bontha 2000a; Kurath, Blanchard, and Bontha 2000b; Fiskum, Blanchard, and Arm 2002; Fiskum et al. 2002). The SL-644 has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

The Cs ion exchange testing was conducted to support the River Protection Project-Waste Treatment Plant (RPP-WTP) Development Requirements Document (DRD)[a] Task 8.2.6, and the RPP-WTP Research and Technology (Barnes et al., 2002) schedule identify the need for Cs and Tc ion exchange process verification tests [WTP Request for Proposal reference Standard 2 item (a)(3)(ii)]. The testing requirements were communicated to Battelle, Pacific Northwest Division (PNWD) according to Test Specification *Tank 241-AN-102 and 241-AP-101 Ion Exchange*, TSP-W375-00-00028, Rev. 1 (M. E. Johnson, CH2M Hill Hanford Group, Dec. 11, 2000). Testing was conducted according to PNWD Test Plan *Actual Waste Ion Exchange Testing for the RPP-WTP Project*, CHG-TP-41500-013, Rev. 0 (D. L. Blanchard, Jan. 24, 2000) and *AP-101 and AN-102/C-104 Actual Waste Ion Exchange Testing*, TP-RPP-WTP-013, Rev. 0 (D. L. Blanchard Jr., August 2001).

This report summarizes batch-contact studies of SL-644 and crystalline silicotitanate[b] (IE-911 provided by UOP LLC, Des Plaines IL), and dual small-column testing of the SL-644 ion exchange material. The test matrix for the small-column ion exchange and SL-644 batch-contact tests was 241-AN-102 Hanford tank waste supernatant, mixed with caustic leaching and wash solutions from the pretreatment of Tank 241-C-104 solids, and evaporated to nominally 5 M Na (hereafter referred to as AN-102/C-104). The test matrix for the IE-911 batch contact was 241-AN-102 supernatant diluted to nominally 6 M Na (hereafter called AN-102). Approximately 753 mL of AN-102/C-104 were processed through the ion exchange column system. 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

- determine distribution coefficients as a function of Na/Cs molar concentration for SL-644 in the AN-102/C-104 matrix and for IE-911 in the AN-102 matrix
- demonstrate the ^{137}Cs decontamination of Envelope C tank waste sample AN-102/C-104 and provide a Cs-decontaminated sample for downstream process testing (i.e., ^{99}Tc removal)

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[a] PL-W375-TE00002, Rev. 1, River Protection Project Waste Treatment Plant Development Requirements Document, October 31, 2000, M. E., Johnson and T. W. Crawford, CH2MHill Hanford Group, Inc., Richland, WA. DRAFT.

[b] IE-911 is an alternative Cs ion exchanger and is being tested in parallel to SL-644 only with batch contacts. The IE-911 was developed and supplied by UOP LLC, Des Plaines, IL.

- develop Cs loading and elution profiles
- produce and characterize a Cs-eluate solution for use in evaporation tests and high-level waste (HLW) vitrification
- demonstrate the effectiveness of all SL-644 ion exchange process steps, including loading, feed displacement, deionized (DI) water washing, elution, and resin regeneration.

2.0 Experimental

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

2.1 SL-644 Resin

The SL-644 was obtained from IBC production batch number 010319SMC-IV-73. This batch exhibited a black-red appearance peppered with light-brown specks. It was received from the vendor as a dry, granular, free-flowing material in a 1-L polyethylene bottle with an approximately 32% gaseous head space. There was no indication that this head space was filled with nitrogen or other inert gas, and no attempt was made to exclude air during storage. The as-received resin form was not identified by the vendor; it was found to contain potassium salts (Fiskum, Blanchard and Arm 2002). The dry-sieved particle-size distribution for this material is shown in Table 2.1. The 212- to 425-μm particle-size dry-sieved fraction of the as-received material was used. This fraction represents 22 wt% of the as-received material. The average particle size of the 212- to 425-μm fraction corresponds to 540-μm diameter when expanded in a solution of 3 M NaOH–2 M $NaNO_3$–0.1M KOH (Fiskum, Blanchard, and Arm 2002). As a general rule, the column diameter should be 20 times greater than the resin particle diameter to minimize wall effects (Korkish 1989, p. 39). Given the diameter of the column at 1.46 cm, the column diameter is 27 times the average diameter of the expanded form of 212- to 425-μm diameter dry-sieved resin. For comparison, the SL-644 particle-size distribution used for AN-102 testing at Savannah River Technology Center (SRTC) (Hassan et al. 2000) is also shown in Table 2.1.

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

Sieve Size(1)	Particle Size (μm)	010319SMC-IV-73 wt %	981020mb48-563 (SRTC) wt %
18	>1000	0.06	
30	600–1000	37.27	57.33
40	425–600	38.23	23.73
50	300–425	18.01	13.71
70	212–300	6.08	5.12
100	150–212	0.26	0.11
140	106–150	0.06	
>140	<106	0.03	

(1) U. S. standard sieve size corresponds to ASTM E-11 specification.

Properties of the 212- to 425-μm 010319SMC-IV-73 SL-644 resin have been previously reported (Fiskum, Blanchard, and Arm 2002), and selected properties are reproduced in Table 2.2. The F-factor is the ratio of the dry mass of exchanger to the initial mass of the exchanger and was determined at the same time the batch contact samples and column resin fractions were weighed. The F-factor was obtained by drying approximately 0.5 g resin, under vacuum, at 50°C to constant mass. The F-factor was determined on the H-form and the as-received form of the resin. The F-factor for the Na-form of the resin was performed differently because of stability problems observed in prior tests on the Na-form of resin (Steimke et al. 2001). Drying to constant mass under vacuum at ambient temperature was considered adequate for removing water from the Na-form resin.[a] The L-factor represents the fractional mass remaining after washing the as-received resin form with 0.5 M HNO_3 and DI water, and correcting for residual water content as described above. The I_{Na} represents the fractional mass gain upon conversion from the H-form to the Na-form, correcting for water content as described above.

Table 2.2. SL-644 Properties

Property	**010319SMC-IV-73**
Bulk density, g/mL (as-received)	0.74
F-factor, as-received	0.877
L-factor, conversion to H-form, fractional mass remaining	0.538
F-factor, H-form	0.932
I_{Na}, fractional mass gain from H-form to Na-form	1.25

2.2 Crystalline Silicotitanate

Crystalline silicotitanate (CST) IE-911, Lot 2081000009, was obtained from UOP LLC, Des Plaines, IL. The UOP particle-size specification of this material was 30 to 60 mesh. The material was provided in the hydrogen form. A 5-g aliquot was converted to the Na form by soaking in 40 mL of 1 M NaOH for two days. The NaOH solution was then decanted, and the CST was rinsed with five successive volumes of DI water. The resin was then dried at 95°C overnight. The F-factor, 0.9708, was determined at the same time the batch-contact samples were weighed. This was obtained by drying approximately 0.5 g at 105°C to constant mass.

2.3 AN102/C-104 and AN-102 Feeds

The AN-102 sample receipt, homogenization, phase separation, subsampling, mixing with C-104 wash and permeate solutions, and analysis have been previously reported (Urie et al. 2002a). This material was then evaporated to nominally 5 M Na (Lumetta et al. 2002) and underwent Sr/TRU[b] precipitation and removal by filtration (Hallen et al. 2002). The total volume of AN-102/C-104 available for Cs ion exchange and batch-contact processing was about 788 mL. To conserve the AN-102/C-104 material for follow-on work, the IE-911 batch contacts were conducted with AN-102. The total volume of AN-102 available for batch contacts was 34 mL. Both feeds, AN-102/C-104 and AN-102, were analyzed, and the results are summarized in Table 2.3 (analytical details are provided in Appendix D). Generally, the AN-102 feed analytes were about a factor of 1.2 more concentrated than the AN102/C104 analytes. An exception is the ^{90}Sr that was removed from the AN-102/C104 combined feed before Cs ion exchange.

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

(b) TRU = transuranic

Table 2.3. Compositions of AN-102/C-104 and AN-102 (Envelope C)

Analyte	**AN-102/C-104**	**AN-102**
Solution density, g/mL	1.244	1.301
Cations, M		
Na^{+}	4.8 E+0	6.1 E+0
K^{+}	[2.4 E-2]	[2.8 E-2]
Cs^{+}(1)	5.99 E-5	7.70 E-5
Ca^{2+}	[3.7 E-3]	[4.7 E-3]
Cd^{2+}	2.31 E-4	2.86 E-4
Cu^{2+}	[1.6 E-4]	[1.3 E-4]
Ni^{2+}	3.20 E-3	4.06 E-3
Pb^{2+}	[3.2 E-4]	[4.2 E-4]
Sr^{2+}	9.87 E-4	6.98 E-4
Mole Ratios		
Na/Cs mole ratio	8.0 E+4	7.9 E+4
K/Cs mole ratio	4.0 E+2	3.6 E+2
Anions, M		
AlO_2^- (2)	3.04 E-1	2.46 E-1
Cl^{-}	5.39 E-2	NA
F^{-}	<2.3 E-1(3)	NA
CO_3^{2-} (hot persulfate method)	6.94 E-1	NA
CO_3^{2-} (furnace method)	4.75 E-1	NA
CrO_4^{2-} (2)	2.08 E-3	2.31 E-3
NO_2^-	9.23 E-1	NA
NO_3^-	1.66 E+0	NA
OH^{-}	2.0 E-1	NA
PO_4^{3-} (2) (ICP-AES)	2.41 E-2	1.47 E-2
PO_4^{3-} (IC)	3.63 E-2	NA
SO_4^{2-}	6.84 E-2	NA
Uranyl	1.15 E-4	NA
Oxalate	3.4 E-2	NA
TOC, g/L (hot persulfate method)	1.19 E+1	NA
TOC, g/L (furnace method)	1.89 E+1	NA
Radionuclides, µCi/mL		
Total alpha	1.26 E-2	NA
^{60}Co	4.06 E-2	5.48 E-2
^{90}Sr	1.70 E+0	1.40 E+1
^{99}Tc	[7.71 E-2](4)	NA
^{137}Cs	1.61 E+2	2.07 E+2
^{154}Eu	< 6 E-2	< 2 E-1

(1) The Cs concentration was calculated from the ^{137}Cs concentration measured by gamma energy analysis (GEA), and the isotopic ratio was measured by thermal ionization mass spectrometry (TIMS).

(2) Al, Cr, and P were determined by inductively-coupled plasma atomic emission spectrometry (ICP-AES). The anionic form was assumed on the basis of waste chemistry.

(3) F concentration is an upper bound; coeluting anions positively interfered with peak integration.

(4) Uncertainty, including standards bias, ranged from -10% to +30%.

NA = not analyzed

Bracketed results indicate that the analyte concentration uncertainty exceeded 15%.

The aluminum concentration was higher in the AN-102/C-104 feed than in the AN-102 feed, most probably from caustic leaching of the C-104 solids. The measured AN-102 feed composition generally agreed within 10% of the AN-102 composition previously reported (Hassan et al. 2000); the AN-102/C-104 composition generally agreed within 30% with the published composition, with the exception of OH. Hassan et al. reported 1.69 M OH, eight times the OH concentration found in AN-102/C-104.

Carbonate and total organic carbon (TOC) are reported here and elsewhere for two different analytical methods: hot persulfate oxidation and furnace oxidation. The differences in results of the two methods reflect the ease or difficulty with which various organic constituents oxidize in the given method.

2.4 Batch Contacts

The batch contacts were performed with the H-form of SL-644 batch 010319SMC-IV-73, 212- to 425-μm dry particle size.[(c)] The Na-form of IE-911 was tested in parallel with the SL-644. Batch contacts were performed using feed at three different Cs concentrations. Aliquots of tank-waste samples were tested without spiking, and additional aliquots were spiked with 0.75 M $CsNO_3$ to obtain stock solutions of nominally 1 E-3 M and 5 E-3 M Cs. The initial Cs concentrations in the stock contact solutions and the corresponding Na/Cs and K/Cs mole ratios are given in Table 2.4.

Table 2.4. Initial Cs Concentrations in the AN-102/C-104 Solutions Used for the Batch K_d Tests

Solution	Target Initial Cs Conc. [M]	Target Nominal Na/Cs$^{(1)}$mole ratio	Target Nominal K/Cs$^{(1)}$ mole ratio
SL-644 Batch Contact with AN-102/C-104			
Un-spiked	6.0 E-5	8.0 E+4	4.0 E+2
Cs Spike 1	1.0 E-3	5.0 E+3	2.4 E+1
Cs Spike 2	5.0 E-3	1.0 E+3	4.8 E+0
IE-911 Batch Contact with AN-102			
Un-spiked	7.7 E-5	7.9 E+4	3.6 E+2
Cs Spike 1	1.0 E-3	6.0 E+3	2.8 E+1
Cs Spike 2	5.0 E-3	1.2 E+3	5.6 E+0
(1) Na^+ and K^+ are the primary cations that compete with Cs^+ for ion exchange with SL-644.			

The batch K_d tests were performed in duplicate 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 feed. The exchanger mass was determined to an accuracy of 0.0002 g. The waste volume was transferred by pipet, and the actual volume was determined by mass difference with an accuracy of 0.0002 g and the solution density. An orbital shaker provided agitation for approximately 24 h for SL-644 and 72 h for IE-911.[(d)] The temperature was not controlled but was generally constant at 25 to 29°C during the 3 days of contact.

(c) Batch-contact testing was conducted according to, and documented in, Test Instruction TI-RPP-WTP-082, Rev. 0, *Batch Contact of AP-101 and AN-102/C104 Waste with SuperLig 644 (Batch ID 010319SMC-IV-73) and CST*, S. K. Fiskum, June 2001.

(d) The 24-h contact time for SL-644 was defined in the test specification. The 72-h contact time for IE-911 was applied based on the results of Brown et al. 1996.

All Cs K_d measurements were determined by measuring ^{137}Cs on both the stock solution (initial concentration) and the contacted solution (final concentration). Initial ^{133}Cs concentrations were confirmed by inductively-coupled plasma-mass spectrometry (ICP-MS). The IE-911 batch-contact samples were also measured for ^{90}Sr to evaluate Sr K_ds.

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

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

where C_0 = the initial ^{137}Cs concentration
C_1 = the final ^{137}Cs concentration
V = the volume of the liquid sample (mL)
M = the mass of the ion exchanger (g) (SL-644 H-form mass corrected for the Na-form mass increase or Na-form IE-911 mass)
F = the dried resin mass divided by the initial resin mass.

For the Sr K_d determination, C_o and C_1 are the initial and final ^{90}Sr concentrations.

The SL-644 dry-bed resin density, ρ (g of resin per mL of resin in contact with solution), was obtained according to the following equation:

$$\rho = \frac{m * L * F * I_{Na}}{BV} \tag{2}$$

where m = resin mass in the ion exchange column, as-received form
L = fractional mass remaining after washing (0.538)
F = water-loss factor, as-received form (0.877)
I_{Na} = fractional mass gain on conversion from H-form to Na-form (1.25) (this factor is set to 1 when calculating the dry-bed density in the H-form or 0.5 M HNO_3 feed)
BV = resin bed volume (BV) in the feed (discussed in Section 3.2.5).

The dry-bed density for IE-911 was calculated according to the following equation:

$$\rho = \frac{m * F}{BV} \tag{3}$$

where m = resin mass
F = water loss factor
BV = resin BV in AW-101 simulant.

The Cs λ value (column-distribution ratio) is a function of the dry-bed density and is obtained as shown in Equation 4.

$$\lambda = K_d * \rho \tag{4}$$

2.5 Experimental Conditions for SL-644 Column Ion Exchange Test

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

The columns were prepared at the SRTC Glassblowing Laboratory. Each column consisted of a 15-cm glass column with a 24/40 taper ground-glass fitting on top and a threaded fitting on the bottom. A polyethylene bushing was installed in the glass-threaded fitting to accommodate a ¼-in stainless steel National Pipe Thread (NPT) fitting. The inside diameter of each column was 1.46 cm, which corresponded to a volume of 1.67 mL/cm. A stainless steel, 200-mesh screen supported the resin bed. The height of the resin bed (and thus shrinkage and swelling) was measured with a decal millimeter scale affixed to the column. The upper section contained four entry ports and a taper joint with a screw cap that securely fitted the column. The lead column assembly used a pressure relief valve (10 psi trigger), pressure gauge, and sample inlet; the remaining port was plugged. The lag column assembly used one port for sample entry, and the other three ports were plugged. In both columns, the inlet sample lines extended through the port opening to the top of the column. The connecting tubing was $^1/_8$-in. OD, $^1/_{16}$-in. ID polyethylene. Valved quick-disconnects (Cole Parmer) were installed in-line to allow for ease of column switching. An FMI QVG50 pump (Fluid Metering, Inc., Syosset, NY) equipped with a ceramic and Kynar® coated low-flow piston pump head was used to introduce all fluids. The flowrate was controlled with a remotely operated FMI stroke-rate controller. The pump was calibrated with the stroke-rate controller and could provide pumping rates from 0.08 to16 mL/min. The volume actually pumped was determined using the mass of the fluid and the fluid density. The pressure indicated on the pressure gauge remained below 5 psi during all runs. The total holdup volume of the Cs ion exchange system was the summed volume of all fluid-filled parts and was estimated to be 42 mL.

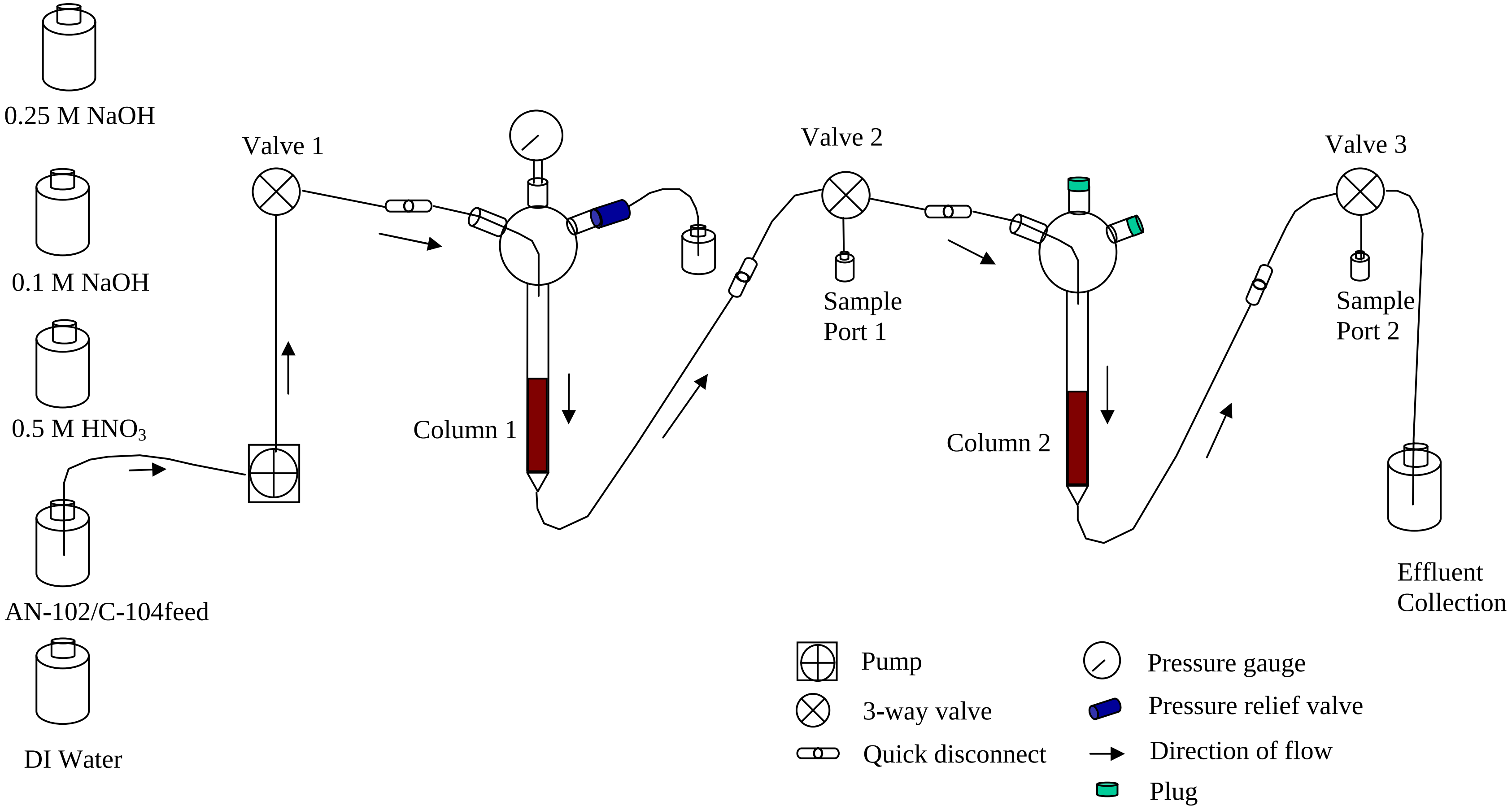


Figure 2.1. Cesium Ion Exchange Column System

Before installing the system into the hot cell, both of the resin beds were individually cycled through the acid form. After the resin cycling, the corrected mass of the Na-form SL-644 was calculated to be 2.4 g, on a dry-weight basis, in each column according to the following equation:

$$M_{bed} = M_{AR} * F_{AR} * L * I_{Na} \quad (5)$$

where M_{bed} = the resin bed mass in the Na-form on a dry-weight basis
M_{AR} = the resin mass loaded in the column, as-received form
F_{AR} = the F-factor for the as-received resin (0.877)
L = the fractional mass remaining after conversion to the H-form (0.538)
I_{Na} = the fractional mass gain on conversion from the H-form to the Na-form (1.25).

The entire ion exchange system was then used for a full shakedown experiment with AW-101 simulant (Fiskum, Blanchard, and Arm 2002). Both columns were individually eluted, rinsed, and regenerated. The ion exchange system was then used to process AP-101 diluted feed (AP-101DF) tank waste (Fiskum et al. 2002). Only the lead column was eluted to a ^{137}Cs concentration C/C_o of 4 E-3, which was equivalent to a ^{137}Cs concentration of 0.5 μCi/mL. The lead column was then rinsed with DI water, regenerated with 0.25 M NaOH, and again rinsed with DI water. The lag column contained an estimated 35 μCi ^{137}Cs from the lead column Cs breakthrough (Fiskum et al. 2002). The beds had been stored approximately 8 weeks in the Na-form in DI water since the end of the AP-101DF column run.

The lead and lag columns were switched, and the apparatus volume (AV) of DI water was displaced with 0.25 M NaOH before introducing AN-102/C-104 feed. All subsequent processing(a) was performed in the hot cells at temperatures ranging from 25 to 29°C. The experimental conditions for each process step are shown in Table 2.5, where one BV is the volume in 0.25 M NaOH, 10.4 mL. The bed conditioning, AN-102/C-104 loading, feed displacement, and DI water-rinse steps were conducted by passing these solutions through both resin beds connected in series. The AN-102/C-104 effluent was collected in two effluent bottles. The first bottle collected 41 mL, nominally one AV, and consisted primarily of the displaced regeneration solution. The remaining effluent was collected as one fraction. The initial 43-mL (1 AV) of feed-displacement solution was collected in the AN-102/C-104 effluent composite bottle. This allowed maximum recovery of the AN-102/C-104 feed and minimized loss associated with feed-displacement sampling. Sampling of the feed-displacement solution began immediately after the 1-AV displacement. The elution was conducted on the lead column only, continuing until 16.8 BV had been processed through the column. The ion exchange system was idled at this point overnight for 13.5 h in 0.5 M HNO_3. Then two additional BVs of 0.5 M HNO_3 were passed through the column. The resin bed was then rinsed, regenerated, and rinsed again as shown in Table 2.5.

(a) Processing and data collection were recorded in Test Instruction TI-PNNL-WTP-080, Rev. 0, “Separation of Cesium from Hanford Tank Waste AN-102/C-104 Wash Solution Integrated Test Sample Using the Dual Small-Column SuperLig 644 Cesium Ion Exchange System,” SK Fiskum, 6/21/02.

Table 2.5. Experimental Conditions for AN-102/C-104 Ion Exchange

Process step	**Solution**	**Total Volume**			**Flowrate**		**Time, h**	**T, °C**
		BV(1)	**AV(2)**	**mL**	**BV/h**	**mL/min**	**h**	
Two columns in series								
DI water displacement	0.25 M NaOH	8.0	2.0	83	2.7	0.47	2.9	27
Loading Lead column(3)	AN-102/ C-104 Feed	72.4	-	753	2.7	0.47	27.2	28
Loading Lag column(4)	AN-102/ C-104 Feed	70.8	-	736	2.7	0.47	27.2	28
Feed displacement	0.1 M NaOH	11.6	2.9	121	2.8	0.48	4.6	28
DI water rinse	DI water	8.2	2.0	86	2.7	0.47	3.2	28
Lead column only								
Elution	0.5 M HNO_3	18.9	-	197	0.95	0.16	33.7(5)	28
Eluant rinse	DI water	5.9	2.7	62	2.8	0.49	2.1	28
Regeneration	0.25 M NaOH	4.9	2.2	51	0.92	0.16	5.5	32
Rinse	DI water	4.6	2.1	48	2.6	0.45	2.0	32

(1) BV = bed volume (10.4 mL in 0.25 M NaOH regeneration condition)
(2) AV = apparatus volume (42 mL for columns in series; 23 mL for lead column and 21 mL for lag column)
(3) Ion exchange run began on 6/26/01.
(4) The feed volume through the lag column is reduced because of sampling from the lead column.
(5) The elution time includes 13.5 h when the system was idled in 0.5 M HNO_3 overnight.

2.6 Column Sampling

The sampling and analysis protocol is shown in Table 2.6. During the loading phase, small samples (about 2 mL) were collected from the lead and lag columns starting at 5 BVs, then at 13 BVs and continuing in nominal 10-BV increments of feed. The flowrate varied between 2.6 and 2.9 BV/h. The flowrate doubled during sample collection from the lead column because of the strong siphoning effect at the sample port. Thus, for the eight samples collected from the lead column and for 2 min for each sample, AN-102/C-104 fed at a nominal flowrate of 6 BV/h. The feed displacement, DI water rinse, elution, and elution-rinse samples were taken at 1-BV increments at flowrates shown in Table 2.5.

Table 2.6. Sampling Interval and Analyses

Process Step	Frequency Lead Column BV	Frequency Lag Column BV	Approximate Sample Size (mL)	Analyses
Loading	Every 10 BV	Every 10 BV	2	GEA
Feed displacement	—	Every 1 BV	10	GEA
DI water rinse	—	Every 1 BV	10	GEA
Elution	Every 1 BV	—	10	GEA
Eluant rinse	Every 1 BV	—	10	GEA
Composite Samples				
Effluent – 1	—	—	2	GEA
Effluent composite	—	—	10	GEA, ICP-AES, IC, U, ICP-MS (Tc), pertechnetate, OH, TOC/TIC
Regeneration	1 composite	—	10	ICP-AES, GEA, OH-
Eluate	1 composite	—	10	ICP-AES, GEA, TOC, IC, U, TIMS, and various radioisotopes

GEA = gamma energy analysis
ICP-AES = inductively coupled plasma atomic energy spectrometry
IC = ion chromatography
ICP-MS = inductively coupled plasma mass spectrometry
OH = hydroxide
TOC = total organic carbon
TIC = total inorganic carbon
TIMS = thermal ionization mass spectrometry

2.7 Sample Analysis

The ^{137}Cs concentration was determined using a benchtop GEA spectrometer, which allowed for rapid sample analysis. Selected sample results were later confirmed by GEA analysis in the Chemical Measurements Center (CMC) analytical laboratory. The effluent composite sample was submitted for various analyses: GEA, ICP-AES, TOC/TIC, IC, ^{99}Tc, pertechnetate, U, and OH^-. The eluate samples required dilution before removal from the hot cell to reduce the dose rate from ^{137}Cs. The extent of dilution was determined by mass difference. Once the GEA results were confirmed for the eluate samples, the eluate samples were composited,(b) and a sample of the composite was submitted for various analyses: GEA, ICP-AES, TOC, IC, U, ^{90}Sr, total alpha, and thermal ionization mass spectrometry (TIMS) for Cs isotopic distribution. A sample of the regeneration solution was taken for ICP-AES and OH^- determination.

The Na and other metal concentrations were determined with ICP-AES. The OH^- concentration was determined by potentiometric titration with standardized HCl. Uranium was determined using kinetic phosphorescence. The total Tc concentration was determined by ICP-MS, and the pertechnetate concentration was determined using radiochemical separations specific for pertechnetate followed by beta counting. Anions were determined using ion chromatography. TOC and TIC were determined by silver-catalyzed hot-persulfate oxidation and furnace oxidation methods.

(b) Compositing was conducted according to Test Instruction TI-RPP-WTP-106, Rev. 0, “Preparing a Composite Solution of the Acid Eluate Samples from AN-102/C-104 Cs Ion Exchange Column 1,” SK Fiskum, 9/4/01.

3.0 Results and Discussion

This section describes the results from batch-contact tests and the column test with SL-644.

3.1 Batch Contact Results

The K_d values were calculated using Equation 1 and are based on ^{137}Cs concentrations as measured by GEA. The Na/Cs mole ratios were calculated based on the measured Na and total Cs concentrations in the uncontacted AN-102/C-104 and AN-102. The equilibrium Cs concentration was based on the ^{137}Cs concentrations and the ratio of ^{137}Cs:total Cs determined for the unspiked and spiked solutions. The Na concentration was assumed to be constant for the batch contacts. Since the quantity of H^+ added with the resin was small relative to the moles of Na^+ and OH^- in the contact solution (phase ratio of 100 mL of solution:gram of exchanger), this was a reasonable assumption. In these experiments the waste solutions were estimated to have 1 meq of OH^- and 24 meq of Na^+, while the resin contained 0.1 meq of H^+ for the 0.05 g resin used in the batch distribution tests.(a)

The calculated ^{137}Cs distribution coefficients (K_ds) are plotted in Figure 3.1 for SL-644 in contact with AN-102/C-104 and IE-911 in contact with AN-102. At the nominal feed condition of 8 E+4 Na/Cs mole ratio, the SL-644 and IE-911 have nearly equivalent K_d values of 950 and 1100 mL/g, respectively.

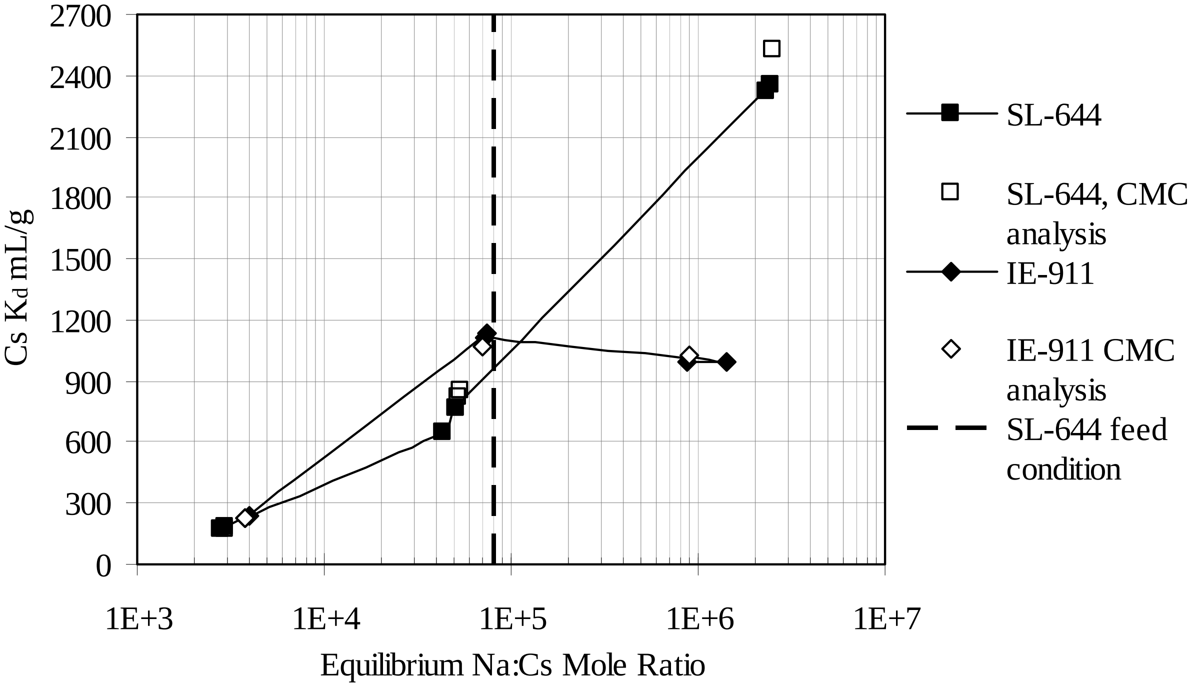


Figure 3.1. ^{137}Cs Distribution Coefficients (K_d) for SL-644 (AN-102/C-104) and IE-911 (AN-102) (T = 27 °C)

(a) The resin contained 2.2 meq H^+ per gram of H-form resin (Rapko et al. 2002).

The calculated dry-bed densities determined using the resin masses and volumes in the ion exchange column system (discussed in Section 3.2.5) are given in Table 3.1. The Na-form resin mass was corrected as defined in Equation 5. Good agreements of the dry-bed densities were obtained between the lead and lag columns. The AN-102/C-104 form dry-bed density was approximately equivalent to the 0.5 M HNO_3 form dry-bed density. In this case, the decrease in mass associated with the change from the Na-form to the H-form was approximately equivalent to the decrease in volume upon contracting from the Na-form to the H-form.

Table 3.1. SL-644 Dry Bed Density

Property	**010319SMC-IV-73, Lead Column**	**010319SMC-IV-73, Lag Column**
Column resin mass, g(1)	4.20	4.17
Corrected column Na-form resin mass, g	2.48	2.46
Bed volume, 0.25 M NaOH, mL	10.4	10.5
Bed volume, AN-102/C-104, mL	9.9	9.9
Bed volume, 0.5M HNO_3	7.7	not performed
Dry bed density, ρ_b, in given matrix		
Na-form resin		
0.25 M NaOH, g/mL	0.238	0.234
AN-102/C-104, g/mL	0.250	0.248
H-form resin		
0.5 M HNO_3, g/mL	0.258	not performed
(1) The 212- to 425-µm particle-size resin mass measured in the as-received form.		

