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

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ST Arm
DL Blanchard, Jr
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February 2004

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

LEGAL NOTICE

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for W. L. Tamosaitis
4/14/04

ACCEPTED FOR
PROJECT USE

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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 CHG-TP-41500-013, Rev. 0 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	September 2002	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
		Edited last paragraph in section 3.2.2 to reflect noted bias in total ^{99}Tc results.	
		Deleted footnote 11 on page 3.6.	
		Noted higher uncertainty in ^{99}Tc, ^{237}Np, ^{239}Pu, ^{240}Pu and AMU-241 values in Table 3.5 as footnotes. Renumbered Table 3.5 footnotes. Deleted the highlight around the ^{151}Sm result.	
		Replaced the following ICP-MS reports with revisions: Rev. 3 CsRbTc, actinides narrative report for ASR 6031, Rev. 1 CsTcRb narrative report for ASR 6121, Rev. 1 Tc-99 report for ASR 6192, Rev. 2 amu-241, Np-237, Pu-239 and Pu-240 report for ASR 6192, Rev. 1 I-129 report for ASR 6192.	
		Split Appendix B into two appendices. New Appendix C and the following appendices were re-lettered.	

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)[1] task 8.2.6 and the RPP-WTP Research and Technology schedule[2] 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-42a, which is included in Appendix C of the *Research and Technology Plan* [2]. 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 AP-101 diluted feed (AP-101DF); produce and characterize the Cs eluate for use in evaporation tests; remove ^{137}Cs from the AP-101DF to meet LAW vitrification criteria; and develop batch distribution coefficient values for AP-101DF. 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 BNI for calculating Na_2O loading was later reduced to 14 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 Hanford tank waste 241-AP-101 (Envelope A) diluted to 5 M Na^+ (AP-101DF). Batch contacts were performed with the waste at three Cs concentrations with a phase ratio of 100 (liquid volume to exchanger mass ratio) with both SL-644 and crystalline silicotitanate (IE-911, an alternative Cs ion exchanger). Ion exchange processing was conducted with two small columns in series with resin bed volumes (BVs) of 10.6 mL (L/D = 4.4) during the conditioning phase with 0.25 M NaOH, and 9.7 mL (L/D = 4) during the AP-101DF loading phase. Proper functioning of the ion exchange apparatus and resin beds had initially been tested with an AW-101 simulant. Approximately 1.2 L, equivalent to 112 BVs of AP-101DF waste were processed. All ion exchange process steps were tested

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[1] 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.

[2] Barnes S, R Roosa, and R Peterson. 2002. *Research and Technology Plan*. 24590-WTP-PL-RT-01-002 Rev. 1, RPP-WTP project.

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 S1. The Cs λ value (column distribution ratio) represents a measure of the effective capacity of the SL-644 resin; the higher the λ value, the higher loading capacity. Results from the SL-644 batch-contact tests, resulted in an estimated batch distribution (K_d) value of 690 mL/g in the feed condition (Na/Cs mole ratio of 1.4 E+5). Using a bed density of 0.230 g/mL in the 0.25 M NaOH condition, the Cs λ value is predicted to be 160 BVs. The IE-911 feed condition K_d value was interpolated to be 710 mL/g. With a bed density of 1.04 g/mL, the estimated λ value is 740 BVs.

Table S1. Summary of Performance Measures

Flow rate (BV/h)	**Extrapolated 50% Cs breakthrough, BV**		**Composite DF(1)**	**Maximum DF(2)**	**K_d, mL/g (feed condition)**		**Predicted Cs λ, BV**	
	Lead column	**Lag column**			**SL-644**	**IE-911**	**SL-644**	**IE-911**
3	190	–	7.78E+5	1.4E+6	690	710	160	740

(1) The decontamination factor was calculated by dividing the feed Cs concentration by the composite effluent Cs concentration.
(2) The maximum DF was obtained by dividing the feed Cs concentration by the lowest lag column sample Cs concentration.