The Cs λ value for SL-644 was calculated according to Equation 4 to be 250 BVs in the AN-102/C-104 feed condition, and 240 BVs in the 0.25 M NaOH regeneration condition. This value is the approximate point at which the Cs breakthrough curve is predicted to pass through $C/C_o = 50\%$ if sufficient feed were available for processing. There was insufficient AN-102/C-104 available to determine the IE-911 bed density in the feed. The dry-bed density was determined in AW-101 simulant to be 1.04 g/mL. Assuming that the bed density of IE-911 is constant in these matrices, the Cs λ value for IE-911 was estimated to be 1100 BVs. Figure 3.2 shows the Cs λ values as a function of Na/Cs mole ratio for the IE-911 (assumed bed density of 1.04 g/mL, dried to 105 ^{o}C) and the SL-644 (calculated bed density in 0.25 M NaOH of 0.25 g/mL, dried at ambient temperature under vacuum).

Strontium-90 was also measured in the IE-911 batch-contacted AN-102 aqueous fractions to evaluate Sr K_ds. The ^{90}Sr K_ds were fairly constant, ranging from 35 to 53 mL/g for the range of Na/Cs mole ratios tested. These results were much lower than previously reported results for IE-911 (Brown et al. 1996), where Sr K_ds >1E+6 mL/g were obtained using an AW-101 simulant (no added complexants). The difference was probably due to the presence of complexants in the AN-102/C-104 waste (Urie et al. 2002b) that may bind with the Sr and inhibit the ion exchange process.

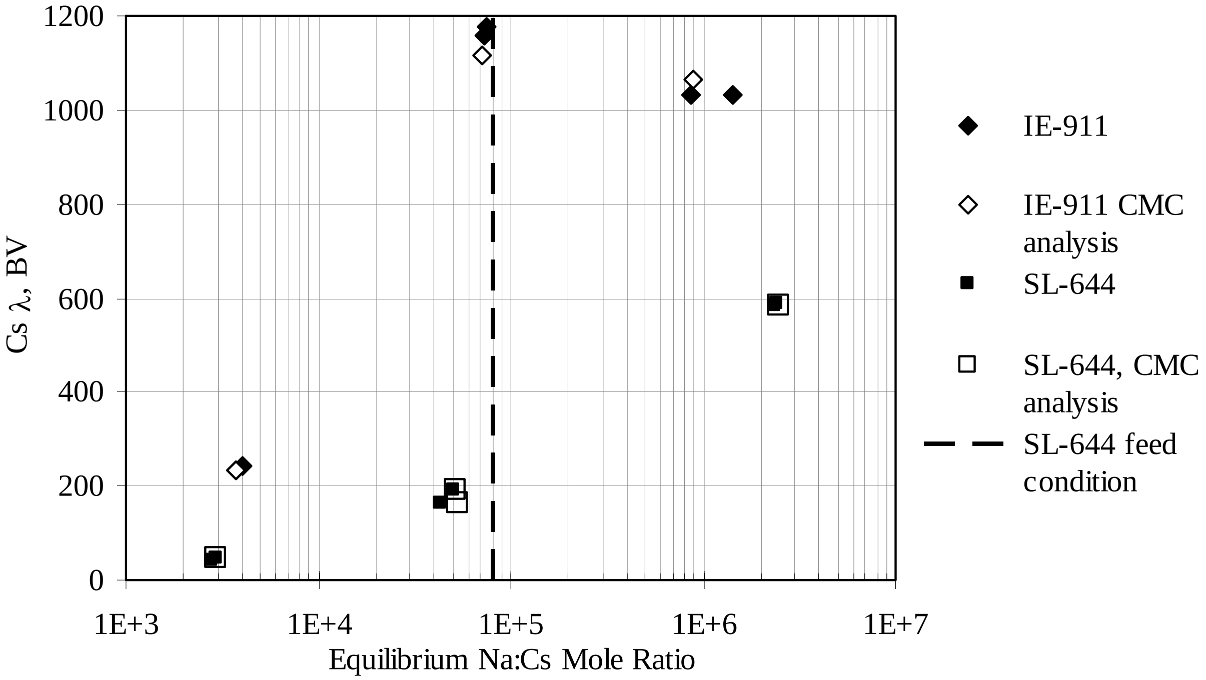


Figure 3.2. Estimated ^{137}Cs λ Values for SL-644 (AN-102/C-104) and IE-911 (AN-102) (T = 26°C)

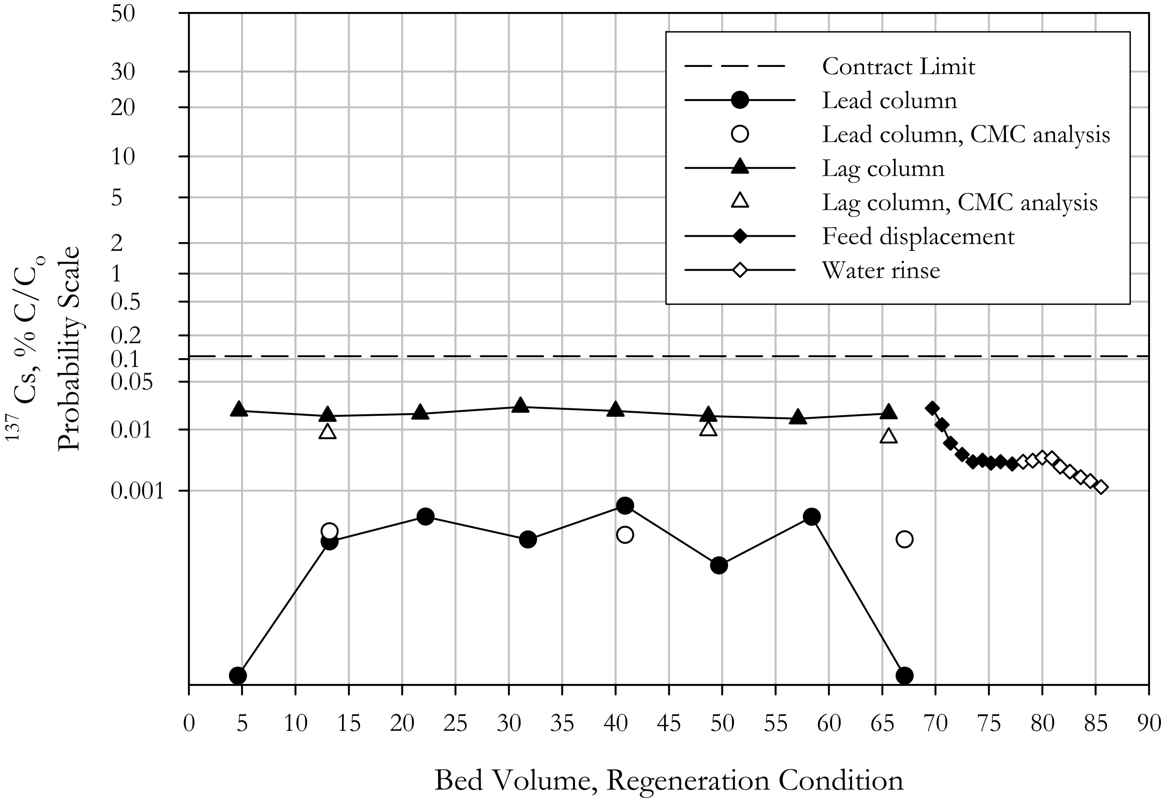
3.2 Column Test with SL-644

The column system and resin beds used for AN-102/C-104 processing were also used for processing the AW-101 simulant and the AP-101DF actual waste. This test represented the third complete cycle using these resin beds. No resin fouling was observed through all ion exchange processing.

3.2.1 Loading, Feed Displacement, and Rinse

The loading phase was initiated with AN-102/C-104. Approximately 1 AV (41 mL) of effluent was initially collected in a separate collection bottle. Most of this effluent was 0.25 M NaOH from the regeneration step mixed with some AN-102/C-104 in the ion exchange apparatus. This solution was maintained separately and not mixed with the final Cs-decontaminated AN-102/C-104 effluent composite; thus, most of the apparatus 0.25 M NaOH solution was prevented from mixing with the AN-102/C104 effluent. The resin beds shrank an average 5.6% to 9.9 mL as they converted from the regeneration solution to the AN-102/C-104 feed.

The effluent Cs concentrations are shown in Figure 3.3 as % C/C$_o$ vs. the BVs of feed processed through each column. The abscissa reflects BVs as a function of the resin in the expanded regeneration condition of 10.4 mL. The C$_o$ value for ^{137}Cs was determined to be 161 µCi/mL. The % C/C$_o$ is plotted on a probability scale as this scale tends to provide a straight-line breakthrough curve. The C/C$_o$ values, determined using an in-house GEA spectrometer, were generally in good agreement with selected samples independently analyzed by the CMC analytical laboratory. Raw analytical results and calculations are located Appendix C.



Conditions: SL-644 batch number 010319SMC-IV073 212- to 425-μm dry particle size
Process temperature = 27 °C
Bed volume in 0.25 M NaOH feed condition = 10.4 mL
Flowrate = 2.7 BV/h
^{137}Cs C_o = 161 μCi/mL
Na concentration = 4.8 M.

Figure 3.3. ^{137}Cs Breakthrough Curves for AN-102/C-104 Sample

There was insufficient feed available to result in any Cs breakthrough from the lead column.(b) Thus the Cs λ value (the point at which the C/C_o value is 50%) could not be estimated or compared with the Cs λ value predicted from batch-contact studies. This load profile is different than that reported by Hassan et al. (2000), where breakthrough was found within 5 BV, and at 60-BV loading they observed 4.5% breakthrough on the lead column. Differences in the load profiles may be attributable in part to the differences in feed composition, the differences in resin particle-size distributions, differences in specific characteristics of the two SL-644 production batches, and differences in the storage and process history of the resins.

(b) Only 753-mL or 72.4 BVs AN-102/C-104 were available; an estimated 2500 mL or 240 BVs were necessary to reach 50% C/C_o.

The ^{137}Cs concentrations in the lag column samples were clearly higher (by a factor of 50) in activity than those of the lead column. This was consistent with ^{137}Cs "bleed-off" from the previous AP-101DF ion exchange processing where the Cs elution ended at a ^{137}Cs concentration of 0.5 μCi/mL. The effluent ^{137}Cs concentration averaged 1.4 E-2 μCi/mL over the entire AN-102/C-104 processing period. Thus a total of 10.3 μCi ^{137}Cs were removed from the lag column as bleed-off and collected in the 736-mL AN-102/C-104 effluent. This is equivalent to 0.007 % of the total ^{137}Cs loaded during the AP-101DF processing, where AP-101DF loading continued through 112 BV and a lead column C/C_o of 0.27 %. The plant-design loading was expected to continue to about 190 BV to a C/C_o of 50 %. The possibility exists that Cs bleed-off during AN-102/C-104 processing could be higher if more AP-101DF feed could have been processed. With plant-design operation continuing through only 74-BV processing, the possibility exists that the subsequent Cs bleed-off during subsequent waste processing could be lower than observed.

The bleed-off was constant over the course of the loading phase, dropping sharply with the introduction of the feed displacement and DI water rinse. The drop in Cs bleed-off is related to the drop in Na concentration in the feed, which acts as a competing ion to the Cs on the ion exchanger.

The contract ^{137}Cs removal limit is also shown in Figure 3.3. The C/C_o value of 0.104% corresponds to the contract limit of 0.3 Ci/m^3 for ^{137}Cs in the LAW glass. The C/C_o value corresponding to this limit was determined using the Na concentration of 4.8 M in the AN-102/C-104, the ^{137}Cs feed concentration of 161 μCi/mL, a 10 wt% total Na_2O loading in the glass, and a glass-product density of 2.66 g/mL.[c] Despite the ^{137}Cs bleed-off from the lag column, the effluent ^{137}Cs concentration remained below the contract limit.

The decontamination factors (DFs) were calculated on selected effluent samples and the composite effluent, and are summarized in Table 3.2. These may be compared to the contract limit of C/C_o = 0.104% (DF = 960). Sufficient Cs was removed so that the contract limit was met.

Table 3.2. Decontamination Factors for ^{137}Cs from AN-102/C-104

Sample	**volume, BV (mL)**	**^{137}Cs Concentration μCi/mL$^{(1)}$**	**C/C_o, %**	**DF**
Second lead column sample	13.2 (137)	2.95 E-4	1.83 E-4	5.5 E+5
Final lead column sample	67.1 (697)	2.10 E-4	1.30 E-4	7.7 E+5
Second lag column sample	13.0 (135)	1.40 E-2	8.70 E-3	1.2 E+4
Final lag column sample	65.6 (682)	1.21 E-2	7.52 E-3	1.3 E+4
Composite effluent	72.4 (753)	1.42 E-2	8.82 E-3	1.1 E+4
(1) The ^{137}Cs uncertainty ranges from 3% to 8% relative error, 1-σ.				

The Cs-decontaminated effluent was characterized, and the results are summarized in Table 3.3. Major analyte concentrations were equivalent to the feed concentrations within the experimental error of the method (typically ±15%). Uranyl and Cu have been notably removed from the tank waste along with the Cs. Oxalate, Ni, PO_4, and ^{60}Co concentrations were also lower in the LAW product than the feed.

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[c] The maximum Envelope C waste Na_2O loading is 14 wt%, corresponding to a C/C_o of 0.074%.

Table 3.3. AN-102/C-104 Cs-Decontaminated Effluent

Analyte	**AN-102/C-104, Effluent M**	**% Change from Feed**
Solution density, g/mL	1.237 T=24°C	-0.6
Cations, M		
Na^+	4.78 E+0	-1.0
K^+	2.76 E-2	13.6
Cs^+	5.28 E-9	>-99
Ca^{2+}	3.72 E-3	0.5
Cd^{2+}	2.15 E-4	-13.0
Cu^{2+}	< 1 E-5	[>-95]
Ni^{2+}	2.47 E-3	-22.8
Pb^{2+}	3.33 E-4	6.1
Sr^{2+}	9.92 E-4	0.5
Anions, M		
$AlO_2^{-\ (1)}$	2.91 E-1	-4.3
Cl^-	5.08 E-2	-5.8
$F^{-\ (2)}$	< 3.3 E-1	NA
CO_3^{2-} (hot persulfate method)	7.6 E-1	9.2
CO_3^{2-} (furnace method)	Indeterminate$^{(3)}$	NA$^{(3)}$
$CrO_4^{2-\ (1)}$	1.89 E-3	-9.1
NO_2^-	9.00 E-1	-2.2
NO_3^-	1.73 E+1	4.0
OH^-	1.80 E-1	-10.0
$PO_4^{3-\ (1)}$ (ICP-AES)	1.81 E-2	-25.2
$PO_4^{3-\ (1)}$ (IC)	2.32 E-2	-36.1
SO_4^{2-}	6.97 E-2	1.9
Uranyl	3.45 E-6	-97.0
Oxalate	1.59 E-2	-53.2
TOC, g/L (hot persulfate method)	12.3	3.4
TOC, g/L (furnace method)	21.2	12.2
Radionuclides, μCi/mL		
Total alpha	1.10 E-2	-12.7
^{60}Co	3.41 E-2	-16.0
^{90}Sr	1.52 E+0	-10.6
^{99}Tc	[7.95 E-2]$^{(4)}$	3.1
^{137}Cs	1.40 E-2	>-99
^{154}Eu	2.20 E-2	NA

(1) Al, Cr, and P determined by ICP-AES. The anionic form is assumed on the basis of waste chemistry.

(2) F concentration is an upper bound; coeluting anions positively interfered with peak integration.

(3) The furnace method TIC could not be determined. The total carbon (TC) was equivalent to the TOC and TIC = TC-TOC.

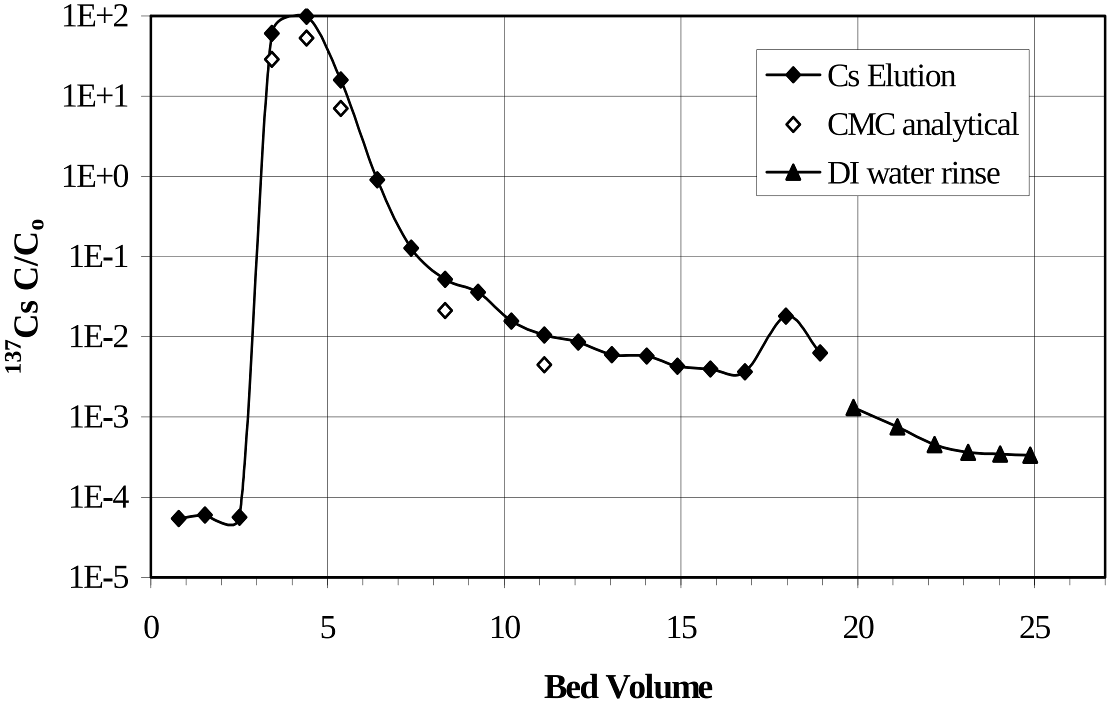
(4) Uncertainty, including standards bias, ranged from -10% to +30%.

NA = not analyzed

Bracketed results indicate the analyte concentration uncertainty exceeds 15%. Less-than results indicate that the analyte concentration was below the instrument detection limit (IDL); the dilution-corrected IDL is given.

3.2.2 Elution and Eluant Rinse

The lead column elution and eluant rinse steps ^{137}Cs C/C$_o$[(d)] values are shown in Figure 3.4. The ordinate is a logarithmic scale to clearly show the large range of C/C$_o$ values obtained. The abscissa is given in BVs relative to the regeneration condition. Most of the ^{137}Cs was contained in elution BVs 3 to 5. The peak value of C/C$_o$ was found to be 53. The elution cutoff of C/C$_o$ = 0.01 was reached at 11 BVs but elution was continued beyond this because of the lag between sample collection and the determination of the Cs concentration. The relative C/C$_o$ increased at 18 BVs. The ion exchange system was idled at this point overnight for 13.5 h in 0.5 M HNO_3. During this time, additional Cs exchanged out of the SL-644, indicating that previous eluate samples from the elution curve tail were not in equilibrium with the Cs still loaded on the resin. Thus, the increase in C/C$_o$ is attributed to a slight non-equilibrium condition during the elution process, not a change in elution kinetics. The C/C$_o$ values for the eluant rinse with DI water drop rapidly in ^{137}Cs concentration, indicating that DI water does not continue Cs elution.



Conditions: SL-644 batch # 010319SMC-IV-073 212- to 425-μm dry particle size
Bed volume in the 0.25M NaOH regeneration condition = 10.4 mL
Flowrate = 0.95 BV/h Process temperature = 28 °C
^{137}Cs C$_o$ = 161 μCi/mL Eluant = 0.5 M HNO_3.

Figure 3.4. ^{137}Cs Elution and Eluant Rinse of the Lead Column

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(d) The C$_o$ refers to the ^{137}Cs concentration in the AN-102/C-104 sample fed to the lead column. For elution, the C/C$_o$ 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.

The eluate samples from the lead column were composited, and a sub-sample was taken for analysis. The analytical results are shown in Tables 3.4 and 3.5. Sodium was the dominant component detected using ICP-AES with some Cd, Cr, Cu, Fe, Ni, Pb, and U eluting as well. A summary of these analyte

Table 3.4. Inorganic and Organic Analytes in the Lead Column Eluate Composite

Analyte	**Method**	**MRQ(1) µg/mL**	**Cs eluate(2) µg/mL**	**Analyte**	**Method**	**MRQ(1) µg/mL**	**Cs eluate(2) µg/mL**
Al	ICP-AES	75	[5.8]	U	KPA	600	167
Ba	ICP-AES	2.3	[0.18]	Zn	ICP-AES	17	[2.8]
Ca	ICP-AES	150	< 3	Cl^-	IC	3	22
Cd	ICP-AES	8	2.35	F^-	IC	150	< 13
Co	ICP-AES	30	< 0.5	NO_3^-	IC	3000	27,800
Cr	ICP-AES	15	23	PO_4^{-3}	IC	2500	< 25
Cs(4)	GEA	1.5	33.2	SO_4^{-2}	IC	2300	< 25
Cu	ICP-AES	17	[31]	TOC	Hot Pers.	1500	130
Fe	ICP-AES	150	[6.4]	TOC	Furnace	1500	< 100
K	ICP-AES	75	< 20	Glycolate	Organic IC	1500	<150
La	ICP-AES	35	< 0.5	Acetate	Organic IC	1500	<120
Mg	ICP-AES	300	< 1	Formate	Organic IC	1500	<200
Mn	ICP-AES	150	< 0.5	Oxalate	Organic IC	1500	<260
Mo	ICP-AES	150	< 0.5	Citrate	Organic IC	1500	<510
Na	ICP-AES	75	790	EDTA	GC/FID	1500	<5
Ni	ICP-AES	30	68	HEDTA	GC/FID	1500	<9
Pb	ICP-AES	300	25	ED3A	GC/FID	1500	<5
Si	ICP-AES	170	[20]	NTA	GC/FID	1500	<6
Sn	ICP-AES	1500	< 15	NIDA/IDA(5)	GC/FID	1500	<11
Sr	ICP-AES	NMRQ(3)	[1.1]	Citric acid	GC/FID	1500	<6
Ti	ICP-AES	17	< 0.3	Succinic acid	GC/FID	1500	19
U	ICP-AES	600	[175]				

EDTA = ethylenediaminetetraacetic acid; HEDTA = N-(2-hydroxyethyl ethylenediaminetriacetic acid
ED3A = ethylenediaminetriacetic acid; NTA = nitrilotriacetic acid; IDA = iminodiactic acid
NIDA = nitrosoiminodiacetic acid

(1) MRQ is minimum reportable quantity requested by Bechtel.

(2) The 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%. Less-than values indicate that the analyte was not detected by the instrument, and the reported value represents the IDL multiplied by the sample dilution factor.

(3) NMRQ is no minimum reportable quantity requested.

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

(5) The IDA was completely converted to NIDA in the presence of nitrite in tank waste.

Table 3.5. Radionuclides in the Lead Column Eluate Composite

Analyte	Method	MRQ$^{(1)}$ μCi/mL	Cs eluate μCi/mL$^{(3)}$	Error %	Analyte	Method	MRQ$^{(1)}$ μCi/mL	Cs eluate μCi/mL	Error %
^{60}Co	GEA	NMRQ$^{(2)}$	< 7 E-3	–	^{152}Eu	GEA	NMRQ	< 9 E-3	–
^{90}Sr	Radchem	1.50 E-1	7.8 E-2	50	^{154}Eu	GEA	2.00 E-3	< 8 E-3	–
^{126}Sn/Sb	GEA	NMRQ	< 7 E-2	–	^{155}Eu	GEA	9.00 E-2	< 3 E-1	–
^{134}Cs	GEA	NMRQ	< 7 E-3	–	^{241}Am	Radchem	7.20E-04	1.18 E-3	3
^{137}Cs	GEA	5.00 E-2	6.65 E+2	3	Total alpha	Radchem	2.30E-01	< 2 E-2	–

(1) MRQ is minimum reportable quantity requested by Bechtel.
(2) NMRQ is no minimum reportable quantity requested.
(3) Less-than values indicate that the analyte was not detected by the instrument, and the reported value represents the IDL multiplied by the sample dilution factor.

recoveries is presented in Table 3.6. The U recovered in the eluate (~33 mg) represented 160% of the U present in the AN-102/C-104 feed (21 mg). The AP-101DF processing apparently loaded some U on the lag column. The total U loaded with the AP-101DF was 48 mg, and the amount recovered in the eluate was 42 mg. The difference of 6 mg U was applied to the lag column U loading. Switching this column into the lead position for AN102/C-104 processing resulted in a partially U-loaded lead column. Thus, the total U recovered in the eluate (33 mg) represented 123% of the total U loaded on the lead column (6 mg from AP-101DF processing plus 21 mg from AN-102/C-104 processing). The apparent Cu loading was surprising with 80% recovered in the eluate. Despite the larger uncertainty in the Cu result (exceeded ±15%), the total calculated mass was supported by the apparent mass loss of Cu in the AN-102/C-104 effluent (Table 3.3). As expected, ^{137}Cs was the dominant radionuclide detected. The only significant anion detected was nitrate, which was not surprising since the eluant was 0.5 M HNO_3.

Table 3.6. Select Analyte Recoveries in Eluate

Analyte	AN102/C-104 feed, total mg	Estimated remaining from AP-101DF processing, mg	Amount recovered in eluate, mg	Amount recovered in eluate, mmoles	Recovery in Eluate, %
U	20.7	6	32.9	0.138	123
Al	6.18 E+3	—	[1.13]	0.042	[0.02]
Cd	19.6	—	0.46	0.0041	2.3
Cr	81.3	—	4.5	0.087	5.6
Cu	[7.5]	—	6.0	0.094	[80]
Fe	[7.1]	—	1.3	0.023	[18]
Na	8.36 E+4	—	156	6.78	0.19
Ni	142	—	13.3	0.227	9.4
Pb	50.5	—	4.9	0.024	9.8

Values in brackets are based on results reported within 10 times the detection limit, and errors are likely to exceed +/-15%.

The minimum reportable quantity (MRQ) specified by the client is provided in Table 3.4 for information along with the actual analytical result. In cases where a result was below the instrument detection limit (IDL), the dilution-corrected IDL is provided as a "less-than" value. Most of the MRQ levels were met with some exceptions. The large amount of ^{137}Cs prevented the detection limits for ^{154}Eu, and ^{155}Eu from meeting the MRQ levels. Relatively high ^{137}Cs activity increases the gamma background level in the detectors because of Compton scattering, thereby making it difficult to detect lower concentrations of other gamma emitters. The large nitrate concentration required large sample dilutions and prevented the estimated quantitation limit (EQL) for Cl (13 μg/mL) from meeting the MRQ level. However, Cl was found above the EQL at 22 μg/mL. The total inorganic carbon (TIC) analysis was not completed because carbonate is known to evolve as CO_2 in acidic solutions.

3.2.3 Regeneration

The composition of the regeneration solution is shown in Table 3.7. Sodium was the only cation found above the blank concentration. The charge balance between Na and OH was not equal. The counter-ion for the extra Na is predicted to be residual NO_3 from the elution step. The regeneration solution ^{137}Cs C/C_o was 4.68E-4.

Table 3.7. Composition of Regeneration Solution

Analyte	**Concentration, μg/mL**	**Concentration, M**
Na^+	301	1.32 E-2
K^+	< 40	< 1 E-3
OH^-	60	3.6 E-3
Total Cs	3.77 E-3	2.8 E-8
^{137}Cs	7.54E-2 μCi/mL	—
Density, g/mL	0.998 (T=24°C)	

3.2.4 Activity Balance for ^{137}Cs

An activity balance for ^{137}Cs was completed to compare the ^{137}Cs recovered in various process streams to the ^{137}Cs present in the feed sample (Table 3.8). As expected, virtually all ^{137}Cs was found in the eluate stream, recovering >99% of the ^{137}Cs present in the initial AN-102/C-104 feed. The eluate resulted in 108% Cs recovery; this high recovery is attributed to analytical error and is most likely closer to 100%. The other processing effluent Cs recoveries were several orders of magnitude below the eluate Cs recovery.

Table 3.8. Activity Balance for ^{137}Cs

Solution	**^{137}Cs, μCi**	**^{137}Cs Relative to Feed Sample, %**
Feed Sample (AN-102/C-104)	1.21 E+5	1.0 E+2
Initial AP-101DF loading	3.5 E+1	2.9 E-2
Effluent	1.0 E+1	8.5 E-3
Load samples	3.8 E-1	3.1 E-4
Feed displacement	8.4 E-1	6.6 E-4
DI Water Rinse	3.4 E-1	2.8 E-4
Column #1 Eluate	1.31 E+5	1.08 E+2
Column #1 DI water rinse	6.0 E+0	5.0 E-3
Column #1 regeneration	3.8 E+0	3.2 E-3
Lag column Cs loading	2.2 E-1	1.8 E-4
Total ^{137}Cs Recovery	**1.31 E+5**	**1.08 E+2**

3.2.5 SL-644 Resin Volume Changes

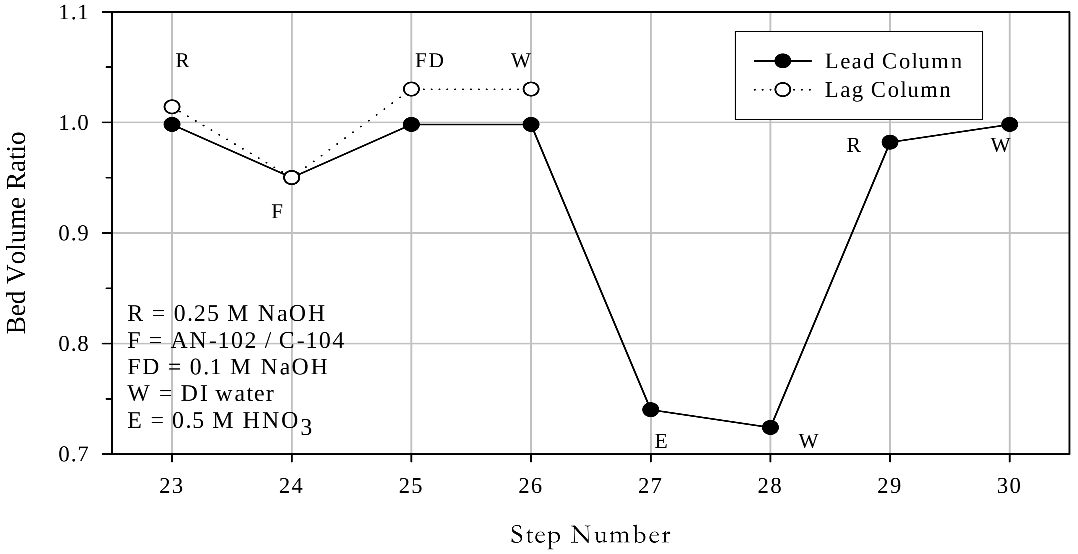
The SL-644 resin is known to change in volume as a function of the solution pH and ionic strength (Hassan, King, and McCabe 1999). The resin BV change history is shown in Table 3.9. The columns are labeled 1 and 2. Column 1 was the lead column for the AW-101 simulant test and the AP-101DF test; Column 2 was the lag column for these tests. Results from both tests have been previously reported (Fiskum, Blanchard, and Arm 2002; Fiskum et al. 2002). These columns were switched for the AN102/C104 ion exchange test. Thus Column 1 was placed in the lag position, and Column 2 was placed in the lead position.

The volume contraction after each subsequent 0.5 M HNO_3 elution step became more pronounced with cycling. The first volume contraction stabilized at 9.2 mL; the final measured volume contraction stabilized at 7.7 mL. The variation in BV as a function of the process steps for both columns is shown in Figure 3.5a and b. In Figure 3.5a, the BVs are normalized to the volume in the 0.25 M NaOH regeneration condition just before AN-102/C-104 loading. Each process step is denoted with a number corresponding to the step number in Table 3.9. In Figure 3.5b, the observed volume changes show clearly the greater contraction and slightly reduced expansion observed over time with repeated cycling. Fluidizing the bed in the H-form (process Step 10) resulted in tighter resin packing, yet the subsequent expansion in the Na-form appeared to have been largely unaffected in that the regeneration volume was equivalent to that obtained before fluidization. There appeared to have been a stepwise decline in resin volume in both the H-form and the Na-form from AW-101 simulant processing and AP-101DF processing. The AN-102/C-104 process-step resin volumes appeared similar to those found for AP-101DF.

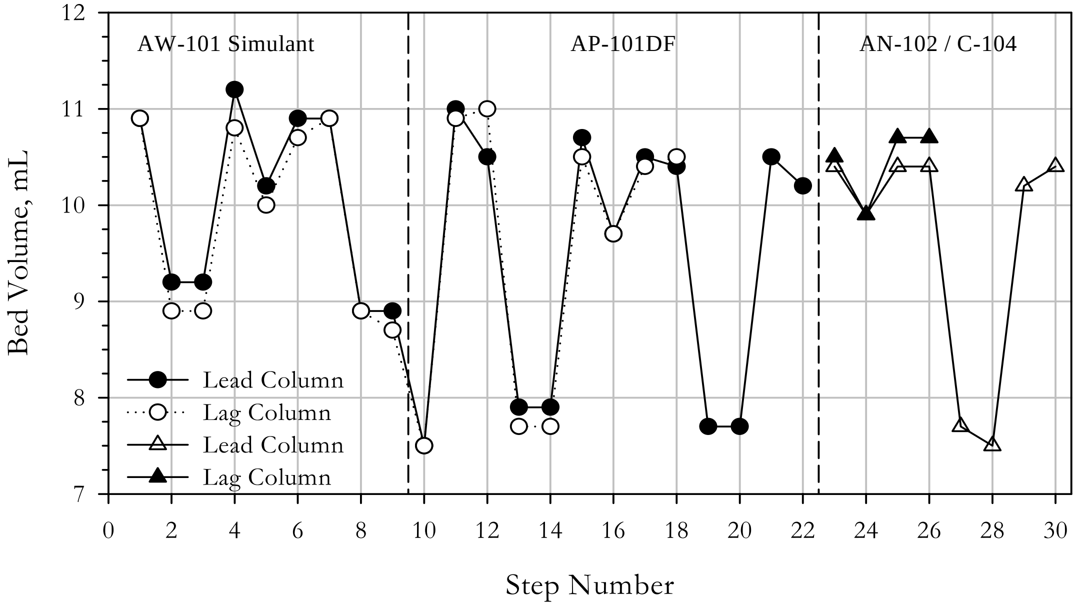
Table 3.9. SL-644 Bed Volumes

Feed	Symbol	Process Step	010319SMC-IV-73 212- to 425-μm particle size	
			Column 1	Column 2
Initial column positions			**Lead column, mL**	**Lag column, mL**
Initial packing	P	1	10.9	10.9
0.5 M HNO_3	E	2	9.2	8.9
DI water	W	3	9.2	8.9
0.25 M NaOH	R	4	11.2	10.8
AW-101 simulant	F	5	10.2	10.0
0.1 M NaOH	FD	6	10.9	10.7
DI water	W	7	10.9	10.9
0.5 M HNO_3	E	8	8.9	8.9
DI water	W	9	8.9	8.7
Re-fluidize bed	RP	10	7.5	7.5
0.25 M NaOH	R	11	11.0	10.9
DI water	W	12	10.5	11.0
0.5 M HNO_3	E	13	7.9	7.7
DI water	W	14	7.9	7.7
0.25 M NaOH	R	15	10.7	10.5
AP-101DF	F	16	9.7	9.7
0.1 M NaOH	FD	17	10.5	10.4
DI water	W	18	10.4	10.5
0.5 M HNO_3	E	19	7.7	—
DI water	W	20	7.7	—
0.25 M NaOH	R	21	10.5	—
DI water	W	22	10.2	—
Switch column positions			**Lag column, mL**	**Lead column, mL**
0.25 M NaOH	R	23	10.5	10.4
AN-102/C-104	F	24	9.9	9.9
0.1 M NaOH	FD	25	10.7	10.4
DI water	W	26	10.7	10.4
0.5 M HNO_3	E	27	—	7.7
DI water	W	28	—	7.5
0.25 M NaOH	R	29	—	10.2
DI water	W	30	—	10.4

a)



b)



a) Relative BV for AN-102/C-104 testing cycle; baseline BV = 10.4 mL in 0.25 M NaOH regeneration condition.

b) Actual BVs for all tested cycles, including simulant AW-101, AP-101DF, and AN-102/C-104 cycles.

Figure 3.5. Comparison of Bed Volumes of the Lead and Lag Columns (SL-644 Batch 010319SMC-IV-73, 212- to 425-µm)

4.0 Conclusions

The objectives of the testing were met.

Batch-distribution coefficients (K_ds) were developed as a function of Na/Cs mole ratios for both SL-644 and IE-911 in AN-102/C-104.

- The SL-644 feed condition equilibrium data resulted in a K_d of 950 mL/g, corresponding to a predicted Cs λ of 240 BVs (0.25 M NaOH condition) at a Na/Cs mole ratio of 8 E+4, 27°C.
- The IE-911 CST feed condition equilibrium data resulted in a K_d value of 1100 mL/g, corresponding to a predicted Cs λ of 1100 BVs at a Na/Cs mole ratio of 8 E+4, 27°C.

Cs decontamination from AN-102/C-104 (Envelope C) was successfully demonstrated.

- An overall DF of 1.13 E+4 was obtained, providing a Cs-decontaminated effluent with 1.42 E-2 µCi/mL ^{137}Cs. This corresponded to 8.1% of the contract limit of 1.68 E-1 µCi/mL in the treated effluent (based on 10% Na_2O loading).(a)

Cs load and elution breakthrough profiles were developed.

- Only 72-BVs (753-mL) of feed were available for processing. The Cs λ value from column testing could not be determined because insufficient feed was available for processing relative to the 10-mL resin bed. The load profile through 72 BVs resulted in no Cs breakthrough.
- The partially Cs-loaded lead column was efficiently eluted with 0.5 M HNO_3. Over 99% of the ^{137}Cs was eluted from the column in 2.5 BVs of eluate, although a total of 11 BVs of eluant were required to reach the elution end point of a C/Co = 0.01. The peak C/Co value for ^{137}Cs was 53. Virtually 100% of the ^{137}Cs present in the feed sample was recovered in the eluate fraction, which had a ^{137}Cs concentration of 6.675 E+2 µCi/mL in 197 mL.

The Cs eluate solution was composited and characterized.(b)

- Along with Cs, U and Cu were recovered in the eluate.

The effectiveness of all SL-644 ion exchange process steps including loading, feed displacement, DI water washing, elution, elution rinse, and resin regeneration were demonstrated.

- Lag column bleed-off, from previous ion exchange waste processing, affected the effluent product with ^{137}Cs contamination. Lag-column samples were a factor of 50 times higher in ^{137}Cs concentration than lead column samples, but were still <10% of the contract ^{137}Cs limit. The elution protocol may have to be revised to avoid subsequent ^{137}Cs contamination in processed tank waste effluent.

(a) The effluent ^{137}Cs concentration was 12 % of the contract limit of 1.2 E-1 µCi/mL based on the maximum loading of 14 wt% waste Na_2O.

(b) Full characterization was not necessary because eluate vitrification was cancelled.

- The SL-644 was adequately regenerated with 4.9 BVs or 2.2 AVs (51 mL) of 0.25 M NaOH. The average ^{137}Cs concentration in the regeneration solution was 7.5 E-2 μCi/mL, equivalent to C/C$_o$ of 4.7 E-4.
- An activity balance for ^{137}Cs indicated that 100% of the ^{137}Cs present in the feed sample was accounted for in the samples and process streams (mostly in the eluate), which is indicative of good experimental integrity.

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Appendix A

General Calculations

Appendix A: General Calculations

^{137}Cs Contractual Limit and Design Basis Limit in AN-102/C-104 Env. C Vitrification Feed

<u>Assumptions-minimum waste loading</u>

1) Concentration of Na_2O in Env. C glass = 10% (= 10 g Na_2O/100 g glass)

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

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

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

5) AN-102/C-104 actual waste Na concentration = 4.8 M

6) AN-102/C-104 actual waste ^{137}Cs concentration = 161 μCi/mL/4.8 M Na

<u>Na Loading in Glass</u>

10 g Na_2O/100g glass * 1 mole Na_2O/62 g Na_2O) * (2 mole Na/ mole Na_2O)*
(23 g Na/mole Na) * (2.66 g glass/mL glass) = 0.197 g Na/mL glass

Maximum ^{137}Cs:Na in glass
(3.0E-7 Ci ^{137}Cs/mL glass)/(0.197 g Na/mL glass) = 1.52 E-6 Ci ^{137}Cs/g Na

(1.52 E-6 Ci ^{137}Cs/g Na) * (23 g Na/mole) = 3.50E-5 Ci ^{137}Cs/mole Na

Maximum ^{137}Cs:Na in feed
(3.50E-5 Ci ^{137}Cs/mole Na) * (4.8 mole Na/L feed) = 1.68 E-4 Ci ^{137}Cs/L
= 1.68 E+2 μCi ^{137}Cs/L
= 1.68 E-1 μCi ^{137}Cs/mL

<u>AN-102/C-104 actual waste Cs fraction remaining (C/Co) Contractual and Design Limit</u>
(1.68 E-1 μCi ^{137}Cs/mL)/(161 μCi ^{137}Cs/mL)= 1.04 E-3 C/C_o; DF = 958
= 0.104 % C/C_o

^{137}Cs Contractual Limit and Design Basis Limit in AN-102/C-104 Env. C Vitrification Feed

Assumptions-maximum waste loading

1) Concentration of Na_2O in Env. C glass = 14% (= 14 g Na_2O/100 g glass)

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

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

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

5) AN-102/C-104 actual waste Na concentration = 4.8 M

6) AN-102/C-104 actual waste ^{137}Cs concentration = 161 µCi/mL/4.8 M Na

Na Loading in Glass

14 g Na_2O/100g glass * 1 mole Na_2O/62 g Na_2O) * (2 mole Na/ mole Na_2O)*
(23 g Na/mole Na) * (2.66 g glass/mL glass) = 0.276 g Na/mL glass

Maximum ^{137}Cs:Na in glass

(3.0E-7 Ci ^{137}Cs/mL glass)/(0.276 g Na/mL glass) = 1.09 E-6 Ci ^{137}Cs/g Na

(1.09 E-6 Ci ^{137}Cs/g Na) * (23 g Na/mole) = 2.50E-5 Ci ^{137}Cs/mole Na

Maximum ^{137}Cs:Na in feed

(2.50E-5 Ci ^{137}Cs/mole Na) * (4.8 mole Na/L feed) = 1.20 E-4 Ci ^{137}Cs/L
= 1.20 E+2 µCi ^{137}Cs/L
= 1.20 E-1 µCi ^{137}Cs/mL

AN-102/C-104 actual waste Cs fraction remaining (C/Co) Contractual and Design Limit

(1.20 E-1 µCi ^{137}Cs/ mL)/(161 µCi ^{137}Cs/mL) = 7.45 E-4 C/C_o; DF = 1343
= 0.0745 % C/C_o

Table A.1. AN-102/C-104 Feed and Effluent Composition

AN102/C104 Feed volume
753 mL
AN102/C104 Feed composition

AN102/C104 Effluent volume
753 mL
AN102/C104 Effluent composition

			ASR 6130	ASR-6130			ASR 6281	ASR 6281	ASR 6281			
	Multiplier=		100.0	100.0			24.9	24.9	124.7			
	RPL/LAB #=		PB-1338	01-1345			PB-776	02-0777	02-0777@5			
Det. Limit	*Client ID=*	*FW*	*process blank*	*ANC102/104-C-F/A*	*Molarity*	*ug total*	*process blank*	*AN102/C104 Cs removed*	*AN102/ C104 Cs removed*	*Molarity*	*ug total*	*% recovered in effluent*
(ug/mL)	(Analyte)		(ug/mL)	(ug/mL)			(ug/mL)	(ug/mL)	(ug/mL)			
0.150	Na	22.99	1010	111000	4.83E+0	83583000	--	over range	110000	4.78E+0	82830000	99.1
2.000	K	39.0983	--	[950]	[2.43E-2]	[715350]	--	1080		2.76E-2	813240	[114]
0.060	Al	26.98	84.10	8210	3.04E-1	6182130	--	7840		2.91E-1	5903520	95.5
0.010	Ba	137.33	[1.3]	[1.6]	high blank	NA	--	--		<1.8E-6		--
0.250	Ca	40.08	--	[150]	[3.74E-3]	[112950]	--	149		3.72E-3	112197	[99]
0.015	Cd	112.41	--	26.0	2.31E-4	19578	--	24.2		2.15E-4	18223	93.1
0.050	Co	58.9332	--	--	<8.5E-5		--	[1.9]		[3.22E-5]	[1431]	--
0.020	Cr	51.996	--	108	2.08E-3	81324	--	98		1.89E-3	73945	90.9
0.025	Cu	63.546	--	[10.0]	[1.57E-4]	[7530]	--	--		<9.8E-6		--
0.025	Fe	55.847	[5.7]	[9.4]	[6.63E-5]	[2786]	--	[2.0]		[3.58E-5]	[1506]	[54]
0.100	Mg	24.305	--	--	<4.1E-4		--	--		<1.0E-4		--
0.050	Mn	54.938	--	--	<9.1E-5		--	--		<2.3E-5		--
0.030	Ni	58.7	--	188	3.20E-3	141564	--	145		2.47E-3	109185	77.1
0.100	Pb	207.2	--	[67]	[3.23E-4]	[50451]	--	69		3.33E-4	52032	[103]
0.050	Zn	65.38	--	[8.6]	[1.32E-4]	[6476]	--	[4.0]		[6.12E-5]	[3012]	[47]
Other Analytes												
0.025	Ag		--	--			--	--				--
0.250	As		--	--			--	--				--
0.050	B	10.81	750	624	high blank	NA	--	86		7.99E-3	65059	NA
0.010	Be		--	--			--	--				--
0.100	Bi		--	--			--	--				--
0.200	Ce		--	--			--	--				--
0.050	Dy		--	--			--	--				--
0.100	Eu		--	--			--	--				--
0.050	La		--	--			--	--				--
0.030	Li		--	--			--	--				--
0.050	Mo	95.94	--	[22]	[2.29E-4]	[16566]	--	23		2.41E-4	17394	[105]
0.100	Nd	144.24	--	--			--	[3.6]		[2.50E-5]	[2711]	--
0.100	P	30.974	--	748	2.41E-2	563244	--	562		1.81E-2	423186	75.1
0.750	Pd		--	--			--	--				--
0.300	Rh		--	--			--	--				--
1.100	Ru		--	--			--	--				--
0.500	Sb		--	--			--	--				--
0.250	Se		--	--			--	--				--
0.500	Si	28.0855	973	1110	3.95E-2	835830	--	179		6.37E-3	134787	16.1
1.500	Sn		--	--			--	--				--
0.015	Sr	87.62	--	86.5	9.87E-4	65135	--	86.9		9.92E-4	65436	100.5
1.500	Te		--	--			--	--				--
1.000	Th		--	--			--	--				--
0.025	Ti		--	--			--	--				--
0.500	Tl		--	--			--	--				--
2.000	U	238	--	--			--	--				--
	U(KPA)	238	0.11	27.49	1.15E-4	20698	0.03	0.82		3.45E-6	619	3.0
0.050	V		--	--			--	--				--
2.000	W		--	--			--	--				--
0.050	Y		--	--			--	--				--
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).
4) The Fe value for ASR 6130 was blank-subtracted
5) The feed sample was also analyzed per ASR 6107

Table A.2. AN-102/C-104 Eluate Composition

AN102/C104 Cs Eluate volume
197 mL
AN102/C104 Cs Eluate composition

			ASR 6281	ASR 6281	ASR 6281						
	Multiplier=		1.0	10.0	10.0						
	RPL/LAB #=		02-00779-DB	02-00779	02-00779 DUP						
Det. Limit	***Client ID=***	***FW***	***Diluent Blank***	***AN102/C104-CsE-Comp 1***	***AN102/C104-CsE-Comp 1-Dup***	***Average***	***M***	***ug total***	***estimated ug from AP-101 processing***	***% recovered in eluate***	
(ug/mL)	**(Analyte)**		**(ug/mL)**	**(ug/mL)**	**(ug/mL)**						
0.150	**Na**	**22.99**	**--**	**775**	**810**	**792.50**	**3.45E-02**	**156123**		**0.19**	
2.000	**K**	**39.0983**	**--**	**--**	**--**					**--**	
0.060	**Al**	**26.98**	**--**	**[5.7]**	**[5.8]**	**[5.8]**	**[2.13E-4]**	**[1133]**		**[0.02]**	
0.010	**Ba**	**137.33**	**--**	**[0.16]**	**[0.19]**	**[0.18]**	**[1.27E-6]**	**[34]**		**NA**	high blank in feed
0.250	**Ca**	**40.08**	**--**	**--**	**--**	**<2.5E+0**				**--**	
0.015	**Cd**	**112.41**	**--**	**2.31**	**2.38**	**2.35**	**2.09E-05**	**462**		**2.36**	
0.050	**Co**	**58.9332**	**--**	**--**	**--**	**<5.0E-1**				**--**	
0.020	**Cr**	**51.996**	**--**	**22.5**	**23.3**	**22.9**	**4.40E-04**	**4511**		**5.55**	
0.025	**Cu**	**63.546**	**--**	**30.1**	**31.1**	**30.6**	**4.82E-04**	**6028**		**[80]**	
0.025	**Fe**	**55.847**	**--**	**6.16**	**6.56**	**6.36**	**1.14E-04**	**1253**		**[45]**	
0.100	**Mg**	**24.305**	**--**	**--**	**--**	**<1.0E+0**				**--**	
0.050	**Mn**	**54.938**	**--**	**--**	**--**	**<5.0E-1**				**--**	
0.030	**Ni**	**58.7**	**--**	**66.6**	**68.9**	**67.8**	**1.15E-03**	**13347**		**9.43**	
0.100	**Pb**	**207.2**	**--**	**24.8**	**25.4**	**25.1**	**1.21E-04**	**4945**		**9.80**	
0.050	**Zn**	**65.38**	**--**	**[2.7]**	**[2.8]**	**[2.8]**	**[4.21E-5]**	**[542]**		**[8.37]**	
Other Analytes											
0.025	**Ag**		**--**	**--**	**--**					**--**	
0.250	**As**		**--**	**--**	**--**					**--**	
0.050	**B**	**10.81**	**--**	**7.28**	**7.47**	**7.38**	**6.82E-04**	**1453**		**NA**	high blank in feed
0.010	**Be**		**--**	**--**	**--**					**--**	
0.100	**Bi**		**--**	**--**	**--**					**--**	
0.200	**Ce**		**--**	**--**	**--**					**--**	
0.050	**Dy**		**--**	**--**	**--**					**--**	
0.100	**Eu**		**--**	**--**	**--**					**--**	
0.050	**La**		**--**	**--**	**--**					**--**	
0.030	**Li**		**--**	**--**	**--**					**--**	
0.050	**Mo**	**95.94**	**--**	**--**	**--**	**<5.0E-1**				**--**	
0.100	**Nd**	**144.24**	**--**	**--**	**--**					**--**	
0.100	**P**	**30.974**	**--**	**--**	**--**	**<1.0E+0**				**--**	
0.750	**Pd**		**--**	**--**	**--**					**--**	
0.300	**Rh**		**--**	**--**	**--**					**--**	
1.100	**Ru**		**--**	**--**	**--**					**--**	
0.500	**Sb**		**--**	**--**	**--**					**--**	
0.250	**Se**		**--**	**--**	**--**					**--**	
0.500	**Si**	**28.0855**	**--**	**[20]**	**[20]**	**[20]**	**[7.12E-4]**	**[3940]**		**[0.47]**	
1.500	**Sn**		**--**	**--**	**--**					**--**	
0.015	**Sr**	**87.62**	**--**	**[1.0]**	**[1.1]**	**[1.1]**	**[1.20E-5]**	**[207]**		**[0.32]**	
1.500	**Te**		**--**	**--**	**--**					**--**	
1.000	**Th**		**--**	**--**	**--**					**--**	
0.025	**Ti**		**--**	**--**	**--**					**--**	
0.500	**Tl**		**--**	**--**	**--**					**--**	
2.000	**U**	**238**	**--**	**[170]**	**[180]**	**[175]**	**[7.35E-4]**	**[34475]**	**6000**	**NA**	not detected in feed by ICP
	U(KPA)	**238**	**0.16**	**169**	**165**	**167**	**7.02E-04**	**32899**	**6000**	**123.22**	
0.050	**V**		**--**	**--**	**--**					**--**	
2.000	**W**		**--**	**--**	**--**					**--**	
0.050	**Y**		**--**	**--**	**--**					**--**	
0.050	**Zr**		**--**	**--**	**--**					**--**	

Note: 1) O Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values 2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indi 3) "--" indicate measurement is below detection. Sample detection limit may be found by
multiplyin multiplying "det. limit" (far left column) by "multiplier" (top of each column).
4) The Fe value for ASR 6130 was blank-subtracted
5) The feed sample was also analyzed per ASR 6107

Table A.3. AN-102 Feed Composition

AN102 Feed composition

		ASR=	6130	6130	6130			
		Multiplier=	100.0	200.0	200.0			
		RPL/LAB #=	PB-1338	01-1354	01-2354D	average	average	
Det. Limit		*Client ID=*	*~~process blank~~*	*~~AN102 CST-C-F/A~~*	*~~AN102CST-C-F/A~~*			
(ug/mL)	FW	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	M	
0.150	22.990	Na	1010	136000	146000	141000	6.13E+0	
2.000	39.098	K	--	[1100]	[1100]	[1100]	[2.8E-2]	
						0		
0.060	26.980	Al	84.10	6520	6,730	6625	2.46E-1	
0.010	137.330	Ba	[1.3]	[2.5]	--			
0.250	40.080	Ca	--	[190]	[190]	[190]	[4.7E-3]	
0.015	112.410	Cd	--	32.1	32.3	32	2.86E-4	
0.050	58.930	Co	--	--				
0.020	51.996	Cr	--	119	121	120	2.31E-3	
0.025	63.546	Cu	--	[8.4]	[7.5]	[8.0]	[1.3E-4]	
0.025	55.847	Fe	[5.7]	[9.1]	[8.8]	[9.0]	[5.8E-5]	blank subtracted
0.100	24.305	Mg	--	--	--			
0.050	54.938	Mn	--	--	--			
0.030	58.700	Ni	--	237	240	239	4.06E-3	
0.100	207.200	Pb	--	[88]	[84]	[86]	[4.2E-4]	
0.050	65.380	Zn	--	--	--			
Other Analytes								
0.025		Ag	--	--	--			
0.250		As	--	--	--			
0.050	10.810	B	750	815	755	785	high blank	
0.010		Be	--	--	--			
0.100		Bi	--	--	--			
0.200		Ce	--	--	--			
0.050		Dy	--	--	--			
0.100		Eu	--	--	--			
0.050		La	--	--	--			
0.030		Li	--	--	--			
0.050	95.940	Mo	--	[26]	[27]	[27]	[2.8E-4]	
0.100		Nd	--	--				
0.100	30.970	P	--	457	455	456	1.47E-2	
0.750		Pd	--	--	--			
0.300		Rh	--	--	--			
1.100		Ru	--	--	--			
0.500		Sb	--	--	--			
0.250		Se	--	--	--			
0.500	28.086	Si	973	1250	1240	1245	high blank	
1.500		Sn	--	--	--			
0.015	87.620	Sr	--	60.3	62.1	61	6.98E-4	
1.500		Te	--	--	--			
1.000		Th	--	--	--			
0.025		Ti	--	--	--			
0.500		Tl	--	--	--			
2.000	238.000	U	--	--	--			
0.050		V	--	--	--			
2.000		W	--	--	--			
0.050		Y	--	--	--			
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).

Appendix B

Batch-Contact Calculations

Appendix B: Batch Contact Calculations

Table B.1. Cs and Sr Kd Determinations

	g/mL
AN-102/C-104 feed density	1.2438
AN-102 density	1.3012

	m	F	I_{Na}			V	C_o		C_1					
Sample ID	Resin, g	F-factor	I_{Na} factor	corrected resin mass, g	waste, g	waste, mL	Starting [Cs], M	Fraction of original ^{137}Cs	Equilibrium [Cs], M	[Na], M	Na:Cs mole ratio	K_d, mL/g	dry bed density, g/mL	λ
ANC102/104-644	0.0507	0.9321	1.25	0.0591	6.1606	4.9530	5.99E-5	3.43E-2	2.05E-6	4.8	2.34E+6	2327	0.25	582
ANC102/104-644 D	0.0518	0.9321	1.25	0.0604	6.1457	4.9411	5.99E-5	3.30E-2	1.98E-6	4.8	2.43E+6	2360	0.25	590
ANC102/104-S1-644	0.0505	0.9321	1.25	0.0588	6.1557	4.9491	9.98E-4	9.59E-2	9.57E-5	4.8	5.01E+4	764	0.25	191
ANC102/104-S1-644 D	0.0499	0.9321	1.25	0.0581	6.124	4.9236	9.98E-4	1.11E-1	1.11E-4	4.8	4.33E+4	653	0.25	163
ANC102/104-S2-644	0.0508	0.9321	1.25	0.0592	6.1045	4.9079	5.10E-3	3.22E-1	1.64E-3	4.8	2.92E+3	187	0.25	47
ANC102/104-S2-644 D	0.0501	0.9321	1.25	0.0584	6.1428	4.9387	5.10E-3	3.39E-1	1.73E-3	4.8	2.77E+3	177	0.25	44
ANC102/104-C	0	NA	NA	0.0000	2.5239	2.0292	5.99E-5	9.85E-1	5.90E-5	4.8	8.13E+4	NA	NA	NA
ANC102/104-S1-C	0	NA	NA	0.0000	2.5845	2.0779	9.98E-4	9.67E-1	9.65E-4	4.8	4.97E+3	NA	NA	NA
ANC102/104-S2-C	0	NA	NA	0.0000	2.6942	2.1661	5.10E-3	1.05E+0	5.35E-3	4.8	8.97E+2	NA	NA	NA
AN-102-CST	0.0507	0.9708	NA	0.0492	6.3992	4.9179	7.70E-5	9.10E-2	7.01E-6	6.1	8.71E+5	992	1.04	1032
AN-102-CST D	0.0507	0.9708	NA	0.0492	3.7533	2.8845	7.70E-5	5.55E-2	4.27E-6	6.1	1.43E+6	992	1.04	1031
AN-102-S3-CST	0.0506	0.9708	NA	0.0491	6.3929	4.9131	1.01E-3	8.29E-2	8.39E-5	6.1	7.27E+4	1112	1.04	1156
AN-102-S3-CST D	0.0507	0.9708	NA	0.0492	6.3359	4.8693	1.01E-3	8.08E-2	8.18E-5	6.1	7.45E+4	1130	1.04	1176
AN-102-S4-CST	0.0488	0.9708	NA	0.0474	6.4947	4.9913	5.12E-3	3.18E-1	1.63E-3	6.1	3.75E+3	227	1.04	236
AN-102-S4-CST D	0.0505	0.9708	NA	0.0490	6.3438	4.8753	5.12E-3	2.97E-1	1.52E-3	6.1	4.01E+3	235	1.04	245
AN-102CST-C	0	NA	NA	0.0000	2.6172	2.0114	7.70E-5	9.94E-1	7.66E-5	6.1	7.97E+4	NA	NA	NA
AN-102CST-S3-C	0	NA	NA	0.0000	2.691	2.0681	1.01E-3	1.00E+0	1.02E-3	6.1	6.00E+3	NA	NA	NA
AN102CST-S4-C	0	NA	NA	0.0000	2.7569	2.1187	5.12E-3	1.00E+0	5.12E-3	6.1	1.19E+3	NA	NA	NA

$K_d = (C_o - C_1)/C_1 \times V/(m*I_{Na}*F)$

Table B.1 contd

Batch Contact of AN-102/C-104 Waste with SuperLig 644 and IE-911
Superlig 644 batch 010319SMC-IV-73, 212-425 um particle size, weighed in the H form
AN-102/C-104 Batch Contact and AN/102 Batch Contact
Continued

CMC Analysis

Sample ID	CMC ID	Net Cs-137 µCi/mL	Equilibrium [Cs], M	Na:Cs mole ratio	K_d, mL/g	λ
ANC102/104-644	01-1341	5.16E+0	1.92E-6	2.50E+6	2532	633
ANC102/104-644 D	–	–	–	–	–	–
ANC102/104-S1-644	01-1342	1.50E+1	9.30E-5	5.16E+4	819	205
ANC102/104-S1-644 D	01-1343	1.45E+1	8.99E-5	5.34E+4	856	214
ANC102/104-S2-644	01-1344	5.14E+1	1.63E-3	2.95E+3	177	44
ANC102/104-S2-644 D	–	–	–	–	–	–
ANC102/104-C	01-1345	1.61E+2	–	–	–	–
ANC102/104-S1-C	01-1346	1.62E+2	–	–	–	–
ANC102/104-S2-C	01-1347	1.60E+2	–	–	–	–

	CMC ID	Net Cs-137 µCi/mL	Equilibrium [Cs], M	Na:Cs mole ratio	K_d, mL/g	λ	Starting [Sr], M	Equilibrium ^{90}Sr µCi/mL	Fraction of original ^{90}Sr	Equilibrium [Sr], M	K_d, mL/g	λ
AN-102-CST	01-1348	1.84E+1	6.86E-6	8.90E+5	1022	1063	6.98E-4	9.14E+0	6.55E-1	4.57E-4	53	55
AN-102-CST D	01-1349	–	–	–	–	–	6.98E-4	8.22E+0	5.89E-1	4.11E-4	41	42
AN-102-S3-CST	01-1350	1.76E+1	8.62E-5	7.08E+4	1074	1117	6.98E-4	1.07E+1	7.67E-1	5.35E-4	30	32
AN-102-S3-CST D	01-1351	–	–	–	–	–	6.98E-4	1.02E+1	7.31E-1	5.10E-4	36	38
AN-102-S4-CST	01-1352	6.59E+1	1.63E-3	3.74E+3	225	234	6.98E-4	1.05E+1	7.53E-1	5.25E-4	35	36
AN-102-S4-CST D	01-1353	–	–	–	–	–	6.98E-4	1.03E+1	7.38E-1	5.15E-4	35	37
AN-102CST-C	01-1354	2.05E+2	–	–	–	–	6.98E-4	1.40E+1				
AN-102CST-S3-C	01-1355	2.10E+2	–	–	–	–						
AN102CST-S4-C	01-1356	2.05E+2	–	–	–	–						

Appendix C

Column Testing Calculations

Appendix C: Column Testing Calculations

Table C.1. Column Flow Calculations

Test Runs
Column Preparation
SL-644 batch 010310SMC-IV-73 212-425 um particle size

Bed volume in AN102/C104 feed	10.4	mL
Apparatus volume	42	mL

	Densities	
HNO3	1.012	g/mL
0.1M NaOH	0.999	
0.25M NaOH	1.006	
AN102/C104 waste	1.2438	

	apparatus volume
total system	42
column 1	23
column 2	21

Conditioning 6/25/01
Lead column + Lag column

start	end	Δt	mass, g	volume, mL	mL/min flow rate	BV/hr	BV	AV
0.25M NaOH wash								
6/25/01 8:55	11:50	2:55	83.2	82.7	0.47	2.73	8.0	1.97

AN102/C104 actual waste loading starting on 6/25/01

Initial effluent collection
1 apparatus volume of feed

start	end	Δt	mass, g	cumul. volume, mL	flow rate, mL/min	BV/hr	apparatus volume
6/25/01 11:57	13:27	1:30	41	40.8	0.453	2.61	1.0

Feed flow

start	end	Δt	mass	cumul. volume, mL	flow rate, mL/min	BV/hr	cumulative BV	Lead column sampling Sample ID	sample mass, g	volume, mL	total vol., mL	BV	total sample vol, mL
6/25/01 13:31	13:47	0:16	7.1	46.5	0.357	2.06	4.5	AN102L-F1	2.351	1.890	48.354	4.6	1.8903
13:57	17:00	3:03	111.5	130.4	0.459	2.65	12.5	AN102L-F2	2.941	2.365	137.026	13.2	4.2549
17:10	20:20	3:10	222.4	219.6	0.469	2.71	21.1	AN102L-F3	2.621	2.107	230.551	22.2	6.3619
20:29	23:40	3:11	342.7	316.3	0.506	2.92	30.4	AN102L-F4	2.492	2.004	330.928	31.8	8.3654
6/25/01 23:52	3:00	3:08	456.1	407.5	0.485	2.80	39.2	AN102L-F5	3.858	3.102	425.867	40.9	11.4675
3:14	6:20	3:06	565.2	495.2	0.472	2.72	47.6	AN102L-F6	2.475	1.989	516.720	49.7	13.4570
6:23	9:40	3:17	671.8	580.9	0.435	2.51	55.9	AN102L-F7	2.368	1.904	607.091	58.4	15.3610
9:51	13:01	3:10	779.4	667.4	0.455	2.63	64.2	AN102L-F8	2.395	1.926	697.375	67.1	17.2865
6/26/01 13:12	15:10	1:58	846.0	720.9	0.454	2.62	69.3						
				average	**0.467**	**2.69**						total volume	753.0
ΔT, total	27:13:00												

Table C.1 contd

Lag column sampling

Sample ID	sample mass, g	volume, mL	total vol., mL	BV	total sample vol, mL
AN102P-F1	2.949	2.371	48.84	4.70	2.371
AN102P-F2	2.269	2.255	135.03	12.98	4.626
AN102P-F3	2.058	1.654	225.84	21.72	6.281
AN102P-F4	0.826	0.664	323.23	31.08	6.945
AN102P-F5	1.429	1.149	415.55	39.96	8.094
AN102P-F6	3.434	2.761	506.02	48.66	10.855
AN102P-F7	2.300	1.849	593.58	57.07	12.704
AN102P-F8	2.527	2.032	682.12	65.59	14.736
				total volume	735.7

total volume	752.95	mL	effluent measure
mass processed	937.20	g	net feed bottle measure
volume processed	753.50	mL	
	72.40	bed volumes total	

0.1M NaOH feed displacement

Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	mL/min	BV/hr	BV	cumul. BV	AV	Cumul. AV
	15:20	16:48	1:28	43.4	34.9	0.40	2.29	3.36	3.36	0.83	0.83
AN102-FD-1	16:51	17:12	0:21	10.1	8.1	0.39	2.23	0.78	4.14	0.19	1.02
AN102-FD-2	17:13	17:26	0:13	9.1	9.1	0.70	4.02	0.87	5.01	0.22	1.24
AN102-FD-3	17:27	17:46	0:19	8.9	8.9	0.47	2.69	0.85	5.86	0.21	1.45
AN102-FD-4	17:47	18:07	0:20	11.0	11.0	0.55	3.16	1.05	6.91	0.26	1.71
AN102-FD-5	18:08	18:29	0:21	10.4	10.4	0.50	2.87	1.00	7.92	0.25	1.96
AN102-FD-6	18:30	18:50	0:20	9.1	9.1	0.46	2.63	0.88	8.79	0.22	2.18
AN102-FD-7	18:51	19:11	0:20	8.8	8.8	0.44	2.54	0.85	9.64	0.21	2.39
AN102-FD-8	19:12	19:32	0:20	9.2	9.2	0.46	2.64	0.88	10.52	0.22	2.61
AN102-FD-9	19:32	19:57	0:25	11.4	11.4	0.46	2.63	1.10	11.62	0.27	2.88
	ΔT, total	4:37			**average**	**0.48**	**2.77**				
				sum	120.8	mL					

The initial rinse was collected in the AN102/C104 effluent bottle.

DI water rinse

Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	mL/min	BV/hr	BV	cumul. BV	AV	Cumul. AV
AN102-FDI-1	20:03	20:23	0:20	10.000	10.000	0.50	2.88	0.96	0.96	0.24	0.24
AN102-FDI-2	20:24	20:44	0:20	9.311	9.311	0.47	2.69	0.90	1.86	0.22	0.46
AN102-FDI-3	20:45	21:05	0:20	9.447	9.447	0.47	2.72	0.91	2.77	0.22	0.68
AN102-FDI-4	21:05	21:25	0:20	9.147	9.147	0.46	2.64	0.88	3.64	0.22	0.90
AN102-FDI-5	21:26	21:46	0:20	9.254	9.254	0.46	2.67	0.89	4.53	0.22	1.12
AN102-FDI-6	21:47	22:07	0:20	9.385	9.385	0.47	2.71	0.90	5.44	0.22	1.35
AN102-FDI-7	22:08	22:28	0:20	9.695	9.695	0.48	2.80	0.93	6.37	0.23	1.58
AN102-FDI-8	22:30	22:50	0:20	9.716	9.716	0.49	2.80	0.93	7.30	0.23	1.81
AN102-FDI-9	22:53	23:12	0:19	9.861	9.861	0.52	2.99	0.95	8.25	0.23	2.04
	ΔT, total	3:09			**average**	**0.48**	**2.77**				
				sum	85.8	mL					

Table C.1 contd

Cs elution from lead column,
Separate columns

Sample ID	start	end	Δt	mass collected, g	volume, mL	flow rate, mL/min	BV/hr	cumul. BV
HNO3 wash--lead column								
AN102L-E1	23:23	0:23	1:00	8.3300	8.23	0.14	0.79	0.79
AN102L-E2	0:24	1:23	0:59	7.7229	7.63	0.13	0.75	1.53
AN102L-E3	1:24	2:23	0:59	10.3513	10.23	0.17	1.00	2.51
AN102L-E4	2:24	3:23	0:59	9.6517	9.54	0.16	0.93	3.43
AN102L-E5	3:24	4:24	1:00	10.3193	10.20	0.17	0.98	4.41
AN102L-E6	4:26	5:26	1:00	10.2201	10.10	0.17	0.97	5.38
AN102L-E7	5:28	6:28	1:00	10.8062	10.68	0.18	1.03	6.40
AN102L-E8	6:29	7:28	0:59	10.0951	9.98	0.17	0.98	7.36
AN102L-E9	7:30	8:30	1:00	10.1534	10.03	0.17	0.96	8.33
AN102L-E10	8:31	9:30	0:59	9.7621	9.65	0.16	0.94	9.26
AN102L-E11	9:31	10:30	0:59	9.9411	9.82	0.17	0.96	10.20
AN102L-E12	10:31	11:30	0:59	9.8147	9.70	0.16	0.95	11.13
AN102L-E13	11:30	12:30	1:00	10.0791	9.96	0.17	0.96	12.09
AN102L-E14	12:31	13:30	0:59	10.0347	9.92	0.17	0.97	13.04
AN102L-E15	13:30	14:31	1:01	10.3327	10.21	0.17	0.97	14.03
AN102L-E16	14:32	15:25	0:53	9.2188	9.11	0.17	0.99	14.90
AN102L-E17	15:26	16:24	0:58	9.7495	9.63	0.17	0.96	15.83
AN102L-E18	16:25	17:28	1:03	10.3382	10.22	0.16	0.94	16.81
AN102L-E19	6:54	8:01	1:07	12.1664	12.02	0.18	1.04	17.97
AN102L-E20	8:02	9:03	1:01	10.1879	10.07	0.17	0.95	18.93
	ΔT, total	33:40:00	**sum**	**199.28**	**196.91**			
					average	**0.16**	**0.95**	

DI water

Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AN102-EDI-1	9:10	9:21	0:11	9.837	9.837	0.89	5.16	0.9	0.9	0.43	0.43
AN102-EDI-2	9:23	9:49	0:26	12.944	12.944	0.50	2.87	1.2	2.2	0.56	0.99
AN102-EDI-3	9:50	10:12	0:22	10.938	10.938	0.50	2.87	1.1	3.2	0.48	1.47
AN102-EDI-4	10:13	10:33	0:20	9.910	9.910	0.50	2.86	1.0	4.2	0.43	1.90
AN102-EDI-5	10:34	10:53	0:19	9.352	9.352	0.49	2.84	0.9	5.1	0.41	2.30
AN102-EDI-6	10:55	11:14	0:19	8.881	8.881	0.47	2.70	0.9	5.9	0.39	2.69
		ΔT, total	2:04		**average**	**0.49**	**2.83**				
				sum	61.9	mL					

Table C.1 contd

Regeneration with 0.25M NaOH

Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AN102L-RGN	7:51	9:35	1:44	16.0	16.0	0.15	0.89	1.5	1.5	0.70	0.70
	9:42	11:32	1:50	17.7	17.7	0.16	0.93	1.7	3.2	0.77	1.47
	11:34	13:19	1:45	17.0	17.0	0.16	0.93	1.6	4.9	0.74	2.20
	ΔT, total	5:28		**sum**	**50.7**	**average**	**0.92**				

Rinse with DI water

Sample ID	Start	End	Δt	Sample mass, g	Sample volume, mL	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV	
AN102-DIRinse-Final	13:25	14:05	0:40	17.0	17.0	0.43	2.45	1.6	1.6	0.74	0.74	Lost about 0.5 mL on transfer
	14:14	14:50	0:36	17.2	17.2	0.48	2.76	1.7	3.3	0.75	1.49	
	14:51	15:21	0:30	13.6	13.6	0.45	2.62	1.3	4.6	0.59	2.08	Lost about 2 mL on transfer
	ΔT, total	1:56		**sum**	**47.8**	**average**	**2.61**					

Table C.2. Column Sample Counting

Counting Data for TI-RPP-WTP-080, Rev. 0

Background Counts

Net counts	Error	Time (sec)	cpm	uncertainty
12	6	3000	0.24	50%
21	6	6000	0.21	29%
18	5	6000	0.18	28%
8	2	3000	0.16	25%
13	3	3000	0.26	23%
		average	**0.21**	

3000 sec MDL
0.35 cpm
1.63E-07 C/Co
0.140 cpm/g

AN102/C104Comparitor Standard density **1.2438** g/mL

0.1020g AP101 brought to 2.0047 g then 0.1 g pipetted into a 2-mL geometry (diluent is 0.1 M NaOH).

0.00980 g in counting geometry

Net counts	Error	Time (sec)	cpm	net cpm	mass	net cpm/g	mL	cpm/mL
43790	287	300	8758	8758	0.0098	8.94E+5	0.0079	1.11E+6
42959	278	300	8592	8592	0.0098	8.77E+5	0.0079	1.09E+6
42361	275	300	8472	8472	0.0098	8.64E+5	0.0079	1.08E+6
42948	278	300	8590	8589	0.0098	8.76E+5	0.0079	1.09E+6
41367	270	300	8273	8273	0.0098	8.44E+5	0.0079	1.05E+6
41229	274	300	8246	8246	0.0098	8.41E+5	0.0079	1.05E+6
40814	269	300	8163	8163	0.0098	8.33E+5	0.0079	1.04E+6
40727	270	300	8145	8145	0.0098	8.31E+5	0.0079	1.03E+6
					Average	**8.58E+5**		**1.07E+6**
					standard deviation	**2.34E+4**		**2.91E+4**
					% standard deviation	**2.7**		**2.7**

AN-102/C104 run starting 6/25/01
Lead column, loading phase

Sample ID	Net counts	Error	Count time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	% C/Co	DF	total uCi
AN102L-F1	-94	451	3000	-2.09	2.2688	1.8241	-0.92	-1.15	-1.07E-6	4.6	-1.07E-4	6.12E+6	-3.15E-4
AN102L-F2	155	483	3000	2.89	2.8326	2.2774	1.02	1.27	1.19E-6	13.2	1.19E-4	8.41E+5	4.36E-4
AN102L-F3	380	469	3000	7.39	2.5156	2.0225	2.94	3.65	3.43E-6	22.2	3.43E-4	2.92E+5	1.12E-3
AN102L-F4	144	474	3000	2.67	2.4079	1.9359	1.11	1.38	1.29E-6	31.8	1.29E-4	7.73E+5	4.03E-4
AN102L-F5	881	520	3000	17.41	3.7413	3.0080	4.65	5.79	5.43E-6	40.9	5.43E-4	1.84E+5	2.63E-3
AN102L-F6	52	490	3000	0.83	2.375	1.9095	0.35	0.43	4.07E-7	49.7	4.07E-5	2.45E+6	1.25E-4
AN102L-F7	347	461	3000	6.73	2.3008	1.8498	2.93	3.64	3.41E-6	58.4	3.41E-4	2.93E+5	1.02E-3
AN102L-F8	14	678	6000	-0.07	2.2771	1.8308	-0.03	-0.04	-3.58E-8	67.1	-3.58E-6	6.12E+6	-1.06E-5

Table C.2 contd

AN-102/C104 run starting 6/25/01

Lag column, loading phase

Sample ID	Net counts	Error	Count time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	DF	total uCi
AN102P-F1	23425	414	3000	468.290	2.8383	2.2820	164.99	205.21	1.92E-4	4.7	1.92E-2	5198	7.07E-2
AN102P-F2	14534	485	3000	290.470	2.1202	1.7046	137.00	170.40	1.60E-4	13.0	1.60E-2	6260	4.38E-2
AN102P-F3	14803	488	3000	295.850	1.9924	1.6019	148.49	184.69	1.73E-4	21.7	1.73E-2	5776	4.47E-2
AN102P-F4	7288	364	3000	145.550	0.7763	0.6241	187.49	233.20	2.19E-4	31.1	2.19E-2	4574	2.20E-2
AN102P-F5	10973	444	3000	219.250	1.3375	1.0753	163.93	203.89	1.91E-4	40.0	1.91E-2	5232	3.31E-2
AN102P-F6	22884	547	3000	457.470	3.3434	2.6881	136.83	170.19	1.60E-4	48.7	1.60E-2	6268	6.90E-2
AN102P-F7	13867	479	3000	277.130	2.2121	1.7785	125.28	155.82	1.46E-4	57.1	1.46E-2	6846	4.18E-2
AN102P-F8	15181	505	3000	303.410	2.0266	1.6294	149.71	186.21	1.75E-4	65.6	1.75E-2	5728	4.58E-2
AN102FEcomp1	4789	101	3000	95.570	2.0507	2	46.60	47.79	5.43E-5		5.43E-3	18402	
AN102FEcomp2	14925	506	3000	298.290	2.4982	2.0085	119.40	148.51	1.39E-4		1.39E-2	7183	

sample activity sum 3.77E-1 uCi

Feed Displacement

Sample ID	Net counts	Error	Count time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	BV	total uCi
AN102-FD-1	21577	508	3000	431.33	2.414	2.4136	178.71	178.71	2.08E-4	4.1	2.08E-2	69.7	2.72E-1
AN102-FD-2	10894	356	3000	217.67	2.150	2.1498	101.25	101.25	1.18E-4	5.0	1.18E-2	70.6	1.72E-1
AN102-FD-3	5412	230	3000	108.03	2.058	2.0582	52.49	52.49	6.12E-5	5.9	6.12E-3	71.4	8.73E-2
AN102-FD-4	3454	161	3000	68.87	1.989	1.9892	34.62	34.62	4.04E-5	6.9	4.04E-3	72.5	7.12E-2
AN102-FD-5	2671	121	3000	53.21	2.013	2.0133	26.43	26.43	3.08E-5	7.9	3.08E-3	73.5	5.18E-2
AN102-FD-6	2765	101	3000	55.09	1.974	1.9744	27.90	27.90	3.25E-5	8.8	3.25E-3	74.4	4.77E-2
AN102-FD-7	2500	98	3000	49.79	1.982	1.9818	25.12	25.12	2.93E-5	9.6	2.93E-3	75.2	4.16E-2
AN102-FD-8	2659	83	3000	52.97	1.993	1.9932	26.58	26.58	3.10E-5	10.5	3.10E-3	76.1	4.57E-2
AN102-FD-9	2442	81	3000	48.63	2.002	2.0018	24.29	24.29	2.83E-5	11.6	2.83E-3	77.2	5.21E-2

sample activity sum 8.42E-1 uCi

DI Rinse

Sample ID	Net counts	Error	Count time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co, %	BV	total uCi
AN102-FDI-1	2624	75	3000	52.27	1.982	1.9818	26.38	26.38	3.08E-5	1.0	3.08E-3	78.2	4.95E-2
AN102-FDI-2	2744	74	3000	54.67	1.981	1.9805	27.60	27.60	3.22E-5	1.9	3.22E-3	79.1	4.83E-2
AN102-FDI-3	3001	77	3000	59.81	1.940	1.9396	30.84	30.84	3.60E-5	2.8	3.60E-3	80.0	5.47E-2
AN102-FDI-4	3038	74	3000	60.55	2.006	2.0063	30.18	30.18	3.52E-5	3.6	3.52E-3	80.9	5.18E-2
AN102-FDI-5	2229	67	3000	44.37	2.015	2.0147	22.02	22.02	2.57E-5	4.5	2.57E-3	81.7	3.83E-2
AN102-FDI-6	1079	44	1800	35.76	1.971	1.9707	18.14	18.14	2.12E-5	5.4	2.12E-3	82.6	3.20E-2
AN102-FDI-7	874	41	1800	28.92	1.989	1.9893	14.54	14.54	1.70E-5	6.4	1.70E-3	83.6	2.65E-2
AN102-FDI-8	764	38	1800	25.26	1.997	1.9968	12.65	12.65	1.47E-5	7.3	1.47E-3	84.5	2.31E-2
AN102-FDI-9	995	53	3000	19.69	1.972	1.9724	9.98	9.98	1.16E-5	8.3	1.16E-3	85.5	1.85E-2

sample activity sum 3.43E-1 uCi

Table C.2 contd

AN-102/C104 run starting 6/25/01.
Lead column, eluting phase

Sample ID	Net counts	Error	Count time (sec)	net cpm	Mass to dilute, g	Final mass, g	Mass to dilute, g	Final mass, g	mass for counting, g	counted mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	C/Co Average	% dev
AN102L-E1-d	33	6	3000	0.45	0.0849	5.0535	1	1	2.0341	3.42E-2	3.38E-2	1.32E+1	1.33E+1	1.25E-5	0.8	5.43E-5	-76.97
AN102L-E2-d	23	6	1800	0.6	0.0459	10.0329	1	1	2.0402	9.33E-3	9.22E-3	5.96E+1	6.04E+1	5.66E-5	1.5	6.01E-5	-5.80
AN102L-E3-d	11	3	3000	0.0	0.0915	10.1184	0.0943	10.103	1.9943	1.68E-4	1.66E-4	5.94E+1	6.01E+1	5.64E-5	2.5		
AN102L-E4-dd	41554	274	300	8311	0.0594	10.069	0.0956	10.1168	2.0128	1.12E-4	1.11E-4	7.41E+7	7.50E+7	7.03E+1	3.4	6.06E+1	15.86
AN102L-E5-dd	89493	547	300	17898	0.0954	10.1062	0.0923	10.0961	1.9884	1.72E-4	1.70E-4	1.04E+8	1.06E+8	9.90E+1	4.4		
AN102L-E6-dd	11816	118	300	2363	0.088	10.0969	0.0824	10.0991	1.9891	1.41E-4	1.40E-4	1.67E+7	1.69E+7	1.58E+1	5.4		
AN102L-E7-d	95507	545	300	19101	0.0798	10.0976	1	1	2.0368	1.61E-2	1.59E-2	1.19E+6	1.20E+6	1.13E+0	6.4	9.08E-1	23.93
AN102L-E8-d	11120	115	300	2224	0.0821	10.0916	1	1	2.0271	1.65E-2	1.63E-2	1.35E+5	1.36E+5	1.28E-1	7.4		
AN102L-E9-d	12291	120	500	1475	0.0631	5.0379	1	1	2.0502	2.57E-2	2.54E-2	5.74E+4	5.81E+4	5.45E-2	8.3	5.20E-2	4.84
AN102L-E10-d	7824	91	500	939	0.0481	5.0165	1	1	2.0513	0.0197	1.94E-2	4.77E+4	4.83E+4	4.53E-2	9.3	3.58E-2	26.51
AN102L-E11-d	5177	75	500	621	0.0932	5.0592	1	1	2.0445	0.0377	3.72E-2	16489	1.67E+4	1.56E-2	10.2		
AN102L-E12-d	5738	77	900	382	0.0846	5.0542	1	1	2.0517	0.0343	0.0339	11133	11266	1.06E-2	11.1		
AN102L-E13-d	8885	97	1800	296	0.0707	5.0325	1	1	2.0287	0.0285	0.0282	10384	10509	9.85E-3	12.1	8.63E-3	14.18
AN102L-E14-d	7105	86	1800	237	0.0933	5.0611	1	1	2.0398	0.0376	0.0372	6293	6368	5.97E-3	13.0		
AN102L-E15-d	5938	79	1800	198	0.073	5.0329	1	1	2.033	0.0295	0.0291	6705	6786	6.36E-3	14.0	5.73E-3	11.09
AN102L-E16-d	4910	70	1800	163	0.0897	5.0518	1	1	2.0278	0.0360	0.0356	4540	4594	4.31E-3	14.9		
AN102L-E17-d	4507	68	1800	150	0.091	5.0999	1	1	1.9974	0.0356	0.0352	4209	4260	3.99E-3	15.8	3.97E-3	0.60
AN102L-E18-dr	Samples were taken during re-preparation aliquoting						–	–	–	–	–	–	–	–	16.8		
AN102L-E19-dr	Samples were taken during re-preparation aliquoting						–	–	–	–	–	–	–	–	18.0		
AN102L-E20-d	6850	84	1800	228	0.0853	4.993	1	1	2.0199	0.0345	0.0341	6611	6690	6.27E-3	18.9		

Selected Re-preps

Sample ID	Net counts	Error	Count time (sec)	net cpm	Mass to dilute, g	Final mass, g	Mass to dilute, g	Final mass, g	mass for counting, g	counted mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV
AN102L-E1-dr	89	10	1800	2.76	0.0699	5.0799	1	1	1.9794	2.72E-2	2.69E-2	1.01E+2	1.02E+2	9.60E-5	0.8
AN102L-E2-dr	62	8	3000	1.0	0.0782	10.17	1	1	1.9997	1.54E-2	1.52E-2	6.70E+1	6.78E+1	6.36E-5	1.5
AN102L-E4-ddr	14528	161	100	8717	0.0843	10.1655	0.0993	10.212	2.0098	1.62E-4	1.60E-4	5.38E+7	5.44E+7	5.10E+1	3.4
AN102L-E7-dr	20196	291	100	12117	0.0836	10.1564	1	1	2.0211	1.66E-2	1.64E-2	7.28E+5	7.37E+5	6.91E-1	6.4
AN102L-E9-dr	3286	59	100	1971	0.0951	5.0912	1	1	2.0245	3.78E-2	3.74E-2	5.21E+4	5.28E+4	4.95E-2	8.3
AN102L-E10-dr	3378	59	200	1013	0.0909	5.0363	1	1	2.025	0.0365	3.61E-2	2.77E+4	2.81E+4	2.63E-2	9.3
AN102L-E13-dr	4491	69	900	299	0.0966	5.1019	1	1	2.0244	0.0383	0.0379	7806	7899	7.41E-3	12.1
AN102L-E15-dr	6167	80	1800	205	0.097	5.1037	1	1	2.0133	0.0383	0.0378	5367	5431	5.09E-3	14.0
AN102L-E17-dr	4507	68	1800	150	0.091	5.0999	1	1	2.0214	0.0361	0.0356	4159	4209	3.95E-3	15.8
AN102L-E18-dr	2294	47	1000	137	0.0903	5.1065	1	1	2.0151	0.0356	0.0352	3857	3903	3.66E-3	16.8
AN102L-E19-dr	21536	155	1800	718	0.0957	5.0961	1	1	2.0006	0.0376	0.0371	19102	19331	1.81E-2	18.0

Table C.2 contd

Deionized water rinse following elution

Sample ID	Net counts	Error	Count time (sec)	net cpm	mass, g	mL	net cpm/g	net cpm/mL	C/Co	BV	BV post Elution	uCi, total
AN102-EDI-1	46715	243	1000	2803	1.9978	2.0000	1403	1401	1.31E-3	0.9	19.9	2.0805795
AN102-EDI-2	26890	176	1000	1613	2.0194	2.0000	799	807	7.56E-4	2.2	21.1	1.5758021
AN102-EDI-3	16050	136	1000	963	1.9944	2.0000	483	481	4.51E-4	3.2	22.2	0.7947607
AN102-EDI-4	12914	120	1000	775	2.0012	2.0000	387	387	3.63E-4	4.2	23.1	0.5793206
AN102-EDI-5	12197	117	1000	732	1.9976	2.0000	366	366	3.43E-4	5.1	24.0	0.5163613
AN102-EDI-6	11843	114	1000	710	1.9855	2.0000	358	355	3.33E-4	5.9	24.9	0.47611
										sample activity sum		6.0229341

Regeneration with 0.25 M NaOH

Sample ID	Net counts	Error	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	C/Co
AN102-RGN	52014	262	1000	3121	2.0012	1.9893	1559	1569	1.47E-3

Regeneration with 0.25 M NaOH Analytical Lab Data

Sample ID	CMC ID	Cs-137, μCi/mL	error, %	C/Co	BV	μCi total
AN102-RGN	01-1732	7.54E-02	3	4.68E-04	25.9	3.82

Table C.3. AN-102/C-104 Column Samples CMC Analytical Results Summary

AN-102/C-104 column run CMC Analytical data

Lead column, loading phase

Sample ID	CMC ID	Cs-137, μCi/mL	error, %	C/Co	% C/Co	BV	DF	
AN102/C104L-F0		161	3	1.00E+0				From ASR 6174
AN102/C104L-F2	01-01714	2.95E-04	5	1.83E-6	1.83E-04	13.2	5.46E+5	
AN102/C104L-F5	01-01715	2.55E-04	8	1.58E-6	1.58E-04	40.9	6.31E+5	
AN102/C104L-F8	01-01716	2.10E-04	6	1.30E-6	1.30E-04	67.1	**7.67E+5**	Maximum
AN102/C104P-F2	01-01717	1.40E-02	3	8.70E-5	8.70E-03	13.0	1.15E+4	
AN102/C104P-F6	01-01718	1.55E-02	2	9.63E-5	9.63E-03	48.7	1.04E+4	
AN102/C104P-F8	01-01719	1.21E-02	3	7.52E-5	7.52E-03	65.6	1.33E+4	
AN102/C104 comp	01-1733	1.42E-02	4.84	8.82E-5	8.82E-03		**1.13E+4**	composite

Feed Displacement

Sample ID	CMC ID	Cs-137, μCi/mL	error, %	C/Co	% C/Co
AN102/C104-FD-1	01-1721	1.73E-2	2	1.07E-4	1.07E-2
AN102/C104-FD-5	01-1722	1.76E-3	2	1.09E-5	1.09E-3
AN102/C104-FD-9	01-1723	1.81E-3	3	1.12E-5	1.12E-3
AN102/C104-FDI-4	01-1724	1.88E-3	3	1.17E-5	1.17E-3
AN102/C104-FDI-8	01-1725	7.03E-4	3	4.37E-6	4.37E-4

Lead column, eluting phase

Sample ID	CMC ID	Cs-137, μCi/mL	error, %	C/Co	BV
AN102/C104LE-4DR	01-1726	4.63E+3	2	2.88E+1	3.43E+0
AN102/C104LE-5DD	01-1727	8.52E+3	2	5.29E+1	4.41E+0
AN102/C104LE-6DD	01-1728	1.13E+3	3	7.02E+0	5.38E+0
AN102/C104LE-9DR	01-1729	3.43E+0	2	2.13E-2	8.33E+0
AN102/C104LE-12D	01-1730	7.21E-1	3	4.48E-3	1.11E+1
AN102/C104L-EDI2	01-1731	5.64E-02	2	3.50E-4	

Table C.4. Integration Column Breakthrough

AN-102/C-104 Cs IX Lead Column Breakthrough Curve Integration

Lead Col Bed Volume in AN102/C104 Feed = 9.9 mL
Lead Col Bed Volume in 0.25 M NaOH = 10.4 mL
Activity of Cs-137 in Feed = 161 uCi/mL

Sample	Processed Vol (mL)	Cs-137 C/Co	Cs-137 Conc (µCi/mL)	Δ Vol (mL)	C/Co Midpoint	Midpoint Conc (µCi/mL)	Area (µCi)	Cs-137 (µCi/mL) CMC Analysis
AN102L-F1	48.4	-1.07E-6	-1.73E-4	48.4	-1.07E-6	-1.73E-4	-8.36E-3	
AN102L-F2	137.0	1.19E-6	1.92E-4	88.7	5.78E-8	9.30E-6	8.25E-4	2.95E-4
AN102L-F3	230.6	3.43E-6	5.51E-4	93.5	2.31E-6	3.72E-4	3.47E-2	
AN102L-F4	330.9	1.29E-6	2.08E-4	100.4	2.36E-6	3.80E-4	3.81E-2	
AN102L-F5	425.9	5.43E-6	8.74E-4	94.9	3.36E-6	5.41E-4	5.14E-2	2.55E-4
AN102L-F6	516.7	4.07E-7	6.56E-5	90.9	2.92E-6	4.70E-4	4.27E-2	
AN102L-F7	607.1	3.41E-6	5.49E-4	90.4	1.91E-6	3.07E-4	2.78E-2	
AN102L-F8	697.4	-3.58E-8	-5.77E-6	90.3	1.69E-6	2.72E-4	2.45E-2	2.10E-4

sum through 67 BV 2.20E-1 µCi Cs-137

AN-102/C-104 Cs IX Lag Column Breakthrough Curve Integration

Lag Col Bed Volume in AN-102/C-104 Feed = 9.9 mL
Lag Col Bed Volume in 0.25 M NaOH = 10.5 mL
Activity of Cs-137 in Feed = 161 uCi/mL

Sample	Processed Vol (mL)	Cs-137 C/Co	Cs-137 Conc (µCi/mL)	Δ Vol (mL)	C/Co Midpoint	Midpoint Conc (µCi/mL)	Area (µCi)
AN102P-F1	48.8	1.92E-4	3.10E-2	48.8	1.92E-4	3.10E-2	1.51E+0
AN102P-F2	135.0	1.60E-4	2.57E-2	86.2	1.76E-4	2.83E-2	2.44E+0
AN102P-F3	225.8	1.73E-4	2.79E-2	90.8	1.66E-4	2.68E-2	2.43E+0
AN102P-F4	323.2	2.19E-4	3.52E-2	97.4	1.96E-4	3.15E-2	3.07E+0
AN102P-F5	415.5	1.91E-4	3.08E-2	92.3	2.05E-4	3.30E-2	3.05E+0
AN102P-F6	506.0	1.60E-4	2.57E-2	90.5	1.75E-4	2.82E-2	2.55E+0
AN102P-F7	593.6	1.46E-4	2.35E-2	87.6	1.53E-4	2.46E-2	2.15E+0
AN102P-F8	682.1	1.75E-4	2.81E-2	88.5	1.60E-4	2.58E-2	2.29E+0

sum through 67 BV 1.95E+1 µCi Cs-137

Table C.5. Cs Recovery in Eluate

AN-102 / C-104 Cs-137 Recovery in Eluate

Cs-137 loaded:	753	mL load volume
	161	uCi/mL Cs-137 concentration
	35	uCi left on column from AP-101DF run
	1.21E+5	uCi total loaded on the lead column

Sample ID	Eluate volume, mL	Cs, µC/mL	CMC Cs µCi	
AN102L-E4-dd	9.6517	4.63E+03	**4.47E+4**	
AN102L-E5-dd	10.3193	8.52E+03	**8.79E+4**	
AN102L-E6-dd	10.2201	1.13E+03	**1.15E+4**	
AN102L-E7-d	10.8062			
AN102L-E8-d	10.0951			
AN102L-E9-d	10.1534	3.43E+00	**3.48E+1**	
		sum	**1.44E+5**	µCi from just the CMC measured peak
			119%	recovery

Eluate composite 02-0779

Isotope	µCi/mL	SpA, uCi/µg	µg/mL		
Cs-134	<4.E-3	2194	<2.E-6	197	mL total eluate
Cs-137	665	87	7.644	131005	µCi total recovered
				108%	**recovered Cs-137 measured in eluate composite**

Thermal ionization mass spectrometry weight percent distribution ratio

Sample #	02-0779	Cs eluate		
	Cs-133	**Cs-135**	**Cs-137**	
02-779	0.6040	0.1667	0.2293	
02-779 dup	0.6016	0.1674	0.2310	
avg	0.6028	0.1671	0.2302	
std	0.0017	0.00049	0.0012	
rpd	0.398	0.419	0.739	Sum
Sample #	02-779	Cs eluate		
µg/mL Cs	20.016	5.547	7.642	3.32E+1
M Cs	1.50E-4	4.11E-5	5.58E-5	2.47E-4

Effluent composite 02-0777

Isotope	µCi/mL	SpA, uCi/µg	µg/mL		
Cs-134	<4.E-3	2194	<2.E-6	753	mL effluent volume
Cs-137	1.42E-02	87	1.63E-04	1.07E+1	µCi total recovered
				8.8E-5%	**recovered Cs-137 measured in effluent composite**

Sample #	02-0779	Cs isotopic distribution is constant		
	Cs-133	**Cs-135**	**Cs-137**	
02-779	0.6040	0.1667	0.2293	
02-779 dup	0.6016	0.1674	0.2310	
avg	0.6028	0.1671	0.2302	
std	0.0017	0.00049	0.0012	
rpd	0.398	0.419	0.739	Sum
Sample #	02-777	Cs effluent		
µg/mL Cs	4.27E-4	1.18E-4	1.63E-4	7.09E-4
M Cs	3.21E-9	8.78E-10	1.19E-9	5.28E-9

Appendix D

Analytical Data

Appendix D: Analytical Data

Table D.1. Sample Identification

Sample Description	ASR	RPL ID	Sample Identification	Extended Sample Description
Batch Contact Testing				
Batch contact with SL-644 010319SMC-IV-73 212-425 µm particle size H form resin	6130	01-1341	AN102/104-644-F/A	Batch contact with SL-644, no spike
		01-1342 – 01-1343	AN102/104S-S1-644-F/A and duplicate	Batch contact with SL-644, 1E-3 M Cs spike
		01-1342	AN102/104-S2-644-F/A	Batch contact with SL-6444, 5E-3 M Cs spike
		01-1345	ANC102/104-C-F/A	Batch contact control (no exchanger) unspiked
		01-1346	ANC102/104-S1C-F/A	Batch contact control (no exchanger) 1E-3M Cs
		01-1347	ANC102/104-S2C-F/A	Batch contact control (no exchanger) 5E-3 M Cs
Batch contact with IE-911 Na form resin		01-1348 – 01-1349	AN102-CST-F/A and duplicate	Batch contact with CST no spike
		01-1350 – 01-1351	AN102-S3-CST-F/A and duplicate	Batch contact with CST, 1E-3M Cs spike
		01-1352 – 01-1353	AN102-S4-CST-F/A and duplicate	Batch contact with CST, 5E-3M Cs spike
		01-1354	AN102CST-C-F/A	Batch contact CST control, no spike
		01-1355	AN102CST-S3C-F/A	Batch contact CST control, 1E-3 M Cs spike
		01-1356	AN102CST-S4C-F/A	Batch contact with CST, 5E-3 M Cs spike
Column Run				
Initial Feed Sample, AN-102/C-104	6107	01-1014 -- 01-1015	LS-12 and LS-13	AN102/C-104 filtrate composite/Cs IX feed
Initial Feed Sample, AN-102/C-104	6130	01-1345	ANC102/104-C-F/A	AN102/C-104 filtrate composite/Cs IX feed
Load - Elution samples	6174	01-1714 – 01-1716	AN102/C104L-F2, -F5, -F8	Lead column load samples
		01-1717 – 01-1719	AN102/C104P-F2, -F6, -F8	Lag column load samples
		01-1720	AN102/C104-F0	Feed sample
		01-1721 – 01-1723	AN102/C104-FD1, FD5, FD9	Feed displacement samples
		01-1724 – 01-1725	AN102/C104-FDI-4, FDI-8	Water rinse samples
		01-1726 – 01-1730	AN102/C104-LE-4-DR, -5D, -6D, -9DR, -12D	Lead column elution analytical samples
		01-1731	AN102/C104-EDI-2	Elution rinse sample
		01-1732	AN102/C104-RGN	Regeneration solution analytical sample
AN-102/C-104 effluent composite		01-1733	Fcomp#2	AN-102/C-104 Cs IX effluent composite (GEA)
AN-102/C-104 effluent composite	6281	02-0777	AN2-Tc-0-C	AN-102/C-104 Cs IX effluent composite
AN-102/C-104 Cs eluate	6281	02-0779	AN-102/C-104-CsE-Comp1	AN-102/C-104 Cs eluate analytical sample

Project / WP#:	42365 / W57984
ASR#:	6107
Client:	R. Hallen
Total Samples:	14 liquids

RPL#:	01-01003	01-01016
Client ID:	"LS-01"	"LS-14"
Sample Preparation: PNL-ALO-128 (1mL/26mL or 1.2g/26mL)		

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

Analyst: D.R. Sanders

Analysis Date (File):
05-25-2001 (A0676)
06-15-2001 (A0682)
06-19-2001 (A0684)
06-20-2001 (A0~~8~~685)

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

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

9-20-01
Reviewed by

Jerry L. Wagner 9-25-01
Concur

Fourteen liquid samples from Analytical Service Request (ASR) 6107 were prepared by acid digestion per PNL-ALO-128 in the Shielded Analytical Laboratory (SAL). The samples were prepared by using nominal 1.0 mL of sample and diluting to a final volume of about 26 mL. The final volume was calculated by using the mass and density of the resulting digestate.

In the ASR, Al, Ba, Ca, Cd, Cr, Fe, K, La, Mg, Mn, Na, Ni, P, Pb, and Sr were identified as analytes of interest for this work. The quality control (QC) results for each of these analytes have been evaluated and are presented below. Analytes other than those detected as part of the ICPAES analysis are reported, but have concentrations less than the method detection limit (MDL) and have not been fully evaluated for QC performance.

The attached ICPAES Results (9 pages; 2 pages from each of the analysis runs on 5-25, 6-19, and 6-20, and 3 pages from analysis run on 6-15) presents the final results. Results are from the direct measurement of the digestates, except for Na which required an additional 5x dilution to bring the Na concentration within the ICPAES linear range. The ICPAES measurement results are reported in µg/g of liquid sample (as requested by the ASR) and have been corrected for all dilutions resulting from sample processing. It should be noted that the preliminary results reported were presented on a µg/mL basis.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan. A digestion processing blank, laboratory control sample (blank spike), matrix spike, and duplicate were prepared with the sample for each processing batch. The blank spikes was prepared by using 3 mL of a custom multi-element solutions "010514i901 and 010514i902" per 26 mL digestate volume, and the matrix spikes were prepared by using 1 mL of the same multi-element solutions.

Process Blank:

Concentration of analytes of interest measured in the three process blanks were all within acceptance criteria of ≤ EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample.

Blank Spike (laboratory control sample):

The blank spike recovery for analytes of interest was within the acceptance criteria of 80% to 120%, except for Na for the 6-19-2001 analysis which recovered slightly high at 121%.

Duplicate RPD (Relative Percent Difference):

For those analytes of interest measured above the EQL, the RPDs were within the acceptance criteria of less than 20%. Even for analytes with concentration between the method detection limit (MDL) and the EQL, the RPDs are quite good, with only the Mg for LS-06 exceeding the 20% criteria.

Matrix Spiked Sample:

Matrix spike were prepared for LS-02, LS-06, LS-10, and LS-12. Except for Al and Na, which had spike concentration less than 20% of the sample concentration, the analytes of interest meet the matrix spike recovery criteria of 75% to 125%. Post spiking or serial dilution is required for the Al and Na.

Post-Spiked Samples (Spike A Elements):

Post spiking was performed on LS-02, LS-06 and LS-10. All post-spiked analytes of interest in samples tested were recovered within tolerance of 75% to 125%, except Al and Na. The post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the sample selected for post spiking, the spike concentration for Al and Na was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Post-Spiked Samples (Spike B Elements):

The post spike recovery for La, which is the only Spike B analyte of interest, were within tolerance of 75% to 125%.

Serial dilution:

Serial dilution was required for Al, Na, Sr, since for these analytes the post spike concentrations were less than 20% of the sample concentration (i.e., recoveries could not be evaluated). These analytes demonstrated a percent difference (%D) within the acceptance criteria of ±10% after correcting for dilution for all the serial dilutions measured. In some case the samples were not have enough serial dilutions to be able to calculate the Na %Diff (i.e., at all dilutions except for the highest dilution, the Na was over range).

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

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

	Run Date=	5/25/2001	5/25/2001	5/25/2001	5/25/2001	5/25/2001	5/25/2001	5/25/2001
	Multiplier=	20.6	103.6	512.8	103.4	103.9	102.8	24.3
	RPL#=	01-1014-PB	01-1003	01-1003-D	01-1014	01-1014-D	01-1015	01-1016
Det. Limit	Client ID=	Process Blank (ALO-128)	LS-01	LS-01-Dup	LS-12	LS-12-Dup	LS-13	LS-14
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	[4.0]	7,230	7,320	6,960	6,930	6,800	1,180
0.010	Ba	--	--	--	--	--	--	--
0.250	Ca	--	[160]	[150]	[130]	[120]	[120]	[34]
0.015	Cd	--	22.7	[23]	22.9	22.2	22.1	3.96
0.020	Cr	--	89.2	[89]	94.0	93.2	91.2	15.8
0.025	Fe	--	[7.3]	--	[3.2]	[3.2]	[3.3]	[1.3]
2.000	K	--	[720]	--	[710]	[680]	[690]	[130]
0.050	La	--	--	--	--	--	--	--
0.100	Mg	--	--	--	--	--	--	--
0.050	Mn	--	--	--	--	--	--	--
0.150	Na	48.2	95,700	99,400	91,100	91,400	90,200	22,900
0.030	Ni	--	165	167	163	162	158	27.8
0.100	P	--	685	670	667	656	638	99.3
0.100	Pb	--	[61]	--	[55]	[52]	[51]	[13]
0.015	Sr	--	--	--	74.5	74.5	73.0	15.2
	Other Analytes							
0.025	Ag	--	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--
0.050	B	39.4	63.1	[60]	58.8	59.3	57.5	87.0
0.010	Be	--	--	--	--	--	--	--
0.100	Bi	--	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--
0.050	Co	--	--	--	--	--	--	--
0.025	Cu	--	[3.7]	--	[6.7]	[6.1]	[6.0]	[0.94]
0.050	Dy	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--	--
0.050	Mo	--	[19]	--	[20]	[19]	[19]	[5.0]
0.100	Nd	--	--	--	--	--	--	--
0.750	Pd	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--
0.500	Si	[81]	[160]	--	[160]	[160]	[160]	669
1.500	Sn	--	--	--	--	--	--	--
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	--	--	--	[5.5]	[5.4]	[5.5]	[2.2]
0.050	Zr	--	--	--	--	--	--	--

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

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

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

QC Performance 5/25/2001

Criteria>	<20%	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%	< +/-10%	< +/-10%
QC ID=	01-1003 & 01-1003D	01-1014 & 01-1014D	010514i901 010514i902 LCS/BS	01-1014 MS	01-1014 + Post Spike A	01-1014 + Post Spike B	01-1003 @5/@25 Serial Dil	01-1014 @5/@25 Serial Dil	01-1015 @5/@25 Serial Dil	01-1016 @1/@5 Serial Dil
Analytes	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff	%Diff	%Diff	%Diff
Al	3.4	0.0	96.7	n.r.	n.r.		-0.5	-0.2	-0.9	2.0
Ba			95.6	98.8	102.8					
Ca	1.1	0.4	95.6	102.2	104.1					
Cd	2.1	2.7	95.2	100.2	103.1					
Cr	2.3	0.5	96.0	101.8	105.4					
Fe		1.8	98.5	104.0	108.1					
K		4.0	93.3	98.5	97.9					
La			92.7	98.4		98.4				
Mg			98.2	106.6	111.3					
Mn			98.8	106.9	109.7					
Na	6.0	0.9	97.4	n.r.	n.r.		-2.4	-1.7	-2.8	4.1
Ni	3.3	0.3	97.6	106.3	108.4					
P	0.1	1.3	95.0	101.9	102.1					
Pb		3.4	97.7	103.1	105.8					
Sr		0.5	95.7	105.3	103.0					
Other Analytes										
Ag			91.2	97.3	100.4					
As					105.3					
B	3.3	1.2			106.5					
Be					104.0					
Bi			95.4	100.1	103.6					
Ce						99.1				
Co					109.5					
Cu		8.3	97.7	100.9	107.4					
Dy						101.7				
Eu						109.0				
Li					102.2					
Mo		1.0			105.2					
Nd			92.5	96.1		98.2				
Pd			93.9	93.0		89.7				
Rh			94.7	93.3		97.4				
Ru			91.5							
Sb					102.5					
Se					108.4					
Si		4.1	124.5	105.8	121.4					
Sn										
Te										
Th						102.3				
Ti			92.9	96.2	101.1					
Tl					104.0					
U			92.4	93.9		96.0				
V					99.5					
W										
Y					100.7					
Zn		1.8	96.6	102.0	105.8					
Zr			96.2	102.0	105.2					

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

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

	Run Date=	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001	6/15/2001
	Multiplier=	1.0	19.6 (Na98.2)	20.3 (Na101.7)	20.1 (Na100.3)	20.3 (Na101.3)	20.0 (Na100.0)	20.1 (Na100.6)	19.9 (Na99.6)	19.1 (Na95.6)
	RPL#=	01-1004-B	01-1004	01-1004-D	01-1005	01-1005-D	01-1006	01-1006-D	01-1007	01-1007-D
Det. Limit	Client ID=	Process Blank (ALO-128)	LS-02	LS-02 Dup	LS-03	LS-03 Dup	LS-04	LS-04 Dup	LS-05	LS-05 Dup
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	17.5	6,710	6,750	6,500	6,570	6,800	6,640	6,750	6,830
0.010	Ba	--	--	--	--	--	--	--	--	--
0.250	Ca	[5.9]	119	121	105	108	117	113	114	116
0.015	Cd	--	20.9	21.0	20.1	20.3	21.0	20.7	20.9	21.1
0.020	Cr	--	76.7	77.5	80.4	80.8	84.0	82.3	83.9	85.0
0.025	Fe	[1.1]	[4.7]	[4.6]	[2.6]	[2.4]	[2.6]	[2.6]	[2.6]	[2.4]
2.000	K	--	814	824	809	816	841	815	817	828
0.050	La	--	[2.2]	[2.3]	--	--	--	--	--	--
0.100	Mg	--	--	--	--	[2.2]	[2.3]	--	--	--
0.050	Mn	--	--	--	--	--	--	--	--	--
0.150	Na	61.1	89,900	90,900	86,200	87,600	91,000	90,200	91,500	92,000
0.030	Ni	--	145	147	143	143	148	145	148	149
0.100	P	--	631	638	624	628	647	634	641	648
0.100	Pb	--	57.5	58.4	42.5	42.2	46.2	45.3	46.0	46.4
0.015	Sr	--	161	168	100	102	95.5	91.0	84.9	83.5
	Other Analytes									
0.025	Ag	--	--	--	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--	--	--
0.050	B	27.9	64.4	75.5	60.1	93.5	95.3	58.7	53.5	65.1
0.010	Be	--	--	--	--	--	--	--	--	--
0.100	Bi	--	[2.4]	[2.2]	--	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--	--	--
0.050	Co	--	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]	[1.5]
0.025	Cu	--	[4.3]	[4.3]	[4.2]	[4.2]	[4.4]	[4.2]	[4.3]	[4.3]
0.050	Dy	--	--	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--	--	--
0.030	Li	--	--	--	--	--	--	--	--	--
0.050	Mo	--	17.6	17.7	17.3	17.4	18.1	17.7	18.1	18.2
0.100	Nd	--	[6.8]	[7.0]	[3.5]	[3.4]	[3.2]	[3.1]	[2.9]	[2.9]
0.750	Pd	--	--	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--	--	--
0.500	Si	103	243	259	217	295	372	228	192	213
1.500	Sn	--	--	--	--	--	--	--	--	--
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	[1.1]	[2.2]	[2.0]	[1.4]	[1.1]	[1.1]	[1.5]	[1.4]	[1.3]
0.050	Zr	--	[2.0]	[2.0]	--	--	--	--	--	--

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

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

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

QC Performance 6/15/2001 - RPDs and LCS/BS Recovery

Criteria>	<20%	<20%	<20%	<20%	80% - 120%
QC ID=	01-1004 & 01-1004D	01-1005& 01-1005D	01-1006 & 01-1006D	01-1007 & 01-1007D	010514i901 010514i902 LCS/BS
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec
Al	3.9	1.0	1.0	2.8	112.0
Ba					99.0
Ca	2.7	3.3	2.2	3.8	99.7
Cd	3.9	0.8	0.1	2.5	99.2
Cr	3.5	0.6	0.6	2.9	99.2
Fe	**7.3**	**4.9**	**2.9**	**8.0**	101.2
K	3.4	0.9	1.8	3.0	101.7
La	**0.5**				98.4
Mg					101.2
Mn					102.4
Na	1.1	1.6	0.9	0.5	119.2
Ni	3.4	0.6	0.5	2.5	99.5
P	3.4	0.7	0.6	2.6	98.0
Pb	2.9	0.6	0.6	2.6	100.6
Sr	0.6	1.9	3.4	0.1	99.0
Other Analytes					
Ag					**59.3**
As					
B	11.4	43.5	46.2	21.2	
Be					
Bi	**13.9**				98.9
Ce					
Co	**0.5**	**2.0**	**0.2**	**1.6**	
Cu	**4.2**	**0.7**	**2.5**	**3.5**	100.0
Dy					
Eu					
Li					
Mo	3.5	0.4	0.8	2.6	
Nd	**2.0**	**3.6**	**0.0**	**0.1**	
Pd					99.5
Rh					100.2
Ru					96.4
Sb					
Se					
Si	2.0	30.5	46.8	12.2	**159.1**
Sn					
Te					
Th					
Ti					95.9
Tl					
U					99.0
V					
W					
Y					
Zn	**16.3**	**22.9**	**30.7**	**0.9**	98.2
Zr	**2.0**				98.7

Shaded results exceed acceptance criteria

Bold and unshaded RPDs indicate one or both results <EQL.

QC Performance 6/15/2001- MS & PS Recovery and %Diff

Criteria>	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%	< +/-10%	< +/-10%
QC ID=	01-1004 MS	01-1004 + Post Spike A	01-1004 + Post Spike B	01-1004 @1/@5 Serial Dil	01-1005 @1/@5 Serial Dil	01-1006 @1/@5 Serial Dil	01-1007 @1/@5 Serial Dil
Analytes	%Rec	%Rec	%Rec	%Diff	%Diff	%Diff	%Diff
Al	n.r.	n.r.		3.7	1.4	2.2	2.7
Ba	93.0	95.3					
Ca	98.5	97.7					
Cd	96.7	99.6					
Cr	91.5	99.1					
Fe	97.2	99.3					
K	96.1	96.0					
La	95.6		97.0				
Mg	99.3	103.4					
Mn	103.4	103.3					
Na	n.r.	n.r.		n.m.	n.m.	n.m.	n.m.
Ni	88.5	99.0					
P	95.5	96.4					
Pb	97.8	100.3					
Sr	n.r.	99.3					
Other Analytes							
Ag	95.7	96.1					
As		100.2					
B		99.6					
Be		97.6					
Bi	96.6	97.7					
Ce			98.7				
Co		101.1					
Cu	96.8	100.0					
Dy			98.5				
Eu			105.7				
Li		98.2					
Mo		98.0					
Nd			99.8				
Pd	103.0		91.1				
Rh	101.6		96.2				
Ru	105.0						
Sb		97.8					
Se		99.9					
Si	120.1	107.2					
Sn							
Te							
Th			98.1				
Ti	91.1	93.2					
Tl		95.4					
U	95.9		96.6				
V		94.4					
W							
Y		96.0					
Zn	98.6	102.1					
Zr	97.4	97.6					

Shaded results exceed acceptance criteria

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

n.m. = not measured; insufficient dilutions prepared to evaluate %Diff.

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

	Run Date=	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001	6/19/2001
	Multiplier=	20.3	20.1 (Na100.3)	20.1 (Na100.6)	21.1 (Na105.4)	20.5 (Na102.6)	20.8 (Na103.9)	20.5 (Na102.3)	19.9 (Na99.4)	19.4 (Na97.2)
	RPL#=	01-1008-B	01-1008	01-1008-D	01-1009	01-1009-D	01-1010	01-1010-D	01-1011	01-1011-D
Det. Limit	Client ID=	Proc. Blk	LS-06	LS-06	LS-07	LS-07	LS-08	LS-08	LS-09	LS-09
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	[4.6]	7,180	6,990	6,640	6,670	6,840	6,960	6,610	6,600
0.010	Ba	[0.22]	[0.23]	--	--	[0.22]	--	[0.23]	--	[0.27]
0.250	Ca	--	127	122	120	123	122	125	122	120
0.015	Cd	--	22.4	21.8	21.1	20.9	21.4	22.0	20.8	20.6
0.020	Cr	--	90.3	88.5	85.2	84.3	87.0	88.6	84.5	84.2
0.025	Fe	[0.65]	[3.2]	[2.7]	[4.6]	[4.7]	[5.0]	5.83	[4.8]	[4.8]
2.000	K	--	895	864	817	794	798	812	769	768
0.050	La	--	--	--	[1.2]	--	--	--	--	--
0.100	Mg	--	[2.7]	[2.1]	--	[2.1]	--	--	[2.1]	--
0.050	Mn	--	--	--	--	--	--	--	--	--
0.150	Na	46.5	98,700	97,200	92,500	92,500	94,500	97,600	93,800	94,200
0.030	Ni	--	159	156	149	147	152	154	147	147
0.100	P	--	665	649	629	622	610	614	622	620
0.100	Pb	--	49.4	48.3	46.5	43.7	45.8	46.4	44.4	44.2
0.015	Sr	--	87.6	86.3	185	185	174	178	160	161
	Other Analytes									
0.025	Ag	--	--	--	--	--	--	--	--	--
0.250	As	--	--	--	--	--	--	--	--	--
0.050	B	36.7	104	52.4	43.5	88.0	43.4	69.0	70.7	65.0
0.010	Be	--	--	--	--	--	--	--	--	--
0.100	Bi	--	[2.9]	[2.6]	[2.6]	--	--	--	--	--
0.200	Ce	--	--	--	--	--	--	--	--	--
0.050	Co	--	[1.7]	[1.7]	[1.6]	[1.4]	[1.5]	[1.5]	[1.4]	[1.4]
0.025	Cu	--	[4.7]	[4.5]	[4.4]	[4.2]	[4.4]	[4.5]	[4.3]	[4.3]
0.050	Dy	--	--	--	--	--	--	--	--	--
0.100	Eu	--	--	--	--	--	--	--	--	--
0.030	Li	--	[0.61]	[0.65]	--	--	--	--	--	--
0.050	Mo	--	19.0	18.8	17.9	17.5	18.1	18.5	17.6	17.6
0.100	Nd	--	[4.4]	[4.5]	[4.8]	[3.4]	[3.4]	[3.5]	[3.2]	[3.1]
0.750	Pd	--	--	--	--	--	--	--	--	--
0.300	Rh	--	--	--	--	--	--	--	--	--
1.100	Ru	--	--	--	--	--	--	--	--	--
0.500	Sb	--	--	--	--	--	--	--	--	--
0.250	Se	--	--	--	--	--	--	--	--	--
0.500	Si	[86]	385	206	199	317	220	268	300	245
1.500	Sn	--	--	--	--	--	--	--	--	--
1.500	Te	--	--	--	--	--	--	--	--	--
1.000	Th	--	--	--	--	--	--	--	--	--
0.025	Ti	--	--	--	--	--	--	--	--	--
0.500	Tl	--	--	--	--	--	--	--	--	--
2.000	U	--	--	[40]	--	--	--	--	--	--
0.050	V	--	--	--	--	--	--	--	--	--
2.000	W	--	--	--	--	--	--	--	--	--
0.050	Y	--	--	--	--	--	--	--	--	--
0.050	Zn	[1.1]	[1.6]	[1.7]	[3.9]	[3.4]	[4.2]	[4.2]	[4.3]	[4.5]
0.050	Zr	--	[1.2]	[1.1]	[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).

QC Performance 6/19/2001

Criteria>	<20%	<20%	<20%	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-1008 & 01-1008D	01-1009 & 01-1009D	01-1010 & 01-1010D	01-1011 & 01-1011D	010514i901 010514i902 LCS/BS	01-1008 MS	01-1008 + Post Spike A	01-1008 + Post Spike B	01-1008 @1/@5 Serial Dil
Analytes	RPD (%)	RPD (%)	RPD (%)	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	2.8	0.5	1.7	0.1	111.0	n.r.	n.r.		2.8
Ba					99.2	92.6	101.9		
Ca	4.3	2.0	2.6	1.3	99.7	96.7	102.5		
Cd	2.5	0.7	2.4	0.8	97.9	95.1	103.3		
Cr	2.0	1.1	1.8	0.3	97.8	89.8	104.5		
Fe	**17.6**	**1.9**	**14.9**	**0.5**	101.3	96.6	105.0		
K	3.6	2.8	1.8	0.0	99.6	95.4	100.6		
La					98.2	97.6		100.5	
Mg	**25.1**				100.9	97.5	108.4		
Mn					100.9	101.1	107.4		
Na	1.5	0.0	3.2	0.4	**121.1**	n.r.	n.r.		**n.m.**
Ni	1.9	1.4	1.5	0.1	99.9	84.2	109.4		
P	2.4	1.1	0.6	0.4	96.1	91.7	102.0		
Pb	2.3	6.2	1.3	0.5	102.3	99.4	107.8		
Sr	1.5	0.1	2.0	0.4	99.4	94.1	104.3		
Other Analytes									
Ag					**55.3**	94.1	99.1		
As							103.6		
B	**66.2**	**67.7**	**45.5**	**8.3**			105.3		
Be							100.9		
Bi	10.3				96.0	93.4	99.8		
Ce								102.7	
Co	**0.3**	**12.6**	**0.7**	**0.7**			107.6		
Cu	**5.6**	**4.5**	**1.7**	**0.3**	99.4	95.8	105.3		
Dy								102.0	
Eu								109.7	
Li	10.8						102.3		
Mo	1.4	2.5	1.9	0.3			103.8		
Nd	**3.0**	33.6	**1.2**	**1.7**				102.9	
Pd					97.0	97.6		88.2	
Rh					97.8	99.8		100.8	
Ru					96.0	104.6			
Sb							101.5		
Se							104.4		
Si	**60.5**	**45.8**	**19.6**	**20.0**	**154.8**	96.9	114.4		
Sn									
Te									
Th								103.1	
Ti					96.0	91.4	99.0		
Tl							101.2		
U					96.9	94.4		99.0	
V							98.7		
W									
Y							98.5		
Zn	**3.8**	13.8	0.4	**2.8**	99.5	100.4	108.4		
Zr	**3.4**				98.9	98.1	103.6		

Shaded results do not meet QC acceptance criteria.

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

Bold and unshaded RPDs indicate one or both results <EQL.

n.m. = not measured; insufficient dilutions prepared to evaluate %Diff

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

	Run Date=	6/20/2001	6/20/2001	6/20/2001	6/20/2001	6/20/2001
	Multiplier=	1.0	21.3 (Na106.7)	20.3 (Na101.4)	21.1 (Na105.5)	21.2 (Na105.8)
	RPL#=	01-1012-B	01-1012	01-1012-D	01-1013	01-1013-D
Det. Limit	Client ID=		LS-10	LS-10	LS-11	LS-11
ug/mL	Analytes	ug/g	ug/g	ug/g	ug/g	ug/g
0.060	Al	[0.17]	6,580	6,540	6,590	6,600
0.010	Ba	[0.011]	--	--	[0.21]	--
0.250	Ca	--	118	117	120	120
0.015	Cd	[0.016]	21.2	21.1	21.2	21.2
0.020	Cr	--	85.1	85.1	88.1	88.7
0.025	Fe	[0.028]	[4.8]	[4.9]	[4.6]	[4.7]
2.000	K	--	795	789	784	780
0.050	La	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.150	Na	2.23	90,100	88,500	90,600	90,700
0.030	Ni	--	147	146	149	149
0.100	P	--	616	614	586	587
0.100	Pb	--	46.9	46.2	47.2	47.3
0.015	Sr	--	129	128	113	113
	Other Analytes					
0.025	Ag	--	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	1.68	54.8	52.6	56.5	52.9
0.010	Be	--	--	--	--	--
0.100	Bi	[0.15]	[3.5]	[2.3]	[2.3]	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	[1.5]	[1.6]	[1.6]	[1.6]
0.025	Cu	--	5.51	5.40	6.46	6.39
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.050	Mo	--	17.6	17.4	17.6	17.8
0.100	Nd	--	[3.9]	[4.1]	[3.8]	[3.9]
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[3.1]	208	189	202	185
1.500	Sn	--	--	--	--	--
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	[0.054]	[5.0]	[5.0]	[5.3]	[5.2]
0.050	Zr	--	[1.1]	[1.1]	[1.1]	[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).

QC Performance 6/20/2001

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-1013 & 01-1013 D	01-1012-BS	01-1012 MS	none + Post Spike A	none + Post Spike B	01-1013 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al	0.0	106.3	n.r.			2.8
Ba		110.1	86.7			
Ca	0.0	111.7	90.3			
Cd	0.1	111.4	87.8			
Cr	0.2	112.6	79.9			
Fe	7.3	115.6	90.5			
K	3.5	111.2	87.3			
La			90.1			
Mg		118.7	92.5			
Mn		116.1	93.0			
Na	0.2	101.7	n.r.			n.m.
Ni	0.1	117.7	78.1			
P	0.1	110.0	80.4			
Pb	2.8	116.0	89.6			
Sr	0.0	111.3	n.r.			3.7
Other Analytes						
Ag		107.1	88.4			
As		109.2				
B	5.9	n.r.				3.4
Be		108.5				
Bi		111.0	87.5			
Ce						
Co		114.5				
Cu	2.7	114.0	88.5			
Dy						
Eu						
Li		114.9				
Mo	0.7					4.7
Nd						
Pd			90.6			
Rh			92.2			
Ru			96.0			
Sb						
Se		109.4				
Si	6.3		97.7			
Sn						
Te						
Th						
Ti			85.0			
Tl		108.8				
U			89.3			
V		105.8				
W						
Y		106.4				
Zn	0.5	119.0	91.6			
Zr			91.6			

Shaded results exceed acceptance criteria

Bold results for information only; LCS or Serical Dilution concentration less than EQL.

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

n/a = not applicable; KOH flux and Ni crucible used for preparing samples.

Battelle PNWD
PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 2

Project / WP#:	42365 / W57984
ASR#:	6107
Client:	Rich Hallen
Total Samples:	4

RPL #	Client ID
01-1014	LS-12
01-1015	LS-13
01-1016	LS-14
01-1017	LS-16

Procedure: PNL-ALO-280 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Analyst: James P Bramson

Analysis Dates: 9/12/01, 9/25/01, 9/26/01, 9/28/01, 10/1/01

Analysis Files: Experiment - 12SEP01, 25SEP01, 25SEP01b, 26SEP01, 28SEP01, 01OCT01
Procedure - 010912a, 010925a, 010925b, 010926a, 010928a, 011001a
Element Menu - CsTcRb, UNp, Uiso Pu, multi, Tc

Revised Report Date: 3 February 2004

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

Revised By — Date: 3 Feb 04

Reviewed By — Date: 3 Feb 04

Revision	Effective Date	Description of Change
1	January 2004	Text has been added in the appropriate narrative section(s) to address issues surrounding the use of biased standards prepared and provided by the RPL Standards Laboratory to the ICP-MS analytical laboratory (reference: project #98620, memo KN Pool to GH Beeman, 9/15/03).
2	February 2004	Text has been added in the appropriate narrative section to include Pu-240 when addressing issues surrounding the use of biased standards (reference: project #98620, memo KN Pool to GH Beeman, 9/15/03).

1. Analysis

Four samples, 2 filtrates, 1 wash composite, and 1 solid prepared by the RPG, were submitted for analysis and analyzed on a radioactive material-contained ICP/MS. The filtrates and wash composite were analyzed for Tc-99. The solids were analyzed for total U, U isotopes, Pu-239 and 240, Np-237, and Ru, Rh, and Pd.

See attached ICP/MS data reports for final results and run order for the analytical batch. The final results have been corrected for all client dilutions and laboratory dilutions performed on the sample during analysis. A process blank, blank spike, duplicate, and matrix spike were also submitted and analyzed with the samples. In addition, replicate and post spike analyses were also performed.

The analysis for Ru, Rh, and Pd was difficult due to the isobaric interferences of SrO isotopes and masses common to both Ru and Pd, and the potential mixture of natural and fission yield isotopic abundances. Some of the sample concentrations were also very near detection limit.

2. Quality Control

Duplicate (DUP). The RPDs for all analyses (duplicate and replicate) were < 15%, with the exception of the solids duplicate analysis for Ru, Pd, and Tc-99. The problem with the Ru and Pd was most likely due to the difficulties mentioned above. The Tc-99 concentrations were very near the detection limit.

Matrix Spike (MS). The MS recoveries for all post spike analyses were within 75% to 125% with the exception of Ru. The analytical lab only had MS information for Ru, Rh, and Pd to calculate recovery, none of which met the QC criteria. The reason for the MS failure is suspected to be due to insufficient spiking level, that is, the MS concentration was less than the EQL (10XMDL).

Process Blank (PB) and Blank Spike (BS). The PBs were all below or near detection. The recovery of the blank spikes met the QC criteria for LCS recovery of 80% to 120%.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB and CCB standards are a 1% high purity nitric acid solution used as the diluent for the samples, except for the Pu analysis, where the eluent for the Pu separation was used. The ICB and CCB standards were at or below the instrument detection level except for the Pu analysis, where the standards were above detection level but 2 to 7 orders of magnitude below the sample concentrations.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). Recoveries of the ICV and CCV standards were within 90% to 110%.

Internal Standard (IS). The ISs were within the range of 30% to 120%.

Analytical results generated were influenced by an identified bias in the calibration and calibration verification standards. The effect on the results, based on the observed bias, will widen the error band for these results:

- The bias in the standards caused results for Tc-99 to be biased low for this report. The error band that should be applied to these results is -10% to +30%.
- The bias in the standards caused results for Np-237 to be biased high for this report. The error band that should be applied to these results is -20% to +10%.
- The bias in the standards caused results for Pu-239 to be biased low for this report. The error band that should be applied to these results is -10% to +20%.
- The bias in the Pu-239 standards, which were used to quantify Pu-240, caused results for Pu-240 to be similarly biased (low) for this report. The error band that should be applied to these results is approximately -10% to +20%.

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Analyst: 2/25/02

Reviewed by: 2/4/02

Client: R.Hallen
WP/Project: W57984 / 42365
ASR/Log-In: 6107, 01-01014 to 01-01016
Report Date: 2/9/02

Unless otherwise specified; the results are reported in µCi analyte/g of original sample.

RPL Log-In #	Sample ID	ICP/MS ID	MDL µCi/g	Tc 99 µCi/g	±	1SD	Rec/RPD %
	1%HNO_3	ICB		2.05E-09			
	1%HNO_3	CCB1		2.80E-09			
	1%HNO_3	CCB2		2.35E-09			
	1%HNO_3	CCB3		3.48E-09			
	1%HNO_3	CCB4		3.51E-09			
	True Value			1.70E-06			
	ICV	ICV		1.62E-06	±	3.43E-08	95%
	CCV1	CCV1		1.62E-06	±	4.61E-08	95%
	CCV2	CCV2		1.66E-06	±	4.23E-08	98%
	CCV3	CCV3		1.82E-06	±	1.56E-07	107%
	CCV4	CCV4		1.67E-06	±	3.74E-08	98%
01-01014-PB	PROCESS BLANK	Sample14	7.16E-07	3.19E-06	±	1.69E-07	
01-01014-PB	PROCESS BLANK post spike	Sample15	8.46E-07	4.70E-04	±	9.08E-06	
Post Spike Concentration expected				4.12E-04			113%
01-01014-BS-2	BLANK SPIKE	Sample16	1.66E-06	5.16E-04	±	9.12E-06	
01-01014	LS-12	Sample17	1.50E-05	6.29E-02	±	2.09E-03	
01-01014	LS-12 replicate	Sample18	1.52E-05	6.30E-02	±	4.24E-04	0.2%
01-01014-Dup	LS-12-Dup	Sample19	1.53E-05	6.26E-02	±	1.94E-04	0.3%
01-01015	LS-13	Sample20	1.54E-05	6.03E-02	±	9.02E-04	
01-01015	LS-13 post spike	Sample22	1.56E-05	6.59E-02	±	2.76E-04	
Post Spike Concentration				6.97E-03			80%
01-01015-MS-2	LS-13 ICP/MS MS	Sample23	1.55E-05	6.10E-02	±	7.72E-04	
01-01016	LS-14	Sample21	8.95E-06	1.20E-02	±	3.08E-04	

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/12/01
Instrument Filenames: Experiment (12SEP01), Procedure (010912a), Element Menu (CsTcRb)

Page 1 of 1

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

Client:	R. Hallen	**Charge Code/Project:**	W57984 / 42365
RPL Numbers:	01-01014 to 01-01017	**ASR Number:**	6107
Analyst:	MJ Steele	**Analysis Date:**	July 19/20/31, 2001

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)

Analysis Results

RPL #	Liquid Sample ID	TIC ugC/mL	TIC RPD	TOC ugC/mL	TOC RPD	TC ugC/mL	TC RPD
01-01014	LS-12	8,540		12,000		20,500	
01-01014 Dup	LS-12	8,550	0%	12,200	2%	20,700	1%
01-01015	LS-13	7,930		11,500		19,400	
01-01015 Dup	LS-13	7,680	3%	11,400	1%	19,100	2%
01-01015 Trip	LS-13	8,700		12,100		20,800	
01-01016	LS-14	1,500		3,140		4,640	
01-01016 Dup	LS-14	1,500	0%	3,030	3%	4,540	2%
01-01016 MS	Recovery	101%		99%		100%	
BS LCS (07-19-01)	Recovery	102%		106%			
BS LCS (07-20-01)	Recovery	103%		102%			
RPL #	**Solid Sample ID**	**TIC ugC/g**	**TIC RPD**	**TOC ugC/g**	**TOC RPD**	**TC ugC/g**	**TC RPD**
01-01017	LS-16	18,500		6,190		24,700	
01-01017 Dup	LS-16	17,700	4%	6,960	12%	24,700	0%
01-01017 MS	Recovery	104%		90%		97%	
BS LCS (07-31-01)	Recovery	101%		102%			

The TOC/TIC analyses of the samples submitted under ASRs 6107 are to be performed by both the hot persulfate and furnace methods. This report presents the results from 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.

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

Q.C. Comments:

The standards for TIC and TOC supernatant analysis are liquid carbon standards from VWR. The lot numbers and Chemical Management System (CMS) numbers for the standards are included on the raw data benchsheets.

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

For TIC and TOC solids analysis pure chemical compounds are used as the calibration, as well as matrix spiking, standards. The TIC analysis uses calcium carbonate and the TOC uses α-Glucose (JT Baker, Aldrich, Sigma, and Mallinckrodt lot numbers and CMS numbers are provided on the raw data benchsheets).

The QC for the methods involves calibration blanks, sample duplicates, laboratory control sample, and matrix spikes. The ASR indicates that the analyses are to be performed to "Conducting Analytical Work in Support of Regulatory Programs". The performance of the QC samples is compared to this QA Plan.

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyze each day that the samples were analyzed. The LCS/BSs for both the liquid analysis and the solids analysis were within acceptance criteria of 80% to 120%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spikes for the LS-14 liquid sample and the LS-16 solids sample demonstrate recoveries well within the acceptance criteria of 75% to 125% recovery.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD) between sample and duplicate. The TIC and TOC RPD results are well within the acceptance criteria of <20% RPD.

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 ±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. The analysis MDLs (total ug C) are based on 3 times the standard deviation of a set of historical data The sample MDLs (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- 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: ______________ **Date** 10-02-01

Review/Approval by: M J. Steele **Date** 10/22/01

Excel Archive File: ASR 6014L 6031L 6107L&S 6121L.xls

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

PO Box 999, Richland, WA 99352

Client	R. Hallen	**Charge Code/Project:**	W57984 / 42365
RPL Numbers:	01-1014 to 01-1017	**ASR Number:**	6107
Analyst:	MJ Steele	**Analysis Date:**	8/22, & 8/23 2001

Procedure: PNL-ALO-380, "Determination of Carbon in Solids Using the Coulometrics Carbon Dioxide Coulometer"

M&TE: Carbon System (WD13071); Balance (360-06-01-023).

TOC/TIC/TC Results

RPL Number	Sample ID	TIC (a) MDL ugC/mL	TIC (b) Results ugC/mL	TOC MDL ugC/mL	TOC Results ugC/mL	TC MDL ugC/mL	TC Results ugC/mL
01-01014	LS-12	220	5,700	180	15,500	220	21,200
01-01014 Dup	LS-12					220	21,200
	RPD						0%
01-01015	LS-13	220	nd	180	22,300	220	19,000
01-01016	LS-14	90	850	180	3,550	90	4,400
01-01016 Dup	LS-14	90	900	180	3,370	90	4,270
	RPD				5%		3%
01-01015 MS	LS-13						107%
01-01016 MS	LS-14				105%		
BS/LCS	Blank Spike/LCS				96%		94%
RPL Number	**Sample ID**	**ugC/g**	**ugC/g**	**ugC/g**	**ugC/g**	**ugC/g**	**ugC/g**
01-01017	LS-16 Washed Solids	190	nd	400	22,700	190	28,000
01-01017 Dup	LS-16 Washed Solids	129	5,400	240	23,100	130	28,500
	RPD				2%		2%
01-01017MS	LS-16 Washed Solids				100%		111%
BS/LCS	Blank Spike/LCS				99%		104%

nd = not detected (i.e., TC results > TOC results)

(a) TIC MDL set to TC MDL

(b) TIC is determined by difference (TC - TOC)

The TOC/TIC analyses of the samples submitted under ASRs 6107 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the furnace oxidation method and the results are compared to the results obtained from the hot persulfate method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample (solids or liquid) in oxygen at 750 °C for 30 minutes. The total carbon is determined on another aliquot of the sample by combusting at 1000 °C for 30 minutes. The total inorganic carbon is obtained by difference.

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

Q.C. Comments:

The calibration and QC standards for TC and TOC analysis are liquid or solid carbon standards or pure chemicals from JT Baker, Aldrich, Sigma, and Mallinckrodt. The identification of the standards and their Chemical Management System (CMS) numbers are included on the raw data benchsheets.

The coulometer analysis system calibration is checked by analyzing calibration standards at the beginning, middle, and end of each day's run. The average recovery from these calibration check standards is applied as a correction factor to the 'raw data' results obtained for the samples. The average recovery for the two analysis days was 100%, and 98%.

System blanks were analyzed similarly to the calibration check, averaged, and subtracted from the sample 'raw data' results prior to calculating the final reported result. The TOC determination produced an average blank of 15 µgC. The TC determination produced an average blank of 54 µgC. The 54 µgC blank level is unusually high; however, the reproducibility of the blank was reasonably good (i.e., 49 to 59 µgC).

For each days analysis run, the QC for the analyses include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes.

Blank Spike/Laboratory Control Sample: The BS/LCS was within acceptance criteria of 80% to 120% required by the governing QA Plan for both the TC and TOC analysis of the liquid and solids samples.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD), is within the acceptance criteria of the governing QA Plan (i.e., <20%).

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The TOC and TC matrix spike for both the liquids and solids samples demonstrates recoveries between 100% and 111%, which are within the acceptance criteria of 75% to 125%.

Furnace Results Compared to Hot Persulfate Results

RPL Number	**Sample ID**	**TIC HP Results ugC/mL**	**TIC Furn Results(a) ugC/mL**	**TOC HP Results ugC/mL**	**TOC Furn Results ugC/mL**	**TC HP Results(b) ugC/mL**	**TC Furn Results ugC/mL**
01-01014	LS-12	8,540	5,700	12,000	15,500	20,500	21,200
01-01015	LS-13	7,930	nd	11,500	22,300	19,400	19,000
01-01016	LS-14	1,500	850	3,140	3,550	4,640	4,400
RPL Number	**Sample ID**	**TIC HP Results ugC/g**	**TIC Furn Results(a) ugC/g**	**TOC HP Results ugC/g**	**TOC Furn Results ugC/g**	**TC HP Results(b) ugC/g**	**TC Furn Results ugC/g**
01-01017	LS-16 Washed Solids	18,500	130	6,190	22,700	24,700	28,000

nd = not detected (i.e., TOC result > TC result)

(a) TIC Furn is determined by difference (TC - TOC)

(b) TC HP is determined by sum (TIC + TOC)

The two method appear to produce comparable results for TC, with the furnace producing slightly higher results. However, there are significant differences between the TIC and TOC results reported by each method. The reason for the discrepancy between the hot persulfate method and furnace method is unknown, but it appears that the inorganic carbon, perhaps in the form of easily oxidized metal carbonates, is being combusted at 750 °C (as TOC) with the furnace method. Typically, the furnace method provides the best TC results and the hot persulfate the best TIC results, thus the TOC would be the difference between these measurements. Based on the furnace TC result for sample LS-16, the TOC result from the hot persulfate method may be about 30% low.

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 ±15% or better for non-complex samples that are free of interferences.
- For both the TC and TOC, the analysis Method Detection Limit (MDL) is based on the standard deviation calculated from the number (n) of system blanks analyzed with the batch of samples. The standard deviation is multiplied by the Student's *t* values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- 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 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.

Report Prepared by: ______________________ **Date** 2-19-01

Review/Approval by: ______________________ **Date** 1-3-02

Raw Data Calculation/Archive Information:
ASR 6019L&S 6025L 6031L 6107S.xls
ASR 6014 6104 5105 6106 6107 6155 6162 6192.xls

Battelle
. . . Putting Technology To Work

Date: 6/20/01

Subject: Hydroxide Analyses for: R. Hallen
ASR: 6107

To: R. Hallen

From: L. Greenwood

Samples of the filtrate composite and wash composite from the Sr/TRU Project AN-102/C-104 blend were analyzed for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed in duplicate using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. Three inflection points were observed for each sample generally corresponding to hydroxide, carbonate, and bicarbonate. The standard hydroxide recovery averaged 95% and a sample spike recovered at 96%. No hydroxide was detected in a reagent blank. The titration curves are included with the report.

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

ASR # 6107

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

Analyst: Lori P. Darnell 6-21-01

Reviewer: L R Greenwood 6-20-01

Summary Report

			Concentration, moles		
RPG #	**Client ID**		**First Point**	**Second Point**	**Third Point**
01-1014	**LS-12**		**0.21**	**1.29**	**0.93**
01-1014	**LS-12**	**Rep**	**0.19**	**1.28**	**0.96**
		RPD	**7%**	**1%**	**4%**
01-1015	**LS-13**		**0.21**	**1.26**	**0.87**
01-1015	**LS-13**	**Rep**	**0.18**	**1.28**	**0.89**
		RPD	**18%**	**1%**	**2%**
01-1016	**LS-14**		**0.057**	**0.24**	**0.18**
01-1016	**LS-14**	**Rep**	**0.053**	**0.22**	**0.12**
		RPD	**6%**	**6%**	**41%**
Standard 1			**95%**		
Standard 2			**95%**		
MS-1015	**Matrix spike**		**96%**		
Blank			**nd**		

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

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

Client:	R. Hallen	Charge Code/Project:	W57984/42365
ASR Number:	6107 Liquids	Sample Receipt Date:	05/11/2001
Sample Prep Date:	N/A	Sample Analysis Date:	06/13-14/2001
Analyst:	MJ Steele		

Preparation Procedure: N/A
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.\\

The final ion chromatography results ASR 6107 Liquid Samples (01-01014 through 01-01016) are presented in Table 1. Table 1 includes the samples, duplicates, processing blank (dilution liquid), low level standard, and laboratory control standard results.

Table 1: Anion Analysis Results – ASR 6107 Liquids

RPL Number	Sample ID	F µg/ml	Cl µg/ml	NO_2 µg/ml	Br µg/ml	NO_3 µg/ml	PO_4 µg/ml	SO_4 µg/ml	C_2O_4 µg/ml
	EQL	0.13	0.13	0.25	0.13	0.25	0.25	0.25	0.25
01-01014 Dilution Blank		< 0.13	< 0.13	< 0.25	< 0.13	< 0.25	< 0.25	< 0.25	< 0.25
	EQL	625	125	625	125	1250	250	250	250
01-01014	LS-12	4,300	1,960	43,600	< 125	106,000	3,260	6,710	1,530
01-01014 DUP	LS-12 Dup	4,500	1,920	43,900	< 125	107,000	3,280	6,710	1,530
	RPD	4%	2%	1%	n/a	1%	1%	0%	0%
01-01015	LS-13	4,400	1,880	41,200	< 125	99,500	3,620	6,430	1,460
01-01015 MS %Rec	LS-13 MS %Rec	114%	104%	106%	106%	110%	97%	100%	107%
	EQL	125	25	250	25	250	250	50	50
01-01016	LS-14	4,800	380	7,290	< 25	16,900	340	1,060	2,770
01-01016 MS %Rec	LS-14 MS %Rec	111%	104%	108%	107%	110%	100%	101%	109%
LLS %Rec		106%	111%	102%	101%	98%	98%	98%	108%
LCS %Rec		97%	100%	97%	98%	93%	91%	91%	100%

Fluoride exhibits significant interference from unknown anions making quantitation difficult. Fluoride results are maximum value.

EQL = Estimate quantitation limit; based on lowest calibration standard times all dilution factors used to calculate the reported results. No results below the EQL are reported.

The sample was prepared for ion chromatography anion analysis by dilution at 200-fold to 5000-fold in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample as dilutions less than 200-fold. The estimated quantitation limits which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in Table 1.

Q.C. Comments:

Duplicates: No duplicate was provided. However, one sample was split and analyzed in duplicate. The duplicate relative percent difference (RPD) meets the acceptance criteria of <20%.

Matrix Spike (HCV 010328): A matrix spike was prepared from two samples all anion recoveries were within the 75% to 125% recovery acceptance criteria, as shown in Table 1.

Laboratory Control Sample-LSC/BS (HCV 010328 @4x): A Blank Spike (i.e., the spike solution used to prepare the matrix spike samples) was prepared and measured at the same time as the Matrix Spike sample and demonstrated recoveries within the 90% to 110% acceptance criteria.

Low Level Standard (LLS/LCV 010328): As shown in Table 1, the LLS meets the acceptance criteria of 75% to 125% recovery.

System Blank/Processing Blanks: Ten system blanks were processed during the analysis of the liquid sample. No anions were detected in the system blanks above the estimate quantitation level.

Quality Control Calibration Verification Check Standards (ICV 010328): Ten mid-range verification standards were analyzed throughout the analysis runs. Except for four oxalate measurements, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The oxalate measurements that failed produced a recovery from 111% to 112%.

General Comments:

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

Report Prepared by: M W Urie Date 7-18-01

Review/Approval: MJ Steele Date 7-19-01

Archive Information:

Files: ASR 6107 Hallen.doc ASR 6104, 6106, 6107 REP.xls

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Project No. 42040

Internal Distribution
File/LB

Date August 13, 2001

To M. W. Urie

From L. R. Greenwood LRGreenwood

Reviewed
C Soderquist 8-13-01

Subject Radiochemical Analyses for AN-102/C-104 Blend – ASR 6107

Samples of the filtrates from tanks AN-102/C-104 blend were analyzed for gamma emitters, ^{90}Sr, alpha/AEA, U, and Am/Cm according to ASR 6107. The samples were acid digested or fused in the hot cells according to procedures PNL-ALO-128 or –115 and aliquots were delivered to the laboratory for analysis. The acid digestions were performed in four different batches in the hot cells, each batch having a separate process blank. The one solid sample, LS-16, was prepared in the hot cells by $KOH-KNO_3$ fusion. The attached reports list measured analyte activities in the original sample material in units of uCi/g. The reported errors ($1-\sigma$) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. Since no sample preparation was involved, no laboratory blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. In order to meet the requested detection limits, diluted aliquots of the hot cell preparations were counted for periods of 3 to 14 hours. All of the samples showed the presence of significant ^{137}Cs activity. Some of the samples also showed the presence of ^{60}Co, ^{154}Eu, ^{155}Eu, and ^{241}Am. The MRQ values for extended counting time GEA were met in all cases. Detection limits are listed in the tables. The hot cell process blanks showed the presence of ^{137}Cs. However, the activities in the blanks were negligible with respect to the samples. Sample duplicates showed good repeatability for sample LS-12. However, agreement for the hot cell preparation duplicates was marginal for sample LS-16 with RPD values of 8% to 15% for all of the isotopes except for ^{60}Co, where the RPD value was 37%, well outside of the expected range. The reason for the large disagreement for this one isotope is not known, but may suggest contamination or heterogeneity in the sampling in the hot cells. The activities measured for ^{241}Am are in good agreement with the Am/AEA results reported below for sample LS-17.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). Some of the separated Sr fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples in addition to the small beta correction from ^{85}Sr. In all cases, the ^{137}Cs correction was small compared to the activity in the samples. The samples were analyzed in two batches in the laboratory. The laboratory blank for the first batch showed significant ^{137}Cs contamination in the gamma count for ^{85}Sr. After correction for the ^{137}Cs beta contribution, the resultant ^{90}Sr value in the lab blank was about 15% of the activity in the samples; however, the uncertainty is very high due to the large correction. The lab blank for the second batch and the hot cell process blanks did not show any significant ^{90}Sr contamination. Sample duplicates showed good repeatability in all cases. The blank spike and matrix spike yields ranged from 97% to 104%. All of the samples showed the presence of ^{90}Sr at levels that were significantly less than the requested MRQ values.

Total Uranium

Total uranium was measured in samples LS-12 to LS-16 according to procedure PNNL-ALO-4014 using Kinetic Phosphoresence Analysis (KPA). Uranium was detected in both the hot cell preparation blanks; however, the levels were not significant relative to the samples. Sample duplicates and a lab replicate showed good repeatability. Since the analyses were performed on the samples as received from the hot cells, no sample spikes were required. LCS samples gave uranium results at 99% and 102% of the expected values. All of the measured uranium values were well below the requested MRQ values.

Total Alpha with Alpha Energy Analysis

The total alpha activity was determined by direct-plating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum ZnS scintillation detectors according to RPG-CMC-408. Alpha energy analyses were performed on all samples according to procedure RPG-CMC-422. Peaks were observed due to ^{234}U+^{237}Np, ^{239}Pu+^{240}Pu, ^{238}Pu+^{241}Am, ^{234}Cm+^{244}Cm, and ^{242}Cm. The sums of the individual alpha emitters are generally in reasonable agreement with the total alpha data indicating minimal losses due to alpha self-absorption.

Most of the hot cell preparation blanks and the laboratory blank did not show any significant alpha contamination. However, for the hot cell acid digestion batch containing samples LS-01, LS-12, LS-13, and LS-14, the process blank contained high alpha contamination. The blank activity is small compared to sample LS-01, but it is significant compared to the other samples. The alpha activity in the blank is 16% of that in sample LS-12, 23% of LS-13, and 115% of sample LS-14. These activities are well below the requested MRQ value of 0.23 uCi/ml, but the samples may need to be reprepared to meet the project reporting requirements. The total alpha and alpha/AEA data are also not in good agreement for this hot cell blank. On this blank, the alpha/AEA result is probably more reliable than the total alpha result. We re-prepped the alpha/AEA from a fresh aliquot, and its counting data agreed with the original alpha/AEA data.

Duplicate samples generally showed acceptable agreement taking into account the statistical uncertainties. The only exception is for $^{243/244}$Cm for sample LS-05 where the RPD value is 34%.

The reason for this difference is not known. The LCS and matrix spike recoveries were 102% and 105%, respectively.

Americium and Curium

The Am/Cm separations were performed for samples LS-12 to LS-16 according to PNL-ALO-417. The separated fractions were precipitation plated according to PNL-ALO-496 and the samples were counted by alpha spectrometry according to PNL-ALO-422. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. As discussed above for the total alpha, the hot cell process blank with samples LS-12 to LS-14 was contaminated with alpha activity. There was no significant contamination of the hot cell process blank for the solids sample LS-16. The Am/Cm AEA results are generally in good agreement with the total alpha AEA results indicating that there is little ^{238}Pu in these samples. The only exception is for the contaminated hot cell process blank (01-1014 PB), which clearly has a different isotopic mix than the samples. The LCS and matrix spike recoveries were 99% and 100%, respectively. RPD values were acceptable taking into account the statistical uncertainties. Most of the sample activities were well below the requested MRQ values.

^{99}Tc

The technetium in the solids sample LS-16 was chemically separated for analysis according to procedure PNL-ALO-432. The separated fractions were then counted according to procedure RPG-CMC-408. No activity was detected in either the hot cell process blank or the laboratory blank. The RPD value was 23% suggesting some heterogeneity in the sample duplicates. The matrix spike recovery was low at 79%, probably due to the high salt content from the fusion preparation. The LCS recovery was 95%. The ^{99}Tc activities were well below the requested MRQ values.

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

01-1003

06/22/01

Client : Urie

Cognizant Scientist: JR Greenwood Date : 6-22-01

Concur : T Trang-le Date : 6/22/01

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

ALO ID Client ID	Cr-51 Error %	Fe-59 Error %	Co-60 Error %	Nb-95 Error %	Ru-103 Error %	Ru-106 Error %	Sn-113 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Ce-144 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-1014PB Process Blank	<6.E-4	<9.E-5	<5.E-5	<5.E-5	<6.E-5	<5.E-4	<8.E-5	<2.E-4	<5.E-5	<6.E-5	6.99E-3 3%	<5.E-4	<2.E-4	<2.E-4	<3.E-4	<8.E-4
01-1014 LS-12	<9.E-2	<3.E-3	3.15E-2 2%	<2.E-3	<1.E-2	<7.E-2	<4.E-3	<4.E-2	<3.E-2	<2.E-3	1.27E+2 2%	<4.E-2	<1.E-3	1.90E-2 6%	<2.E-2	<2.E-2
01-1014 DUP LS-12 DUP	<9.E-2	<3.E-3	3.14E-2 2%	<2.E-3	<1.E-2	<7.E-2	<4.E-3	<4.E-2	<3.E-2	<2.E-3	1.27E+2 2%	<5.E-2	<1.E-3	1.84E-2 6%	<2.E-2	<2.E-2
RPD			0%								0%			3%		
01-1015 LS-13	<6.E-2	<2.E-3	3.22E-2 2%	<1.E-3	<8.E-3	<5.E-2	<3.E-3	<3.E-2	<2.E-2	<1.E-3	1.32E+2 2%	<3.E-2	<9.E-4	2.01E-2 4%	<1.E-2	<1.E-2
01-1016 LS-14	<2.E-2	<3.E-4	5.07E-3 2%	<2.E-4	<2.E-3	<1.E-2	<4.E-4	<7.E-3	<7.E-4	<2.E-4	2.42E+1 3%	<1.E-2	<3.E-4	2.25E-3 7%	<6.E-3	<2.E-2
01-1017PB Process Blank	<2.E-2	<2.E-3	<2.E-3	<1.E-3	<2.E-3	<1.E-2	<3.E-3	<5.E-3	<2.E-3	<2.E-3	8.64E-2 3%	<1.E-2	<7.E-3	<4.E-3	<8.E-3	<1.E-2
01-1017 LS-16	<4.E-1	<2.E-2	4.88E-2 7%	<1.E-2	<5.E-2	<3.E-1	<3.E-2	<2.E-1	<6.E-2	<2.E-2	1.37E+2 3%	<4.E-1	<5.E-2	4.46E+0 2%	2.52E+0 5%	3.82E+0 5%
01-1017 DUP LS-16 DUP	<4.E-1	<2.E-2	7.07E-2 5%	<1.E-2	<5.E-2	<3.E-1	<2.E-2	<2.E-1	<6.E-2	<1.E-2	1.48E+2 3%	<3.E-1	<4.E-2	3.84E+0 2%	2.23E+0 5%	3.42E+0 6%
RPD			37%								8%			15%	12%	11%

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

08/13/01

Client : R. Hallen

Cognizant Scientist: L. R. Greenwood Date : 8-13-01

Concur : C. Soderquist Date : 8-13-01

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

		Alpha Energy Analysis						Americium/Curium Analysis				
ALO ID Client ID	Total Alpha ± 1s	U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of individual alpha emitters	Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of Am + Cm	Uranium µg/g ± 1s
01-1003 LS-01	5.90E-2 ± 6%	3.23E-4 ± 16%	2.02E-3 ± 6%	5.02E-2 ± 2%	2.43E-3 ± 6%	2.16E-4 ± 19%	5.52E-2 ± 2%					
01-1003 DUP LS-01 DUP	5.73E-2 ± 6%	3.58E-4 ± 12%	1.93E-3 ± 5%	4.73E-2 ± 2%	2.36E-3 ± 5%	1.70E-4 ± 17%	5.21E-2 ± 2%					
RPD	3%	10%	5%	6%	3%	24%	6%					
01-1004 PB Process Blank	3.41E-4 ± 7%	7.82E-7 ± 28%	3.42E-5 ± 4%	2.05E-4 ± 2%	9.81E-5 ± 2%	<4.E-7	3.38E-4 ± 1%					
01-1004 LS-02	3.57E-2 ± 7%	2.17E-4 ± 16%	1.87E-3 ± 6%	3.38E-2 ± 2%	1.93E-3 ± 5%	9.69E-5 ± 25%	3.79E-2 ± 2%					
01-1004 DUP LS-02 DUP	4.03E-2 ± 7%	2.47E-4 ± 16%	1.56E-3 ± 6%	3.28E-2 ± 2%	1.69E-3 ± 6%	1.47E-4 ± 20%	3.64E-2 ± 2%					
RPD	12%	13%	18%	3%	13%	41%	4%					
01-1005 LS-03	1.16E-2 ± 13%	1.66E-4 ± 8%	7.57E-4 ± 4%	9.51E-3 ± 2%	8.04E-4 ± 4%	5.87E-5 ± 14%	1.13E-2 ± 2%					
01-1005 DUP LS-03 DUP	1.13E-2 ± 15%	1.75E-4 ± 8%	8.55E-4 ± 4%	9.80E-3 ± 2%	7.93E-4 ± 4%	7.11E-5 ± 13%	1.17E-2 ± 2%					
RPD	3%	5%	12%	3%	1%	19%	3%					
01-1006 LS-04	8.92E-3 ± 16%	1.78E-4 ± 8%	7.54E-4 ± 4%	8.10E-3 ± 2%	6.54E-4 ± 4%	4.45E-5 ± 16%	9.73E-3 ± 2%					
01-1006 DUP LS-04 DUP	1.44E-2 ± 12%	1.49E-4 ± 10%	6.40E-4 ± 5%	8.46E-3 ± 2%	6.97E-4 ± 5%	3.73E-5 ± 21%	9.98E-3 ± 2%					
RPD	47%	18%	16%	4%	6%	18%	3%					

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

		Alpha Energy Analysis						Americium/Curium Analysis				
ALO ID Client ID	Total Alpha ± 1s	U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of individual alpha emitters	Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of Am + Cm	Uranium µg/g ± 1s
01-1006 Lab DUP LS-04		2.15E-4 ± 9%	7.27E-4 ± 5%	8.99E-3 ± 2%	7.25E-4 ± 5%	6.83E-5 ± 16%	1.07E-2 ± 2%					
01-1007 LS-05	1.12E-2 ± 14%	1.62E-4 ± 8%	7.64E-4 ± 4%	9.06E-3 ± 2%	1.07E-3 ± 3%	5.22E-5 ± 15%	1.11E-2 ± 2%					
01-1007 DUP LS-05 DUP	9.83E-3 ± 16%	1.38E-4 ± 13%	6.28E-4 ± 6%	8.84E-3 ± 2%	7.57E-4 ± 5%	4.90E-5 ± 22%	1.04E-2 ± 2%					
RPD	13%	16%	20%	2%	34%	2%	6%					
01-1008 PB Process Blank	2.63E-4 ± 9%	<4.E-7	2.83E-5 ± 5%	1.68E-4 ± 2%	7.30E-5 ± 3%	<4.E-7	2.70E-4 ± 2%					
01-1008 LS-06	1.87E-2 ± 11%	1.93E-4 ± 11%	9.50E-4 ± 5%	1.30E-2 ± 2%	9.65E-4 ± 5%	7.27E-5 ± 19%	1.52E-2 ± 2%					
01-1008 DUP LS-06 DUP	1.24E-2 ± 12%	1.92E-4 ± 11%	8.66E-4 ± 5%	1.14E-2 ± 2%	8.26E-4 ± 5%	7.26E-5 ± 18%	1.34E-2 ± 2%					
RPD	41%	1%	9%	13%	16%	0%	13%					
01-1009 LS-07	1.43E-2 ± 14%	1.81E-4 ± 12%	1.03E-3 ± 5%	1.34E-2 ± 2%	1.18E-3 ± 5%	8.09E-5 ± 18%	1.59E-2 ± 2%					
01-1009 DUP LS-07 DUP	1.41E-2 ± 12%	1.70E-4 ± 12%	1.00E-3 ± 5%	1.25E-2 ± 2%	9.82E-4 ± 5%	4.54E-5 ± 24%	1.47E-2 ± 2%					
RPD	1%	6%	3%	7%	18%	56%	8%					
01-1010 LS-08	1.82E-2 ± 11%	2.41E-4 ± 11%	1.10E-3 ± 5%	1.32E-2 ± 2%	9.65E-4 ± 6%	1.09E-4 ± 17%	1.56E-2 ± 2%					
01-1010 DUP LS-08 DUP	1.29E-2 ± 13%	1.98E-4 ± 13%	9.70E-4 ± 6%	1.29E-2 ± 2%	1.00E-3 ± 6%	9.26E-5 ± 19%	1.52E-2 ± 2%					
RPD	34%	20%	13%	2%	4%	16%	3%					
01-1011 LS-09	1.35E-2 ± 13%	1.86E-4 ± 11%	9.00E-4 ± 5%	1.13E-2 ± 2%	8.53E-4 ± 5%	8.06E-5 ± 17%	1.33E-2 ± 2%					
01-1011 DUP LS-09 DUP	1.55E-2 ± 11%	1.83E-4 ± 8%	1.07E-3 ± 3%	1.17E-2 ± 2%	8.70E-4 ± 4%	5.32E-5 ± 15%	1.39E-2 ± 2%					
RPD	14%	2%	17%	3%	2%	41%	4%					

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

		Alpha Energy Analysis						Americium/Curium Analysis				
ALO ID Client ID	Total Alpha ± 1s	U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of individual alpha emitters	Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of Am + Cm	Uranium µg/g ± 1s
01-1012 PB Process Blank	2.25E-4 ± 9%	<5.E-7	2.61E-5 ± 5%	1.67E-4 ± 2%	6.17E-5 ± 3%	<5.E-7	2.56E-4 ± 2%					
01-1012 LS-10	1.94E-2 ± 10%	2.43E-4 ± 11%	1.05E-3 ± 5%	1.19E-2 ± 2%	1.02E-3 ± 5%	6.90E-5 ± 21%	1.43E-2 ± 2%					
01-1012 DUP LS-10 DUP	1.51E-2 ± 13%	2.02E-4 ± 11%	1.12E-3 ± 5%	1.31E-2 ± 2%	9.36E-4 ± 5%	7.84E-5 ± 18%	1.54E-2 ± 2%					
RPD	25%	18%	6%	10%	9%	13%	8%					
01-1012 Lab Dup LS-10 DUP		2.11E-4 ± 11%	1.07E-3 ± 5%	1.22E-2 ± 2%	9.69E-4 ± 5%	6.62E-5 ± 20%	1.45E-2 ± 2%					
01-1013 LS-11	1.44E-2 ± 12%	1.87E-4 ± 11%	1.01E-3 ± 5%	1.13E-2 ± 2%	8.70E-4 ± 5%	5.53E-5 ± 21%	1.34E-2 ± 2%					
01-1013 DUP LS-11 DUP	1.63E-2 ± 12%	1.89E-4 ± 10%	1.07E-3 ± 4%	1.17E-2 ± 2%	8.82E-4 ± 5%	7.23E-5 ± 17%	1.39E-2 ± 2%					
RPD	12%	1%	6%	3%	1%	27%	4%					
01-1014 PB Process Blank	1.78E-3 ± 3%	<1.E-6	3.41E-4 ± 2%	2.23E-3 ± 2%	2.01E-3 ± 2%	2.79E-6 ± 24%	4.58E-3 ± 1%	3.07E-4 ± 3%	7.72E-4 ± 2%	<8.E-7	1.08E-3 ± 2%	1.06E-1 2%
01-1014 LS-12	1.18E-2 ± 15%	1.71E-4 ± 11%	6.64E-4 ± 6%	8.68E-3 ± 2%	5.29E-4 ± 6%	3.54E-5 ± 25%	1.01E-2 ± 2%	8.49E-3 ± 2%	5.48E-4 ± 7%	5.06E-5 ± 21%	9.09E-3 ± 2%	2.20E+1 4%
01-1014 Dup LS-12 DUP	1.03E-2 ± 15%	1.79E-4 ± 11%	7.56E-4 ± 5%	8.54E-3 ± 2%	5.47E-4 ± 6%	3.17E-5 ± 27%	1.01E-2 ± 2%	8.39E-3 ± 2%	5.42E-4 ± 6%	6.23E-5 ± 18%	8.99E-3 ± 2%	2.17E+1 4%
RPD	14%	5%	13%	2%	3%	11%	0%	1%	1%	21%		1%
01-1014 Lab DUP LS-12								8.18E-3 ± 3%	5.32E-4 ± 8%	5.95E-5 ± 24%	8.77E-3 ± 3%	
01-1015 LS-13	7.90E-3 ± 17%	1.88E-4 ± 11%	7.32E-4 ± 5%	8.48E-3 ± 2%	6.10E-4 ± 6%	3.93E-5 ± 24%	1.00E-2 ± 2%	8.30E-3 ± 2%	5.57E-4 ± 6%	3.59E-5 ± 25%	8.89E-3 ± 2%	2.23E+1 4%
01-1016 LS-14	<4.E-3	3.69E-5 ± 24%	1.20E-4 ± 13%	1.32E-3 ± 4%	1.07E-4 ± 14%	<2.E-5	1.60E-3 ± 4%	1.17E-3 ± 3%	8.36E-5 ± 9%	6.69E-6 ± 33%	1.26E-3 ± 3%	3.15E+0 2%
01-1016 Rep LS-14												3.20E+0 2%
RPD												2%

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

		Alpha Energy Analysis						Americium/Curium Analysis				
ALO ID Client ID	Total Alpha ± 1s	U-234+ Np-237 ± 1s	Pu-239+ Pu-240 ± 1s	Pu-238+ Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of individual alpha emitters	Am-241 ± 1s	Cm-243+ Cm-244 ± 1s	Cm-242 ± 1s	Sum of Am + Cm	Uranium µg/g ± 1s
01-1017 PB Process Blank	6.31E-3 ± 9%	<2.E-5	9.48E-4 ± 5%	4.82E-3 ± 2%	1.75E-3 ± 3%	<2.E-5	7.56E-3 ± 2%	2.84E-3 ± 4%	1.84E-3 ± 5%	<3.E-5	4.71E-3 ± 3%	1.82E+0 2%
01-1017 LS-16	4.70E+0 ± 3%	<1.E-3	2.52E-1 ± 3%	4.37E+0 ± 2%	1.70E-1 ± 3%	1.58E-2 ± 10%	4.81E+0 ± 2%	3.99E+0 ± 2%	1.64E-1 ± 6%	1.09E-2 ± 21%	4.16E+0 ± 2%	3.25E+2 3%
01-1017 DUP LS-16 DUP	4.16E+0 ± 3%	8.33E-3 ± 28%	2.25E-1 ± 5%	3.70E+0 ± 2%	1.52E-1 ± 6%	1.31E-2 ± 22%	4.10E+0 ± 2%	3.45E+0 ± 3%	1.37E-1 ± 7%	1.08E-2 ± 24%	3.60E+0 ± 3%	2.97E+2 3%
RPD	12%		11%	17%	11%	19%	16%	15%	18%	1%		9%
MS-1003	105%											
MS-1006			95%									
MS-1012			104%									
MS-1014								100%				
BS-1003	102%		113% 116%					99%				102% 99%
BLK-1003	<4.E-3	<2.E-5 <2.E-5	<2.E-5 <2.E-5	<3.E-5 <2.E-5	<2.E-5 <9.E-6	<2.E-5 <9.E-6		<2.E-5	<2.E-5	<7.E-6		<2.E-5

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

8/1/01

Client : Urie

Cognizant Scientist: L.R. Greenwood   Date : 8/1/01

Concur : C Soderquist   Date : 8-13-01

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

ALO ID Client ID	Sr-90 Error +/-	Tc-99 Error +/-
01-1003 LS-01	2.01E+1 3%	
01-1003 DUP LS-01 DUP	2.03E+1 3%	
RPD	1%	
01-1004 PB Process Blank	5.58E-2 3%	
01-1004 LS-02	3.07E+0 4%	
01-1004 DUP LS-02 DUP	3.61E+0 4%	
RPD	16%	
01-1005 LS-03	1.88E+0 5%	
01-1005 DUP LS-03 DUP	1.93E+0 5%	
RPD	3%	
01-1006 LS-04	1.73E+0 5%	
01-1006 DUP LS-04 DUP	1.82E+0 5%	
RPD	5%	

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

ALO ID Client ID	Sr-90 Error +/-	Tc-99 Error +/-
01-1006 Lab DUP	1.65E+0	
LS-04 DUP	4%	
01-1007	1.75E+0	
LS-05	5%	
01-1007 DUP	1.79E+0	
LS-05 DUP	5%	
RPD	2%	
01-1008 PB	1.88E-2	
Process Blank	3%	
01-1008	1.68E+0	
LS-06	5%	
01-1008 DUP	1.56E+0	
LS-06 DUP	6%	
RPD	7%	
01-1009	3.33E+0	
LS-07	4%	
01-1009 DUP	3.14E+0	
LS-07 DUP	4%	
RPD	6%	
01-1010	2.92E+0	
LS-08	4%	
01-1010 DUP	3.06E+0	
LS-08 DUP	4%	
RPD	5%	
01-1011	2.67E+0	
LS-09	3%	
01-1011 DUP	2.71E+0	
LS-09 DUP	3%	
RPD	1%	

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

ALO ID Client ID	Sr-90 Error +/-	Tc-99 Error +/-
01-1012 PB	6.26E-2	
Process Blank	3%	
01-1012	2.18E+0	
LS-10	3%	
01-1012 DUP	2.55E+0	
LS-10 DUP	3%	
RPD	16%	
01-1012 Lab Dup	2.19E+0	
LS-10 DUP	3%	
01-1013	1.99E+0	
LS-11	3%	
01-1013 DUP	2.04E+0	
LS-11 DUP	3%	
RPD	2%	
01-1014 PB	6.52E-2	
Process Blank	3%	
01-1014	1.28E+0	
LS-12	3%	
01-1014 Dup	1.40E+0	
LS-12 DUP	3%	
RPD	9%	
01-1015	1.40E+0	
LS-13	3%	
01-1016	3.26E-1	
LS-14	5%	
01-1017 PB	1.44E-1	<4.E-4
Process Blank	4%	

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

ALO ID Client ID	Sr-90 Error +/-	Tc-99 Error +/-
01-1017	1.75E+3	1.03E-1
LS-16	3%	4%
01-1017 DUP	1.40E+3	8.17E-2
LS-16 DUP	3%	4%
RPD	22%	23%
MS-1203		79%
MS-1006	104%	
MS-1012	97%	
BS-1003	101%	95%
BS-1010	103%	
Blk-1017		<6.E-7
Blk-1006	2.38E-1	
	33%	
Blk-1010	<9.E-2	

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

PO Box 999, Richland, Washington 99352

Project / WP#:	42365 / W58166
ASR#:	6130
Client:	S. Fiskum
Total Samples:	2

RPL#:	01-01345	01-01354
Client ID:	"ANC102/104-C-F/A"	"AN102CST-C-F/A"
Sample Preparation: PNL-ALO-106 (0.2mL/20mL)		

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

Analyst: D.R. Sanders

Analysis Date (File): 07-18-2001 (A0700)

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

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

9-19-01
Reviewed by

Jerry Wagner 9-18-01
Concur

Two liquid samples (ANC 102/104-C F/A and AN102CST-C F/A) from Analytical Service Request 6130 were prepared by acid digestion per PNL-ALO-106. The samples were digesting in the laboratory (i.e., not in the Shielded Analytical Laboratory) by using 0.2 mL of sample and diluting to a final volume of 20 mL.

In the Analytical Service Request (ASR), Na and K were identified as analytes of interest for this work along with 'minors as found'. Therefore, any analyte detected in the samples besides Na and K was considered an analyte of interest; i.e., Al, B, Ba, Ca, Cd, Cr, Cu, Fe, Mo, Ni, P, Pb, Si, Sr, and Zn. The quality control (QC) results for each of these analytes has been evaluated and is presented below. Analytes other than those detected part of the ICPAES analysis are reported, but have concentrations less than the method detection limit (MDL) and have not been fully evaluated for QC performance.

The attached ICPAES Results (2 pages) presents the final results. Results are from the direct measurement of the digestates, except for the AN102CST-C F/A duplicate which was measured following an additional 2x dilution at the ICPAES. The ICPAES measurement results are reported in µg/mL of liquid sample and have been corrected for all dilutions resulting from sample processing.

The following is a list of quality control measurement results relative to ICPAES analysis requirements of the controlling QA plan A digestion processing blank, laboratory control sample (blank spike), and duplicate were prepared with the sample. Through a mix up in the processing laboratory, no matrix spike was prepared from either of the samples analyzed by ICPAES; therefore, post spike were used to assess matrix interferences. The blank spike was prepared by using 1 ml of a custom multi-element solution "INT-QC-MCVA-1B" per 20 mL digestate volume.

Process Blank:

Concentration of analytes of interest measured in the process blank were all within tolerance limit of ≤ EQL or less than ≤5% of the concentration in the sample, except for B, Ba, Fe, and Si. The sample concentration for B, Ba, Fe, and Si are essentially the same as the processing blank concentrations, suggesting that there is little, if any, B, Ba, Fe, or Si in the samples.

Blank Spike (laboratory control sample):

The blank spike recovery for analytes of interest was within the acceptance criteria of 80% to 120%, except for B, Mo, and Si. For B the high recovery (i.e., 210%) is attributed to digesting the BS in glass vials. Silicon and Mo were not included in the BS since the only analytes identified as required were Na and K.

Duplicate RPD (Relative Percent Difference):

For those analytes of interest measured above the estimated Method Detection Limit (MDL), the RPDs were within the acceptance criteria of less than 20%.

Matrix Spiked Sample:

No matrix spike was analyzed with this batch of samples.

Post-Spiked Samples (Group A; all analytes of interest):

All post-spiked analytes of interest in samples tested were recovered within tolerance of 75% to 125%, except Al and Na. The post spike analysis uses a general spiking solution intended to be usable on the majority of sample analyzed by ICPAES. However, for the sample selected for post spiking, the spike concentration for Al and Na was less than 20% of the sample concentration and the recovery results are considered meaningless. For these analytes, the use of serial dilution results is required to evaluate potential matrix interferences.

Post-Spiked Samples (Group B; other analytes):

The post spiked analytes (i.e., analytes other than those identified as analytes of interest) were within tolerance of 75% to 125%.

Serial dilution:

Serial dilution was required for Al and Na, since both the post spike concentrations were less than 20% of the sample concentration (i.e., recoveries could not be evaluated). These analytes demonstrated a percent difference (%D) within the acceptance criteria of ±10% after correcting for dilution.

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

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

	Multiplier=	100	100	200	200
	RPL#=	01-1338 PB	01-1345	01-1354	01-1354 D
Det. Limit	Client ID=	Process Blank	ANC 102 /104-C-F/A	AN102 CST-C-F/A	AN102 CST-C-F/A
ug/mL	Analytes	ug/mL	ug/mL	ug/mL	ug/mL
0.150	Na	1,010	111,000	136,000	146,000
2.000	K	--	[950]	[1,100]	[1,100]
	Other Analyte Detected				
0.060	Al	84.1	8,210	6,520	6,730
0.050	B	750	624	815	755
0.010	Ba	[1.3]	[1.6]	[2.5]	--
0.250	Ca	--	[150]	[190]	[190]
0.015	Cd	--	26.0	32.1	32.3
0.020	Cr	--	108	119	121
0.025	Cu	--	[10]	[8.4]	[7.5]
0.025	Fe	[5.7]	[9.4]	[9.1]	[8.8]
0.050	Mo	--	[22]	[26]	[27]
0.030	Ni	--	188	237	240
0.100	P	--	748	457	455
0.100	Pb	--	[67]	[88]	[84]
0.500	Si	973	1,110	1,250	1,240
0.015	Sr	--	86.5	60.3	62.1
0.050	Zn	--	[8.6]	--	--
	Other Analytes Measured but Not Detected				
0.025	Ag	--	--	--	--
0.250	As	--	--	--	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.050	La	--	--	--	--
0.030	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.100	Nd	--	--	--	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
1.500	Sn	--	--	--	--
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	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/RSE/Inorganic Analysis ... ICPAES Results

QC Performance

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-1354 & 01-1354 D	01-1388-AES-LCS-BS	none	01-1345 + Post Spike A	01-1345 + Post Spike B	01-1354 @2/@3 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Na	7.1	93.5		n.r.		0.7
K	5.2	103.4		93.6		
Other Analyte Detected						
Al	3.2	97.6		n.r.		-0.2
B	7.6	209.8		102.0		
Ba		110.3		95.5		
Ca	1.0	105.6		97.5		
Cd	0.5	106.7		98.1		
Cr	2.2	105.9		98.4		
Cu	11.2	107.9		99.2		
Fe	2.3	109.0		100.5		
Mo	1.3			97.0		
Ni	1.3	109.0		100.5		
P	0.5	105.3		93.8		
Pb	4.6	109.7		100.3		
Si	0.8			106.7		
Sr	2.9	105.4		99.1		
Zn		114.2		101.9		
Other Analytes Measured but Not Detected						
Ag		101.2		93.2		
As		103.6		98.7		
Be		102.2		95.1		
Bi		104.1		95.9		
Ce					94.0	
Co		108.9		101.7		
Dy					94.0	
Eu					101.1	
La					91.9	
Li		106.6		97.7		
Mg		112.3		103.6		
Mn		109.8		102.1		
Nd					92.2	
Pd					85.4	
Rh					94.3	
Ru						
Sb				96.0		
Se		105.0		98.4		
Sn						
Te						
Th					95.1	
Ti				94.2		
Tl		101.9		93.7		
U					90.3	
V		99.8		92.7		
W						
Y		99.4		93.2		
Zr				99.0		

n.r. = not recovered; spike at <20% of sample concentration

Battelle, PNNL / AIAL
Inorganic Analysis / ICP-MS Data Report

Project / WP#:	42365 / W58168
ASR#:	6130
Client:	Sandy Fiskum
Total Samples:	8

RPL #	Client ID
01-01338	AP101-S3C-F/A
01-01339	AP101-S2a
01-01340	AP101-S1a
01-01345	ANC 102/104-C-F/A
01-01346	ANC 102/104-S1C-F/A
01-01354	AN102 CST-C-F/A
01-01355	AN102 CST-S3C
01-01356	AN102 CST-S4C

Procedure: PNL-ALO-280 Rev. 1, *Inductively-Coupled Plasma Mass Spectrometric (ICP-MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Analyst: James P Bramson

Analysis Date: 9/12/01, 9/17/01, 9/18/01

Analysis Files: Experiment – 12SEP01b, 17SEP01b, 18SEP01b
Procedure - 010912b, 010917b, 010918b
Element Menu - CsTcRb, Multi

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

11-26-01
Prepared By

Eight samples, a process blank, and a blank spike submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte, Cs^{133}.

1. Analysis

See attached ICP/MS data reports for final results and run order for the analytical batch. The final results have been corrected for all client and laboratory dilutions performed on the sample during analysis.

2. Quality Control

Duplicate (DUP). In addition to the duplicate sample submitted (AN102 CST-C-F/A), a replicate analysis on sample AP101-S3C-F/A, was also performed. The RPD for both duplicate and replicate analyses met the QC criteria of < 20%.

Matrix Spike (MS). In addition to the matrix spike samples submitted, a post spike was also performed on AN1102 CST-C-F/A. The spike recovery for both the ANC 102/104-S1C-F/A matrix spike and AN1102 CST-C-F/A post spike met the QC criteria of 75% – 125%. However, the AN102 CST-C-F/A matrix spike recovery was above this range (129%).

Process Blank (PB), Blank Spike (BS). The PB concentration was near detection limit and below the MRQ. A post spike of the PB met the spike recovery QC criteria of 75% – 125%. However, the BS spike recovery (132%) was above the QC criteria of 80% - 120%.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than the estimated quantitation limit (EQL, taken to be the lowest calibration standard), was met.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). The ICV/CCV standards met the QC criteria of 90-110%.

Batteile, PNNL, AIAL
ICP/MS Analysis Data Report

Client: Sandy Fiskum
WP/Project: W58166 / 42365
ASR/Log-In: 6130, 01-01338 to 01-01356
Report Date: 11/25/01

Reviewed by: 11-26-01

Analyst:
11/27/01

Unless otherwise specified, the results are reported in µg analyte/ml of original sample.

Log-In	Sample	ICP/MS	MDL		Cs-133			Rec/RPD
Number	ID	ID	µg/ml		µg/ml	±	1SD	%
	1%HNO$_3$	ICB		<	1.85E-06			
	1%HNO$_3$	CCB1			2.44E-06	±	2.88E-07	
	1%HNO$_3$	CCB2			2.61E-06	±	3.29E-07	
	1%HNO$_3$	CCB3		<	1.85E-06			
	1%HNO$_3$	CCB4			2.40E-06	±	3.73E-07	
	1%HNO$_3$	CCB5			2.32E-06	±	7.13E-07	
	1%HNO$_3$	ICB		<	1.54E-06			
	1%HNO$_3$	CCB3		<	2.13E-06			
	1%HNO$_3$	CCB4		<	2.17E-06			
	1%HNO$_3$	ICB		<	8.00E-06			
	1%HNO$_3$	CCB1		<	8.42E-06			
	1%HNO$_3$	CCB2		<	1.09E-05			
	0.1ppb Cs	ICV			9.85E-05	±	1.25E-06	99%
	0.1ppb Cs	CCV1			1.03E-04	±	1.10E-06	103%
	0.1ppb Cs	CCV2			1.04E-04	±	1.90E-06	104%
	0.1ppb Cs	CCV3			1.02E-04	±	1.67E-06	102%
	0.1ppb Cs	CCV4			1.01E-04	±	3.67E-07	101%
	0.1ppb Cs	CCV5			1.03E-04	±	9.08E-07	103%
	1ppb Cs	ICV			9.75E-04	±	3.40E-06	98%
	1ppb Cs	CCV3			9.42E-04	±	4.75E-06	94%
	1ppb Cs	CCV4			9.73E-04	±	5.32E-06	97%
	1ppb Cs	ICV			1.03E-03	±	3.59E-06	103%
	1ppb Cs	CCV1			1.03E-03	±	1.99E-05	103%
	1ppb Cs	CCV2			1.04E-03	±	1.17E-05	104%
01-01338 PB	PROCESS BLANK	Sample1	1.87E-03		7.55E-03	±	4.93E-04	
01-01338 PB	PROCESS BLANK post spike	Sample4	1.91E-03		1.03E-01	±	5.49E-04	
Post Spike Concentration expected					1.00E-01			96%
01-01338 MS-LCS/BS	BLANK SPIKE	Sample10	1.94E-01		6.60E+01	±	9.07E-01	
Matrix Spike Concentration expected					5.00E+01			132%
01-01338	AP101-S3C-F/A	Sample21	4.41E-01		1.20E+02	±	1.73E+00	
01-01338	AP101-S3C-F/A replicate	Sample22	4.41E-01		1.27E+02	±	9.36E-01	6.0%
01-01339	AP101-S2a	Sample26	1.86E-01		4.54E+01	±	4.84E-01	
01-01340	AP101-S1a	Sample27	1.82E-01		9.33E+00	±	1.59E-01	
01-01345	ANC 102/104-C-F/A	Sample28	1.86E-01		5.10E+00	±	1.30E-01	
01-01346	ANC 102/104-S1C-F/A	Sample5	2.11E+00		1.30E+02	±	1.69E+00	
01-01346 MS-MS	ANC 102/104-S1C-F/A	Sample25	4.31E-01		1.69E+02	±	2.13E+00	
Matrix Spike Concentration expected					5.00E+01			79%
01-01354	AN102 CST-C-F/A	Sample30	1.86E-01		9.77E+00	±	8.75E-01	
01-01354 DUP	AN102 CST-C-F/A	Sample31	1.91E-01		8.97E+00	±	5.61E-02	8.5%
01-01354	AN102 CST-C-F/A post spike	Sample34	1.88E-01		1.92E+01	±	5.41E-01	
Post Spike Concentration expected					1.00E+01			95%
01-01354 MS	AN102 CST-C-F/A	Sample36	1.84E-01		7.45E+01	±	3.59E-01	
Matrix Spike Concentration expected					5.00E+01			129%
01-01355	AN102 CST-S3C-F/A	Sample24	4.28E-01		1.19E+02	±	1.27E+00	
01-01356	AN102 CST-S4C-F/A	Sample33	1.85E+00		8.95E+02	±	6.16E+00	

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1

File: 01-1336

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

8/10/01

Client : S. Fiskum

Cognizant Scientist: LRGreenwood Date : 8/13/01

Concur : T Trang-le Date : 8|10|01

PNL-ALO-476 (Sr-90)

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

ALO ID Client ID	Sr-90 Error %
01-1348 PB	<7.E-3
Process Blank	
01-1348	9.14E+0
AN-102-CST-F/A	3%
01-1349	8.22E+0
AN-102-CSTD-F/A	3%
RPD	11%
01-1350	1.07E+1
AN-102-S3-CST-F/A	7%
01-1351	1.02E+1
AN-102-S3-CSTD-F/A	7%
RPD	5%
01-1352	1.05E+1
AN-102-S4-CST-F/A	7%
01-1353	1.03E+1
AN-102-S4-CSTD-F/A	8%
RPD	2%
01-1354	1.34E+1
AN-102CST-C-F/A	7%
01-1354 DUP	1.45E+1
AN-102CST-C-F/A	7%
RPD	8%
Blank Spike	108%
Matrix Spike	126%
Blank	1.03E-2
	36%

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

7/25/01

Client : S. Fiskum

Cognizant Scientist: LRGreenwood Date : 7/25/01

Concur : T Trang-le Date : 7/25/01

PNL-ALO-450 (GEA)

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

ALO ID	Co-60	Cs-134	Cs-137	Eu-154	Eu-155	Am-241
Client ID	Error %	Error %	Error %	Error %	Error %	Error %
01-1336	2.23E-3	4.45E-3	2.18E+1	<2.E-3	<2.E-2	<2.E-2
AP101-S3-644- F/A	12%	10%	2%			
01-1337	2.44E-3	5.46E-3	2.34E+1	<2.E-3	<3.E-2	<3.E-2
AP101-S3-644D-F/A	11%	11%	2%			
01-1338	2.39E-3	3.26E-2	1.38E+2	<4.E-3	<4.E-2	<4.E-2
AP101-S3-C-F/A	14%	5%	2%			
01-1341	3.57E-2	<4.E-4	5.16E+0	2.32E-2	1.31E-2	9.76E-3
ANC102/104-644- F/A	2%		2%	2%	7%	13%
01-1342	3.51E-2	<5.E-4	1.50E+1	2.29E-2	1.27E-2	6.80E-3
ANC102/104-S1-644- F/A	2%		2%	2%	9%	26%
01-1343	3.65E-2	<7.E-4	1.45E+1	2.26E-2	1.24E-2	1.17E-2
ANC102/104 S1-644D- F/A	2%		2%	3%	14%	23%
01-1344	3.50E-2	<2.E-3	5.14E+1	2.36E-2	<3.E-2	<3.E-2
ANC102/104- S2-644- F/A	3%		2%	6%		
01-1345	4.09E-2	<5.E-3	1.61E+2	<9.E-3	<6.E-2	<6.E-2
ANC102/104-C- F/A	3%		2%			
01-1346	4.05E-2	<5.E-3	1.62E+2	<1.E-2	<7.E-2	<7.E-2
ANC102/104- S1-C- F/A	3%		2%			
01-1347	4.04E-2	<4.E-3	1.60E+2	<8.E-3	<6.E-2	<6.E-2
ANC102/104-S2C-F/A	3%		2%			
01-1348	5.28E-2	<3.E-3	1.84E+1	8.19E-2	4.77E-2	5.31E-2
AN-102-CST-F/A	3%		2%	3%	12%	17%
01-1350	5.18E-2	<3.E-3	1.76E+1	7.84E-2	3.84E-2	2.77E-2
AN-102-S3-CST- F/A	3%		2%	3%	14%	28%
01-1352	5.28E-2	<2.E-3	6.59E+1	7.85E-2	3.51E-2	3.87E-2
AN-102-S4-CST-F/A	2%		2%	3%	17%	26%

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

ALO ID	Co-60	Cs-134	Cs-137	Eu-154	Eu-155	Am-241
Client ID	Error %	Error %	Error %	Error %	Error %	Error %
01-1354	5.46E-2	<1.E-2	2.05E+2	<4.E-2	<3.E-1	<3.E-1
AN-102CST-C- F/A	6%		2%			
01-1355	5.52E-2	<2.E-2	2.10E+2	<6.E-2	<4.E-1	<4.E-1
AN-102CST-S3-C- F/A	8%		2%			
01-1356	5.46E-2	<8.E-3	2.05E+2	<3.E-2	<2.E-1	<2.E-1
AN102CST-S4-C- F/A	5%		2%			

AN-102 / C-104 Column Run Analytical Results

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

Project / WP#:	42365/ W58166
ASR#:	6174
Client:	S. Fiskum
Total Samples:	1

RPL#:	**01-01732**	—
Client ID:	**AN102/C104-RGN**	—
Sample Preparation: Sample prepared by PNL-ALO-106 (0.5mL/10mL). **AOI**: Na.		

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

Analyst: **D.R. Sanders**

Analysis Date (File): **10-22-01 (A0731)**

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

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

3-14-02
Reviewed by

Renée Russell 3-14-02
Concur

One liquid sample (RPL# 01-01732) submitted under Analytical Service Request (ASR) 6174 was prepared by acid digestion per PNL-ALO-106. The sample was digested by using 0.5 mL of sample and diluting to a final volume of 10 mL. Analytes of interest (AOI) were specified on the ASR as Na. All other analytes that were not requested are reported, but have not been fully evaluated for QC performance.

A summary of the ICPAES analyses of the sample, including QC performance, is given in the attached ICPAES Data Report (2 pages). ICPAES measurement results are reported in µg/mL and have been corrected for dilution resulting from sample processing.

The process blank had detectable amounts of Ag, Al, B, Ba, Dy, and Na present. The Ag, Al, B, Ba, and Dy were below estimated quantitation limits (EQL) and were not AOIs. The Na present in the process blank was at a concentration greater than the estimated EQL and greater than 5% of the sample concentration. The Na concentration in the process blank is approximately 30% of that found in the AN102/C104-RGN sample. This is outside of the tolerance limit, and most likely results from processing the samples by method PNL-ALO-106 as per the ASR. This method uses glass digestion vessels and high Na and B values are not uncommon.

Quality control standard results met tolerance requirements for the specific AOI's except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan. Blank spikes, Matrix-spikes and duplicates were prepared with the sample and analyzed. Blank-spike and matrix-spike samples were prepared using 1.0 mL of multi-element solution "INT-QC-MCVA-1B" per 10 mL of digestate volume.

Process Blanks:

The concentration of sodium (the AOI) measured in the process blank was outside the tolerance limit of ≤ EQL or less than ≤5% of the sample. One Na blank concentration is approximately 30% of the sample concentration. The other analytes detected in the process blank meet the <EQL acceptance criteria.

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample (RPL# 01-01732) were outside the tolerance limit of ≤ 3.5% RPD for Na. This indicates poor precision. The process blank had very high and variable levels of Na, which leads to poor precision.

Blank Spike:

Blank-Spike recoveries for the analyte of interest were within tolerance of 80% to 120%. However, the blank spike recovery for B was outside the tolerance limit at 63% and 51%; most likely due to high B in the sample blanks.

Matrix Spiked Sample:

Matrix-Spike recovery for the analyte of interest was within tolerance of 75% to 125%. However, the matrix spike recovery for B was outside the tolerance limit at 61%; most likely due to high and variable B in the sample blanks.

Post-Spiked Samples (Group A):

No post-spike A was analyzed with this batch of samples.

Post-Spiked Samples (Group B):

No post-spike A was analyzed with this batch of samples.

Five fold serial dilution:

All analytes above EQL in the sample tested were within tolerance limit of 10% after correcting for dilution.

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%.

Comments:

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

	Run Date=	10/22/2002	10/22/2002	10/22/2002
	Multiplier=	20.0	20.0	20.0
	RPL/LAB #=	01-01732-BLK	01-01732	01-01732-DUP
Det. Limit	*Client ID=*	*process blank*	*AN102/C104-RGN*	*AN102/C104-RGN-DUP*
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)
0.150	Na	99.1	275	326
Other Analytes				
0.025	Ag	[0.60]	--	--
0.060	Al	[10]	[6.5]	[6.6]
0.250	As	--	--	--
0.050	B	[8.3]	[3.6]	[4.5]
0.010	Ba	[0.21]	--	--
0.010	Be	--	--	--
0.100	Bi	--	--	--
0.250	Ca	--	--	--
0.015	Cd	--	--	4.20
0.200	Ce	--	--	--
0.050	Co	--	--	--
0.020	Cr	--	--	--
0.025	Cu	--	--	--
0.050	Dy	[6.8]	16.0	--
0.100	Eu	--	--	--
0.025	Fe	--	--	[0.71]
2.000	K	--	--	--
0.050	La	--	--	--
0.030	Li	--	--	--
0.100	Mg	--	--	--
0.050	Mn	--	--	--
0.050	Mo	--	--	--
0.100	Nd	--	--	--
0.030	Ni	--	--	--
0.100	P	--	--	--
0.100	Pb	--	--	--
0.750	Pd	--	--	--
0.300	Rh	--	--	--
1.100	Ru	--	--	--
0.500	Sb	--	--	--
0.250	Se	--	--	--
0.500	Si	--	--	--
1.500	Sn	--	--	--
0.015	Sr	--	[2.4]	--
1.500	Te	--	--	--
1.000	Th	--	--	--
0.025	Ti	--	--	--
0.500	Tl	--	--	--
2.000	U	--	--	--
0.050	V	--	[2.4]	--
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).

QC Performance 10/22/2001

Criteria>	<20% [a]	80% - 120%		75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	01-01732 & 01-01732-D	LCS/BS		01-01732 & 01-01732-MS	Post Spike A (none)	Post Spike B (none)	01-01732 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec		%Rec	%Rec	%Rec	%Diff
Na	16.9	101	100	109			2.6
Other Analytes							
Ag		92	93	100			
Al		100	99	103			
As		102	101	103			
B		63	51	61			
Ba		98	96	100			
Be		100	99	100			
Bi		101	100	102			
Ca		102	99	103			
Cd		103	102	104			
Ce							
Co		104	102	104			
Cr		102	100	102			
Cu		101	100	103			
Dy							
Eu							
Fe		102	102	103			
K		105	106	111			
La							
Li		103	103	108			
Mg		105	102	106			
Mn		104	103	105			
Mo							
Nd							
Ni		106	103	103			
P		102	101	103			
Pb		103	102	104			
Pd							
Rh							
Ru							
Sb							
Se		102	102	104			
Si							
Sn							
Sr		98	86	76			
Te							
Th							
Ti							
Tl		99	96	99			
U							
V		97	96	86			
W							
Y		97	96	98			
Zn		104	104	104			
Zr							

Shaded results exceed acceptance criteria

Bold results for information only; LCS, MS, or Serial Dilution concentration less than EQL

(a) Na RPD Criteria ≤ 3.5%.

Battelle

. . . *Putting Technology To Work*

Date: 9/21/01

Subject: Hydroxide Analyses for: AN-102/C104 - RGN (0.025M NaOH)
ASR: 6174 RPL # 01-1732

To: Sandy Fiskum

From: Bob Swoboda 9/21/01

A 0.025M NaOH fraction of **AN-102/C104-RGN tank waste** was analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto Titrator. A 0.0103 N NaOH (ChemRec_72), solution was used as a standard and for the sample spike and the titrant was a 0.0051 M HCl prepared solution for all the samples. The attached Report Summary shows average OH molarity (1st inflection point) of 0.0036M on the sample and replicate results with an RPD of 11%, which is good considering the very weak base concentration of the sample being titrated. The hydroxide ug/ml results averaged 61+/- 4.6 ug/ml and was above the required MRQ value of 17 ug/mL which is equivalent to 0.01M NaOH. The hydroxide recoveries averaged 100% for the standards and the matrix spike recovery on 01-1732 was 102%. No hydroxide was detected in the reagent blank. The second inflection point frequently associated with carbonate, showed an RPD of 13%. There wasn't a third inflection point recorded for this sample. All of the results meet the QC acceptance criteria for spike recovery and RSD of duplicate measurements. The titration curves are included with the report.

Reviewer:
LRGreenwood 9/21/01

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

ASR 6174

WP# W58166

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

Report Summary for ASR # -- 6174

				Concentration, moles					
RPG #	**Client ID**		**OH conc ug/mL**	**First Point**	**RPD**	**Second Point**	**RPD**	**Third Point**	**RPD**
01-1732	**AN-102/C104 -RGN**		5.7E+01	**0.0034**		**0.0029**		**none**	
01-1732	**AN-102/C104 -RGN**	**Rep**	6.4E+01	**0.0038**	11%	**0.0026**	13%	**none**	
		Ave	6.1E+01	3.6E-03		2.8E-03			
		std de	4.6E+00	2.7E-04		2.5E-04			

Reag. Blk.1		**0**
Standard 3		**99%**
Standard 4		**100%**
MS 01-1732	**Matrix spike**	**102%**

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

Analyst: 9/21/01

Reviewer: LRGreenwood 9/21/01

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

8/2/01

Client : Fiskum

Cognizant Scientist: L.R. Greenwood Date : 8/2/01

Concur : T Trang-le Date : 8/2/01

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

ALO ID Client ID	Be-7 Error %	Co-60 Error %	Eu-152 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-1714	4.75E-4	3.55E-2	4.18E-4	5.49E-5	4.54E-4	2.95E-4	2.22E-2	1.28E-2	9.98E-3
AN102/C104L- F2	16%	2%	4%	34%	3%	5%	2%	5%	4%
01-1715	4.58E-4	3.79E-2	4.88E-4	8.83E-5	4.62E-4	2.55E-4	2.37E-2	1.44E-2	1.14E-2
AN102/C104L-F5	19%	2%	6%	33%	2%	8%	2%	3%	4%
01-1716	6.03E-4	4.01E-2	4.52E-4	9.03E-5	4.82E-4	2.10E-4	2.47E-2	1.54E-2	1.21E-2
AN102/C104L-F8	12%	2%	4%	26%	2%	6%	2%	4%	4%
01-1717	4.00E-4	3.34E-2	4.39E-4	<2.E-4	4.67E-4	1.40E-2	2.25E-2	1.39E-2	1.06E-2
AN102/C104P-F2	39%	2%	8%		3%	3%	2%	4%	4%
01-1718	<2.E-3	3.68E-2	<4.E-4	<5.E-4	4.36E-4	1.55E-2	2.37E-2	1.46E-2	1.17E-2
AN102/C104P-F6		2%			8%	2%	2%	3%	5%
01-1719	<4.E-4	3.58E-2	<1.E-4	<2.E-4	4.28E-4	1.21E-2	2.26E-2	1.29E-2	1.05E-2
AN102/C104P-F8		2%			4%	3%	2%	5%	4%
01-1720	<4.E-1	3.78E-2	<8.E-3	<1.E-1	<1.E-2	1.39E+2	<2.E-2	<8.E-2	<2.E-1
AN102/C104 F0		5%				3%			
01-1721	<7.E-4	4.00E-2	<2.E-4	<3.E-4	3.50E-4	1.73E-2	1.95E-2	1.19E-2	9.53E-3
AN102/C104 FD-1		2%			6%	2%	2%	3%	4%

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

ALO ID Client ID	Be-7 Error %	Co-60 Error %	Eu-152 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-1722	<2.E-4	6.27E-3	<3.E-5	<4.E-5	<2.E-5	1.76E-3	3.74E-4	2.49E-4	2.26E-4
AN102/C104 FD-5		2%				2%	3%	5%	7%
01-1723	<6.E-5	2.43E-3	<2.E-5	<2.E-5	1.28E-5	1.81E-3	1.16E-4	7.45E-5	4.70E-5
AN102/C104 FD-9		2%			10%	3%	4%	8%	27%
01-1724	<5.E-5	1.35E-3	<2.E-5	<2.E-5	9.00E-6	1.88E-3	5.78E-5	3.10E-5	3.65E-5
AN102/C104 FDI-4		2%			11%	3%	6%	6%	41%
01-1725	<4.E-5	7.02E-4	<2.E-5	<2.E-5	6.96E-6	7.03E-4	3.34E-5	<2.E-5	2.68E-5
AN102/C104 FDI-8		2%			12%	3%	7%		27%
01-1726	<2.E1	<6.E-2	<2.E-1	<6.E0	<3.E0	4.63E+3	<3.E-1	<4.E0	<4.E0
AN102/C104 LE-4DR						2%			
01-1727	<3.E1	<1.E-1	<5.E-1	<7.E0	<4.E0	8.52E+3	<3.E-1	<4.E0	<4.E0
AN102/C104 LE-5DD						2%			
01-1728	<4.E0	<4.E-2	<2.E-1	<2.E0	<7.E-1	1.13E+3	<1.E-1	<8.E-1	<2.E0
AN102/C104 LE-6DD						3%			
01-1729	<2.E-2	1.20E-3	<2.E-3	<6.E-3	<3.E-3	3.43E+0	<7.E-4	<3.E-3	<3.E-3
AN102/C104 LE-9DR		7%				2%			
01-1730	<6.E-3	6.91E-4	<7.E-4	<2.E-3	<2.E-4	7.21E-1	<5.E-4	<3.E-2	<2.E-1
AN102/C104 LE-12D		9%				3%			
01-1731	<3.E-4	2.13E-4	<2.E-5	<1.E-4	<5.E-5	5.64E-2	<2.E-5	<5.E-5	<5.E-5
AN102/C104 EDI-2		2%				2%			
01-1732	<4.E-4	1.17E-3	<2.E-5	<2.E-4	<9.E-6	7.54E-2	<2.E-5	<1.E-4	<2.E-4
AN102/C104 RGN		2%				3%			
01-1733	<9.E-4	3.50E-2	<3.E-4	<3.E-4	4.92E-4	1.42E-2	2.30E-2	1.38E-2	1.12E-2
AN102/C104 Fcomp#2		2%			5%	2%	2%	3%	4%

S. Fiskum

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

Project / WP#: 42365 / W60567
ASR#: 6281
Client: I. Burgeson
Total Samples: 4

RPL#:	02-00776	02-00779
Client ID:	Tc-IX-Eluate Composite (AN102/C104)	AN102/C104-CsE-Comp 1
Sample Preparation: 02-00776 to 02-00778 –PNL-ALO-129; 02-00779--10x dilution (SAL/vh). Not the same analytes of interest for each sample. See sample Results for analytes of interest.		

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

Analyst: D.R. Sanders

Analysis Date (File): 01-29-02 (A0761) & 02-19-02 (A0771)

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

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

MW Urie 3-28-02
Reviewed by

Renée Russell 3/28/02
Concur

Four AN102/C104 ion exchange samples (RPL# 02-00776….02-00779) were received under Analytical Service Request (ASR) 6281. Three of the samples, 02-00776 through 02-00778, were subjected to an acid digestion per PNL-ALO-128. The fourth sample, 02-00779, was simply diluted by 10x with 0.5M HNO_3 solution. Analytes of interest (AOIs) and additional 'opportunistic analytes' for each sample were specified in four individual tables attached to the ASR. Analytes other than those identified as AOIs or opportunistic analytes are included in the results for information only, since these analytes have not been fully evaluated for QC performance.

A summary of the ICPAES analysis results for the samples, as well as a summary of the QC performance, is given in the attached ICPAES Report (6 pages). Since the samples have different AOIs, the results for samples 02-00776 and 02-00777, sample 02-00778, and sample 02-00779 are presented in different tables, along with the applicable QC performance. The QC performance was evaluated for two batch runs, since samples 02-00776 through 02-00778 were prepared and analyzed in a single batch, with sample 02-00779 being analyzed in a different batch ICPAES measurement results are reported in µg/mL and have been corrected for dilution resulting from sample processing.

Quality control check-standard results met tolerance requirements for the specific AOIs except as noted below. Below is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan (applicable to process blank, post spike recoveries, and serial dilution tolerance) and QC parameter tables from the ASR (applicable to blank spike, matrix spike, and duplicate precision.

Analysis Run January 29, 2002 – Samples 02-00776 through 02-00778

Process Blanks:

No AOIs were detected in the process blank at concentrations above the method detection limit, thus meeting the requirement that process blank concentration be ≤ EQL.

Duplicate RPD (Relative Percent Difference):

Per the Quality Control Parameter tables included with the ASR, samples 02-00776 and 02-00777 have a precision requirement of <20% RPD and sample 02-00778 has a precision requirement of <15% RSD (<3.5% RSD for Na). As part of the batch processing for these samples, sample 02-00776 was selected for analysis in duplicate, and is used to estimate the precision for all samples processed in the batch. Unfortunately, only Na and B were detected at concentrations above the EQL in sample 02-00776, and are the only results available for accessing precision. It should be noted that no instructions were provided in the ASR to perform replicate analyses for 'each' sample.

Blank Spike:

For the blank spike control sample analyzed with samples 02-00776 through 02-00778, all AOI were within the tolerance limit of 80% to 120% recovery except for K, which had a recovery of 34%. Both the blank spike and matrix spike are from the same spiking solution and both demonstrate very low K recoveries. Low K recoveries have been experiences when the blank spiking solution has been transferred to the SAL hot cells in polycarbonate vials. This may be an explanation for the low K recovery. However, this can not be verified since the containers used for transferring the spiking solution were discarded prior to discovering the low K recovery problem. Use of polycarbonate containers has been discontinued.

Matrix Spiked Sample:

A matrix spike was prepared by adding 0.5 ml each of stock multi-element standards 'BPNL-QC part 1' and 'BNPL-QC part 2' to 1.0 mL of sample 02-00777, processing, and diluting to a final volume to 25 mL. All AOI were within the tolerance limit of 75% to 125% recovery except for K, Al, Na, Co, and Sn. K had a recovery of only 35% (See blank spike explanation). Co and Sn were not included in either 'part 1' or 'part 2' of the spiking solution. The matrix spike analysis uses a general multi-element spiking solution intended to be usable on the majority of samples analyzed by ICPAES. However, for the sample selected for matrix spiking, the spike concentration for Al and Na were less than 20% of the sample concentration and the recovery results are not considered valid. For Al and Na, the use of serial dilution results is used to evaluate potential matrix interferences.

Post-Spiked Samples (Group A):

Post spike analysis for 'Group A' analytes was performed on sample 02-00776. All post-spiked AOIs and opportunistic analytes in the sample were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

Post spike analysis for 'Group B' analytes was performed on sample 02-00776. There are no AOIs in the 'Group B' spiking solution; however, the opportunistic analytes in the sample were recovered within tolerance of 75% to 125%.

Serial Dilution:

Serial dilution was performed on sample 02-00777. All AOIs above EQL were within tolerance limits of 10% after correcting for dilution.

Analysis Run February 19, 2002 – Sample 02-00779

Process Blanks:

No processing, other than dilution with acid, was performed; thus no process blank was prepared. However, the dilution matrix (0.5 M HNO_3) was analyzed and reported. No AOIs were detected in the dilution blank at concentrations above the method detection limit.

Duplicate RPD (Relative Percent Difference):

Per the Quality Control Parameter tables included with the ASR, sample 02-00779 has a precision requirement of <20% RPD . All AOIs measured above the EQL demonstrated good precision, with %RPDs less than 5%.

Blank Spike:

No sample processing, other than dilution with acid, was performed on the sample; thus no blank spike sample was prepared.

Matrix Spiked Sample:

No sample processing, other than dilution with acid, was performed on the sample; thus no matrix spike sample was prepared.

Post-Spiked Samples (Group A):

All post-spiked AOIs and opportunistic analytes in the sample were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

There are no AOIs in the 'Group B' spiking solution; however, the opportunistic analytes in the sample were recovered within tolerance of 75% to 125%.

Serial Dilution:

All AOIs above EQL were within tolerance limits of 10% after correcting for dilution.

Comments:

1) The "Final Results" have been corrected for all laboratory dilutions performed on the sample during processing and analysis unless specifically noted.

2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.

3) Routine precision and bias is typically ± 15% or better for samples in dilute, acidified water (e.g. 2% v/v HNO_3 or less) at analyte concentrations greater than ten times detection limit up to the upper calibration level. This also presumes that the total dissolved solids concentration in the sample is less than 5000 µg/mL (0.5 per cent by weight).

4) Absolute precision, bias and detection limits may be determined on each sample if required by the client.

5) The maximum number of significant figures for all ICP measurements is 2.

	Run Date=	1/29/2002	1/29/2002	1/29/2002	1/29/2002	1/29/2002
	Multiplier=	24.9	24.9	24.9	24.9	124.7
	RPL/LAB #=	PB-00776	02-00776	02-00776-DUP	02-00777	02-00777 @5
Det. Limit	*Client ID=*	*process blank*	*Tc-IX-Eluate*	*Tc-IX-Eluate-Dup*	*AN102/C104Cs-Rem*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	--	[9.0]	[8.6]	7,840	
0.010	Ba	--	--	--	--	
0.250	Ca	--	--	--	149	
0.015	Cd	--	--	--	24.2	
0.050	Co	--	--	--	[1.9]	
0.020	Cr	--	--	--	98.2	
0.025	Cu	--	--	--	--	
0.025	Fe	--	--	--	[2.0]	
2.000	K	--	--	--	1,080	
0.050	La	--	--	--	--	
0.100	Mg	--	--	--	--	
0.050	Mn	--	--	--	--	
0.050	Mo	--	--	--	23.1	
0.150	Na	--	279	280	over range	110,000
0.030	Ni	--	--	--	145	
0.100	Pb	--	--	--	69.1	
0.500	Si	--	[26]	[26]	179	
1.500	Sn	--	--	--	--	
0.025	Ti	--	--	--	--	
0.050	Zn	--	--	--	[4.0]	
Other Analytes						
0.025	Ag	--	--	--	--	
0.250	As	--	--	--	--	
0.050	B	--	20.0	19.9	86.4	
0.010	Be	--	--	--	--	
0.100	Bi	--	--	--	--	
0.200	Ce	--	--	--	--	
0.050	Dy	--	--	--	--	
0.100	Eu	--	--	--	--	
0.030	Li	--	--	--	--	
0.100	Nd	--	--	--	[3.6]	
0.100	P	--	--	--	562	
0.750	Pd	--	--	--	--	
0.300	Rh	--	--	--	--	
1.100	Ru	--	--	--	--	
0.500	Sb	--	--	--	--	
0.250	Se	--	--	--	--	
0.015	Sr	--	--	--	86.9	
1.500	Te	--	--	--	--	
1.000	Th	--	--	--	--	
0.500	Tl	--	--	--	--	
2.000	U	--	--	--	--	
0.050	V	--	--	--	--	
2.000	W	--	--	--	--	
0.050	Y	--	--	--	--	
0.050	Zr	--	--	--	--	

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

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

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

QC Performance 1/29/02 -- Applicable to Batch Containing 02-00776, 02-00777, and 02-00778

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	75%-125%	< +/-10%
Batch QC ID=	02-00776 Dup	LCS/BS	02-00777 MS	02-00777 MS (@5)	02-00776 + Post Spike A	02-00776 + Post Spike B	02-00777 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff
Al		100	nr		100		1.8
Ba		100	94		99		
Ca		101	101		103		
Cd		101	101		103		-0.5
Co					105		
Cr		101	109		104		2.9
Cu		103	99		103		
Fe		104	100		105		
K		34	35		97		
La (a)		101	98			102	
Mg		100	98		108		
Mn		103	99		105		
Mo (a)		103	100		102		
Na	0.4	102	over range	nr	100		3.6 (b)
Ni		103	106		107		3.3
Pb		118	117		120		
Si (a)		9	4		116		
Sn (a)						98	
Ti (a)		98	92		98		
Zn		103	101		104		
Other Analytes							
Ag					99		
As					103		
B	0.3	104	99		101		1.3
Be		98	98		101		
Bi		104	105		100		
Ce		100	98			99	
Dy						103	
Eu						103	
Li		103	98		101		
Nd		101	97			101	
P		101	107		103		-0.2
Pd						80	
Rh						98	
Ru							
Sb					103		
Se					102		
Sr		102	nr		102		2.5
Te						110	
Th		99	96			101	
Tl					100		
U		105	99			105	
V		97	93		98		
W		nr	103				
Y					99		
Zr		95	90		101		

Shaded results did not meet the acceptance criteria

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

(a) Opportunistic analytes; no LCS or spiking required.

(b) Value obtained from @5 and @25 dilutions.

	Run Date=	1/29/2002	1/29/2002	1/29/2002
	Multiplier=	24.9	24.9	124.7
	RPL/LAB #=	PB-00776	02-00778	02-00778 @5
Det. Limit	*Client ID=*	*process blank*	*Tc-IX-Effluent*	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	--	7,890	
0.010	Ba	--	--	
0.250	Ca	--	146	
0.015	Cd	--	23.4	
0.020	Cr	--	96.4	
0.025	Fe	--	[2.0]	
2.000	K	--	1,060	
0.050	La	--	--	
0.100	Mg	--	--	
0.150	Na	--	over range	109,000
0.030	Ni	--	141	
0.100	P	--	618	
0.100	Pb	--	67.5	
2.000	U	--	--	
Other Analytes				
0.025	Ag	--	--	
0.250	As	--	--	
0.050	B	--	37.7	
0.010	Be	--	--	
0.100	Bi	--	--	
0.200	Ce	--	--	
0.050	Co	--	[1.9]	
0.025	Cu	--	--	
0.050	Dy	--	--	
0.100	Eu	--	--	
0.030	Li	--	--	
0.050	Mn	--	--	
0.050	Mo	--	22.5	
0.100	Nd	--	[3.5]	
0.750	Pd	--	--	
0.300	Rh	--	--	
1.100	Ru	--	--	
0.500	Sb	--	--	
0.250	Se	--	--	
0.500	Si	--	[61]	
1.500	Sn	--	--	
0.015	Sr	--	85.3	
1.500	Te	--	--	
1.000	Th	--	--	
0.025	Ti	--	--	
0.500	Tl	--	--	
0.050	V	--	--	
2.000	W	--	--	
0.050	Y	--	--	
0.050	Zn	--	[4.2]	
0.050	Zr	--	--	

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

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

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

QC Performance 1/29/02 -- Applicable to Batch Containing 02-00776, 02-00777, and 02-00778

Criteria>	<15% (Na <3.5%)	80% - 120%	75%-125%	75%-125%	75%-125%	75%-125%	< +/-10%
Batch QC ID=	02-00776 Dup	LCS/BS	02-00777 MS	02-00777 MS (@5)	02-00776 + Post Spike A	02-00776 + Post Spike B	02-00777 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Rec	%Diff
Al		100	nr		100		1.8
Ba		100	94		99		
Ca		101	101		103		
Cd		101	101		103		-0.5
Cr		101	109		104		2.9
Fe		104	100		105		
K		34	35		97		
La (a)		101	98			102	
Mg		100	98		108		
Na	0.4	102	over range	nr	100		3.6^(a)
Ni		103	106		107		3.3
P (a)		101	107		103		-0.2
Pb		118	117		120		
U		105	99			105	
Other Analytes							
Ag					99		
As					103		
B	0.3	104	99		101		1.3
Be		98	98		101		
Bi		104	106		100		
Ce		100	98			99	
Co					105		
Cu		103	99		103		
Dy						103	
Eu						103	
Li		103	98		101		
Mn		103	99		105		
Mo		103	100		102		
Nd		101	97			101	
Pd						80	
Rh						98	
Ru							
Sb					103		
Se					102		
Si		9	4		116		
Sn						98	
Sr		102	nr		102		2.5
Te						110	
Th		99	96			101	
Ti		98	92		98		
Tl					100		
V		97	93		98		
W		nr	103				
Y					99		
Zn		103	101		104		
Zr		95	90		101		

Shaded results did not meet the acceptance criteria

n.r. = not recovered, spike concentration less than 20% of sample concentration or measured sample concentration <EQL.

(a) Opportunistic analytes, no LCS or spiking required.

(b) Value obtained from @5 and @25 dilutions.

	Run Date=	2/19/2002	2/19/2002	2/19/2002
	Multiplier=	1.0	10.0	10.0
	RPL/LAB #=	02-00779-DB	02-00779	02-00779 DUP
Det. Limit	*Client ID=*	*Diluent Blank*	*AN102/C104-CsE-Comp 1*	*AN102/C104-CsE-Comp 1-Dup*
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)
0.060	Al	--	[5.7]	[5.8]
0.010	Ba	--	[0.16]	[0.19]
0.250	Ca	--	--	--
0.015	Cd	--	2.31	2.38
0.050	Co	--	--	--
0.020	Cr	--	22.5	23.3
0.025	Cu	--	30.1	31.1
0.025	Fe	--	6.16	6.56
2.000	K	--	--	--
0.050	La	--	--	--
0.100	Mg	--	--	--
0.050	Mn	--	--	--
0.050	Mo	--	--	--
0.150	Na	--	775	810
0.030	Ni	--	66.6	68.9
0.100	Pb	--	24.8	25.4
0.500	Si	--	[20]	[20]
1.500	Sn	--	--	--
0.025	Ti	--	--	--
0.050	Zn	--	[2.7]	[2.8]
Other Analytes				
0.025	Ag	--	--	--
0.250	As	--	--	--
0.050	B	--	7.28	7.47
0.010	Be	--	--	--
0.100	Bi	--	--	--
0.200	Ce	--	--	--
0.050	Dy	--	--	--
0.100	Eu	--	--	--
0.030	Li	--	--	--
0.100	Nd	--	--	--
0.100	P	--	--	--
0.750	Pd	--	--	--
0.300	Rh	--	--	--
1.100	Ru	--	--	--
0.500	Sb	--	--	--
0.250	Se	--	--	--
0.015	Sr	--	[1.0]	[1.1]
1.500	Te	--	--	--
1.000	Th	--	--	--
0.500	Tl	--	--	--
2.000	U	--	[170]	[180]
0.050	V	--	--	--
2.000	W	--	--	--
0.050	Y	--	--	--
0.050	Zr	--	--	--

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

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

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

QC Performance 02/19/02 - Applies to Sample 02-0779

Criteria>	<20%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%
QC ID=	02-00779 & 02-00779-D	LCS/BS (none)	MS (none)	02-00779 + Post Spike A	02-00779 + Post Spike B	02-00779 @1/@5 Serial Dil
Analytes	RPD (%)	%Rec	%Rec	%Rec	%Rec	%Diff
Al				99		
Ba				98		
Ca				101		
Cd	3.2			102		
Co				105		
Cr	3.7			101		0.2
Cu	3.4			101		0.2
Fe	6.3			101		
K				101		
La (a)					99	
Mg				106		
Mn				104		
Mo (a)				101		
Na	4.4			98		0.3
Ni	3.4			98		0.9
Pb	2.4			104		
Si (a)				106		
Sn (a)					92	
Ti (a)				96		
Zn				102		
Other Analytes						
Ag				104		
As				100		
B	2.6			100		
Be				103		
Bi				101		
Ce					98	
Dy					102	
Eu					102	
Li				100		
Nd					100	
P				100		
Pd					93	
Rh					100	
Ru						
Sb				99		
Se				103		
Sr				100		
Te					100	
Th					99	
Tl				100		
U					104	
V				96		
W						
Y				98		
Zr				100		

Shaded results exceed acceptance criteria

(a) Opportunistic analytes; no LCS or spiking required.

Battelle PNWD
PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 2

Project / WP#:	42365 / W58950
ASR#:	6281
Client:	Ingrid Burgeson
Total Samples:	7

Analysis: Cs and Tc-99

Procedure: 329-OP-SCO1 Rev. 0, *Inductively-Coupled Plasma Mass Spectrometry (ICP/MS) Analysis*

M&TE Number:	WB36913	ICP/MS, VG Elemental
	512-06-01-014	Mettler AJ100 Balance

Point of Contact: Orville Thomas Farmer III

Revised Report Date: 2 February 2004

Analysis Files:
Experiment – 05feb02
Procedure – 020205a, 020201a
Element Menu – rbtc, 1feb02

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

Analyst(s): James Bramson / Teresa Wilson
Original Author: Orville Thomas Farmer III

Tom P Thomas — 2 Feb 04
Revised By — Date

Orville Thomas Farmer — 2 Feb 04
Reviewed By — Date

Revision	Effective Date	Description of Change
1	January 2004	Text has been added in the appropriate narrative section(s) to address issues surrounding the use of biased standards prepared and provided by the RPL Standards Laboratory to the ICP-MS analytical laboratory (reference: project #98620, memo KN Pool to GH Beeman, 9/15/03).
2	February 2004	Tc-99 results for the AN102/C104W/Cs Removed (02-777) and Tc-IX Effluent (02-778) samples were inadvertently switched in Rev. 1 of this report. This revision corrects the Tc-99 result table.

Samples Submitted for Analysis:

RPL #	Client I.D.
PB-776	Reagents Only
02-776	Tc-IX-Eluate
DUP-776	Tc-IX-Eluate-Dup
02-777	AN102/C104W/Cs Removed
02-778	Tc-IX Effluent
PS-776	Reagents Spikes
MS-776	Spiked & AN102/C104/Cs Removed

The samples (AN102/C104) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Tc-99.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis.

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

2. Results

Cs-133 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
PB-776	<1.04E-04		1.04E-04
02-776	<2.85E-02		2.85E-02
DUP-776	<2.66E-02		2.66E-02
02-777	1.33E-03	6.77E-04	6.21E-04
02-778	<2.40E-03		2.40E-03
PS-776	1.20E+00	6.92E-03	2.53E-05
MS-776	3.13E+01	6.49E-02	6.57E-03

Tc-99 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
PB-776	6.56E-06	1.07E-06	6.89E-07
02-776	1.86E-01	1.92E-03	1.89E-04
DUP-776	1.74E-01	8.18E-03	1.77E-04
02-777	6.41E-02	2.14E-03	1.94E-05
02-778	3.59E-02	2.83E-04	1.59E-05
PS-776	4.52E-04	3.58E-06	8.22E-07
MS-776	7.79E-02	8.10E-03	2.15E-04

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 1% high purity nitric acid solution used as the diluent for the samples. The QC criteria of less than < 10 X MDL, was met for all analytes.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT). The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS). Sample solution (02-777) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (02-777) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD). Sample solution ICSD (02-777) is a duplicate preparation of the above sample and is used to determine the RPD between the two solutions as an ICP/MS instrument and preparation QC. The duplicate analysis of the ICS and ICSD (02-777) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS). The ICSDS is a post matrix spike of sample (02-777) and is used to provide information on instrument solution preparation and instrument performance. The ICSDS met the QC success criteria of ± 25% recovery for all analytes.

Post Blank Spike (PBS). An instrument post spike was performed on sample (PB 02-776) and met the QC success criteria of ± 20% recovery for all analytes.

Internal Standard (IS). The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB). The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS). The LCS/BS met the QC success criteria is + 20% recovery for Cs. Tc-99 was not spiked in the LCS/BS, however a PBS was analyzed and Tc-99 met the success criteria of +20%.

Duplicate (DUP). All elements met the success criteria of + 20% RPD except Cs which was < 10XMDL. However the ICS and ICSD instrument QC samples met the success criteria of + 20% RPD for all analytes analyzed.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD). No MSD was submitted for analysis. The MS for Cs met the success criteria of + 25%. Tc-99 was not spiked in the MS, however an ICSDS (matrix post spike) was analyzed and Tc-99 and met the success criteria of +25%.

Laboratory Control Standard (LCS). No LCS was submitted for analysis.

Analytical results generated were influenced by an identified bias in the calibration and calibration verification standards. The bias in the standards caused results for Tc-99 to be biased low for this report. The effect on the results, based on the observed bias, will widen the error band for these results. The error band that should be applied to these results is -10% to +30%.

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Project No.

Internal Distribution
File/LB

Date February 26, 2002

To Sandy Fiskum

From Stan Bos Stan Bos

Subject Cesium isotopic analysis (RPL # 02-00779)

Sandy

Cesium isotopic distribution analyses of samples AN102-C104 and An102-C124 (dup) have been completed. Approximately two micro liters of the sample was plated on a rhenium carbide filament and analyzed on the single stage mass spectrometer (M&TE # WB76849) in accordance with PNNL technical procedure PNNL-98523-264. Since natural cesium in mono-isotopic, a sample of rubidium chloride isotopic standard (NBS 984) was run to standardize the mass spectrometer. Work package W60553 will be charged for the analysis.

In addition to the samples a diluent blank was also analyzed. A small ^{133}Cs signal was detected for about 1 minute before it died away. During that time no ^{137}Cs could be detected. Do to the low concentration of cesium no attempt was made to do isotopic distribution analysis.

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

Sample Id. AN-102 / C104 RPL # 02-00779

	^{133}Cs	^{135}Cs	^{137}Cs
	Weight percent	Weight percent	Weight percent
AN-102 / C104	60.4 ± 0.5	16.7 ± 0.2	22.9 ± 0.2
AN-102 / C104 (dup)	60.2 ± 0.5	16.7 ± 0.2	23.1 ± 0.2
Diluent Blank	Unable to analyze	Not detected	Not detected

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

Concurrence 2-26-02

D. Fiskum

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

Client:	I. Burgeson	**Charge Code/Project:**	W60567 / 42365
RPL Numbers:	02-00776 to 02-00779	**ASR Number:**	6281
Analyst:	MJ Steele	**Analysis Date:**	March 7, 2002

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)

Analysis Results

		TIC			TOC			TC	
RPL Number	**Sample ID**	**MDL µgC/mL**	**Results µgC/mL**	**RPD**	**MDL µgC/mL**	**Results µgC/mL**	**RPD**	**Results µgC/mL**	**RPD**
02-00776	Tc-IX- Eluate Composite (AN102/C104)	26	110		72	140		250	
02-00777	AN2-Tc-O-C	26	9,100		72	12,300		21,400	
02-00779	AN-102/C104-CsE-Comp 1	26	30		72	130		160	
02-00778	Tc-IX- Effluent Composite (AN102/C104)	26	7,800		72	11,200		19,000	
02-00778 Dup	Tc-IX- Effluent Composite (AN102/C104)	65	7,900	1%	180	11,400	2%	19,300	1%
02-00778 MS	Recovery		104%			110%		107%	
Blank Spike/LCS	Recovery		100%			100%			

RPD = Relative Percent Difference

The TOC/TIC analyses of the samples submitted under ASRs 6281 are to be 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.

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

Q.C. Comments:

The TIC analysis uses calcium carbonate and the TOC uses α-D-Glucose as the calibration, laboratory control, and matrix spike standars. (The JT Baker, Aldrich, Sigma, and Mallinckrodt chemical lot numbers and Chemical Measurement System numbers are provided on the raw data benchsheets).

The QC for the methods involves calibration blanks, sample duplicates, laboratory control sample, and matrix spikes per analysis batch. The ASR indicates that the analyses are to be performed per the QA Plan "Conducting Analytical Work in Support of Regulatory Programs" Sections 4 and 5; the performance of the QC samples is compared to this Plan.

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

Laboratory Control Sample (LCS)/Blank Spike(BS): At 100% recovery for both TIC and TOC, the LCS/BS samples recovered well within acceptance criteria of 80% to 120%.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The matrix spike recoveries (104% for TIC and 110% for TOC) are within the acceptance criteria of 75% to 125% recovery.

Duplicates: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD) between sample and duplicate. With RPDs less than 2%, the TIC and TOC RPD results are within the QP Plan acceptance criteria of <20% RPD.

MDL/MRQ: Except for the duplicate analysis for the Tc-IX-Effluent sample, all estimated MDLs for the TIC and TOC measurements are less that one-third of the minimum reportable quantities for TIC (150 µg/mL) and TOC (1500 µg/mL).

- The reported "Final Results" have been corrected for all dilution performed on the sample during processing or analysis.
- Routine precision and bias are typically ±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. The analysis MDLs (total ug C) are based on 3 times the standard deviation of a set of historical data. The sample MDLs (in ug C/ml or ug C/g) are calculated by using the analysis MDL adjusted for the sample volume or weight.
- 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: ____________________ **Date** 3-8-02

Review/Approval by: ____________________ **Date** 3-8-02

Excel Archive File: ASR 6281 Burgeson.xls

Battelle

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Client:	Ingrid Burgeson	Date: 01/16/02
Subject:	Hydroxide Analyses for: AN2-Tc-O-C	
ASR:	6281	

Two samples of **AN-102 / C-104 tank waste** (the first, 02-0777 with Cs removed and the second, 02-0778 with Cs and Tc removed) were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228. These samples were analyzed using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH (ChemRec_57) solution was used as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution for the liquid fraction. The attached Report Summary indicates good RPD on the OH molarity (1st inflection point) on the sample and replicate results. The hydroxide results were all well below the required MRQ value of 7.5E+04 ug/mL. The hydroxide standard recovery was 98 % and the matrix spike recovery was 96%. No hydroxide was detected in the reagent blank. The second and third inflection points frequently associated with carbonate and bicarbonate, showed excellent RPD, well below the +/- 20% required. All of the results meet the QC acceptance criteria for spike recovery and RSD of duplicate measurements. Following is the report summary, the sample results calculated from the raw data, and the record file for the standardized acid and base used. Also included in this report are copies of the titration curves.

Prepared by: ________ Date: 1-17-02

Reviewed by: ________ Date: 1-17-02

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

ASR 6281

WP# W60567

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

Report Summary for ASR # -- 6281

RPG #	Client ID		OH conc ug/mL	Concentration, moles / Liter First Point	RPD	Second Point	RPD	Third Point	RPD
02-0777	AN2-Tc-O-C		3.1E+03	0.18		1.29		0.84	
02-0777	AN2-Tc-O-C	Rep	2.9E+03	0.17	6%	1.29	0%	0.86	3%
02-778	Tc-IX-Effluent		3.6E+03	0.21		1.21		0.79	
02-778	Tc-IX-Effluent	Rep	3.6E+03	0.21	0%	1.16	4%	0.81	3%

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

Reag. Blk.1		0
Standard 3		98%
MS 02-778	**Matrix spike**	96%

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

Analyst: 1-17-02

Reviewer: 1-18-02

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Project No. 42365

Internal Distribution
File/LB

Date March 12, 2002

To I. Burgeson and S. Fiskum

From L. R. Greenwood

Subject Radiochemical Analyses for ASR 6281

Samples of the Tc-IX eluate composite and effluent for tanks AN102 and C104 were analyzed for gamma emitters, ^{90}Sr, total alpha, Am/Cm, U and Tc as pertechnetate according to ASR 6281. The analyses were performed on direct sample material prepared by acid digestion in the laboratory. The attached reports list measured analyte activities in units of µCi/ml. The reported errors (1-σ) represent the total propagated error including counting, dilution, yield, and calibration errors, as appropriate. Laboratory and process blank values given with each analysis are the best indicators of the method detection limits, taking into account the actual sample sizes and counting times used for each analysis.

Gamma Spectrometry

Sample aliquots were directly counted for gamma emitters according to procedure PNL-ALO-450. Since no sample preparation was involved, no laboratory blanks or spikes were prepared for these analyses other than the standard laboratory control samples and background counts. Measured activities or detection limits generally met the requested MRQ values except for sample 02-779 where the high level of Cs-137 made it impossible to meet the MRQ values after 14 hours of counting. ^{95}Tc and ^{95m}Tc dominated the gamma spectrum for sample 02-0776.

Total Alpha

The total alpha activity was determined by evaporating small aliquots of the samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. Alpha activity was detected in all of the samples except for 02-0779; however, the detection limits were well below the requested MRQ value. Blank and matrix spikes gave good recovery and no alpha activity was detected in the reagent blanks.

Americium and Curium

The Am/Cm separations were performed according to PNL-ALO-417 for sample 02-0779 only. This test was cancelled for sample 02-0776 since the GEA detection limit was well below the requested MRQ value, as specified on the ASR. The separated fractions were precipitation plated according to PNL-ALO-496 and the samples were counted by alpha spectrometry according to PNL-ALO-422. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. The americium radiochemical yields were acceptable, averaging about 96%. The LCS and matrix spike recoveries were acceptable at 95-98%. No Am or Cm activities were detected in the reagent blanks. The ^{241}Am activities measured by alpha energy analysis are in very good agreement with the GEA data.

Strontium-90

The Sr separation was performed according to PNL-ALO-476 and radiochemical yields were traced with ^{85}Sr. The separated fractions were then beta-counted according to RPG-CMC-408 and gamma counted according to PNL-ALO-450 (for ^{85}Sr determination and ^{137}Cs impurity assessment). ^{137}C was not observed in the gamma counting, indicating a clean strontium separation. The process blank prepared with the acid digestion in the laboratory was found to have a low level of ^{90}Sr contamination that is negligible except for sample 02-0776 where the level in the blank is about 10% of the sample activity. The reagent blank did not show any contamination. The LCS and matrix spike showed good recovery at nearly 100% in both cases. Duplicate analyses showed acceptable agreement with RPD values of about 11-16%. The detection limits as well as many of the measurements were well below the requested MRQ values.

Uranium

Uranium was measured by kinetic phosphorescence analysis (KPA) according to procedure PNL-ALO-4014. Duplicate analyses were in good agreement with RPD values below 3%. LCS and matrix spike recoveries were nearly 100% and laboratory blanks did not show any uranium contamination. The uranium measurements and detection limits were well below the requested MRQ values.

Tc-99 Not done yet.

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

03/06/02

Client : I. Burgeson
ASR: 6281

Cognizant Scientist: LRGreenwood   Date : 3/6/02

Concur : T Trang-le   Date : 3/7/02

Reference Date: Feb 4, 2002 for 02-776 & 777, 778
Reference Date: Feb 22, 2002 for 02-779
Procedure: PNL-ALO-450 for GEA & PNL-ALO-476 Sr-90

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

ALO ID Client ID	Sr-90 Error %	Co-60 Error %	Tc-95 Error %	Tc-95M Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
02-776 BLK Process Blank	1.16E-4 22%										
02-776 Tc-1X-Eluate Composite AN102/C101	1.02E-3 4%	6.05E-5 5%	9.24E-5 7%	1.80E-3 2%	<7.E-6	<7.E-6	1.55E-5 34%	<2.E-5	5.00E-5 9%	<3.E-5	<5.E-5
02-776 REP Tc-1X-Eluate Composite AN102/C101	1.14E-3 4%										
RPD	11%										
02-777 AN2-Tc-O-C	1.52E+0 3%	3.41E-2 2%			4.34E-4 4%	<4.E-5	1.40E-2 4%	<7.E-5	2.20E-2 2%	1.23E-2 6%	1.04E-2 4%
02-778 Tc-1X-Effluent-Composite AN102/C104 Removed	1.50E+0 3%	3.16E-2 2%			4.49E-4 3%	<6.E-5	1.35E-2 3%	<9.E-5	2.10E-2 2%	1.21E-2 4%	1.03E-2 4%
02-778 DUP Tc-1X-Effluent-Composite AN102/C104 Removed	1.68E+0 3%										
RPD	11%										

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

ALO ID Client ID	Sr-90 Error %	Co-60 Error %	Tc-95 Error %	Tc-95M Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
02-779	7.19E-2	<7.E-3			<7.E-2	<7.E-3	6.65E+2	<9.E-3	<8.E-3	<3.E-1	<6.E-1
AN102/C104-CsE-Comp 1	55%						3%				
02-779 Dup	8.41E-2										
AN102/C104-CsE-Comp 1	47%										
RPD	16%										
Blank Spike	100%										
Blank Spike 02-779	97%										
Matrix Spike 02-778	102%										
Matrix Spike 02-779	99%										
Lab Blank	<5.E-5										
Lab Blank 02-779	<6.E-6										

Note: Samples were analyzed for Sr-90 in two batches.

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

03/07/02

Client : I. Burgeson
ASR: 6281

Cognizant Scientist: L. R. Greenwood Date : 3/7/02

Concur : T Trang-le Date : 3/7/02

Procedure: PNL-ALO-417/496/422 Americium
Procedure: PNL-ALO-420 & 421 for Alpha

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

ALO ID Client ID	Alpha Error %	Am-241 Error %	Cm-243+ Cm-244 Error %	Cm-242 Error %
02-776 Tc-1X-Eluate Composite AN102/C101	2.40E-5 26%			
02-777 AN2-Tc-O-C	1.10E-2 9%			
02-778 Tc-1X-Effluent-Composite AN102/C104 Removed	1.12E-2 10%			
02-778 DUP Tc-1X-Effluent-Composite AN102/C104 Removed	1.21E-2 9%			
RPD	8%			
02-779 AN102/C104-CsE-Comp 1	<2.E-2	1.22E-3 3%	6.12E-5 13%	<6.E-6
02-779 DUP AN102/C104-CsE-Comp 1	<2.E-2	1.13E-3 4%	6.88E-5 13%	<7.E-6
RPD		8%	12%	
Blank Spike	109%			
Blank Spike 02-779	104%	95%		
Matrix Spike	107%			
Matrix Spike 02-779	108%	98%		
Blank	<2.E-5			
Blank 02-779	<2.E-6	<7.E-7	<6.E-7	<3.E-7

Note: Samples were analyzed in two batches.

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

filename 02-0776
3/5/2002

Client: I. Burgeson
ASR 6281

Report prepared by: C Soderquist 3-5-02

Concur: 3-5-02

Procedure RPG-CMC-4014 Rev 1, Uranium Analysis by Kinetic Phosphorescence
Chem-Chek Instruments model KPA 11R uranium analyzer

Sample	Lab ID	Measured uranium concentration, µg U per mL ± 1s		Detection Limit
Tc IX Eluate Composite, AN102/C104	02-0776	**1.77E-3**	**± 2%**	4.E-4
	02-0776 Dup	**1.80E-3**	**± 2%**	4.E-4
AN2-Tc-0-C	02-0777	**8.22E-1**	**± 2%**	4.E-2
Tc IX Effluent Composite, AN102/C104	02-0778	**6.98E-1**	**± 2%**	4.E-2
AN102/C104-CsE-Comp 1	02-779	**1.69E+2**	**± 4%**	2.E-1
	02-779 Dup	**1.65E+2**	**± 4%**	2.E-1
	Blank 0776	2.27E-4	± 5%	
	Blank 0777	3.15E-2	± 4%	
	Blank 0779	1.55E-1	± 5%	
	Matrix spike 0776	98%		
	Matrix spike 0779	103%		
	LCS 0776	102%		
	LCS 0779	103%		

Notes: 1) The suffix letter “F” indicates the sample was filtered; “A” indicates that the sample was loaded out of the hot cell into a clean analytical vial; the suffix letter “D” indicates the sample was diluted prior to submission for analysis. Sample results not identified with the above samples are associated with other tests unrelated to this work.

Appendix E

Cesium Ion Exchange and Batch Contacts Testing Personnel

Appendix E: Cesium Ion Exchange and Batch Contacts Testing Personnel

Cognizant Scientists
S. K. Fiskum
D. L. Blanchard
S. A. Arm
B. M. Rapko

Hot Cell Technicians
F. V. Hoopes
M. A. Mann

Analytical Support
S. J. Bos
J. P. Bramson
L. P. Darnell
O. T. Farmer, III
S. K. Fiskum
L. R. Greenwood
D. R. Sanders
C. Z. Soderquist
M. J. Steele
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Neguib Hassan
PO Box 616, Road 1
Building 773-A
Aiken, South Carolina 29808

Jim Marra
PO Box 616, Road 1
Building 773-43A
Aiken, South Carolina 29808

Charles Nash
PO Box 616, Road 1
Building 773-42A
Aiken, South Carolina 29808

Robert Pierce
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Building 773-A
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