The ion exchange column processing performance data are also summarized in Table S1. The maximum ion exchange Cs breakthrough obtained was 0.27% at 112 BVs (regeneration condition). Based on the AP-101DF column load data, the lead column 50% Cs breakthrough was extrapolated to be 190 BVs (resin BV in the 0.25 M NaOH regeneration condition). A 50% Cs breakthrough value for the lag column could not be estimated; virtually no measurable Cs broke through the lag column. The decontamination factor (DF) for ^{137}Cs, listed in Table S1, was based on ^{137}Cs concentration in the feed divided by the ^{137}Cs concentration in the composite effluent. The DF for ^{137}Cs was 7.78 E+5, and is equivalent to an effluent with a ^{137}Cs concentration of 1.1E-4 μCi/mL. The maximum DF, 1.4 E+6, measured the best performance that could be expected from this column system.

The LAW vitrified waste form must be no greater than 0.3 Ci ^{137}Cs/m^3. This limit can be converted to ^{137}Cs concentration in the Cs ion exchange effluent of 0.125 μCi ^{137}Cs/mL.[3] The product effluent ^{137}Cs concentration was a factor of 1000 below this contract limit.

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[3] The conversion requires the following assumptions: Envelope A LAW will contain 14 wt% Na_2O, all Na comes from the tank waste, the glass density is 2.66 g/mL, and the waste Na concentration is 5 M Na. For the maximum 20 wt% waste Na_2O loading, the Cs ion exchange effluent must be less than 0.087 μCi ^{137}Cs/mL.

The lead column was eluted with 0.5 M HNO_3 to C/C_o of 1% in 12 BVs with >94% of the ^{137}Cs contained in 2.5 BVs of eluant. The peak ^{137}Cs C/C_o value was 64 (based on 1-BV collection increments of nominally 10-mL).

Quality Requirements

This work was designated as QL-3 per the RPP-WTP Quality Assurance Program, BNFL-5193-QAP-01, Rev. 6. PNWD implemented the RPP-WTP quality requirements by performing work in accordance with the quality assurance plan, CHG-QAPjP, Rev. 0.

Issue

U and Pu were found in the Cs eluate, indicating these materials exchanged onto the resin. No additional issues were observed.

Terms and Abbreviations

AV	apparatus volume
BV	bed volume
C/C_o	analyte concentration in column effluent divided by analyte concentration in feed
CMC	Chemical Measurement Center
DI	deionized
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
HLRF	High Level Radiation Facility
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
λ	column distribution ratio
L/D	length over diameter ratio
M	molarity, moles/liter
MDL	method detection limit
MRQ	minimum reportable quantity
NMRQ	no minimum reportable quantity
NPT	National Pipe Thread
PSD	particle size distribution
SAL	Shielded Analytical Laboratory
TIC	total inorganic carbon
TOC	total organic carbon

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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 pretreatment flow sheet for the Hanford high-level tank wastes includes the use of SuperLig® 644 (SL-644) material for ^{137}Cs removal from the aqueous waste fraction. The SL-644 is a Cs-selective, organic, 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, McCabe, King and Crowder, 2000; Hassan, King, McCabe, and Crowder 2001; King, Hassan, and McCabe 2001; Kurath, Blanchard, and Bontha 2000a; Kurath, Blanchard, and Bontha 2000b). The SL-644 has been developed and supplied by IBC Advanced Technologies, Inc., American Fork, UT.

This report summarizes batch-contact studies of SL-644 and crystalline silicotitanate[4] (IE-911, UOP LLC, Des Plaines, IL), and dual small-column testing of the SL-644 ion exchange material. The test matrix was 241-AP-101 Hanford tank waste diluted to 5 M Na^+ (hereafter referred to as AP-101DF) (Goheen, et al., 2001). Approximately 1.2 L of AP-101DF 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 batch distribution coefficients as a function of Na/Cs molar concentration for SL-644 and IE-911 in the AP-101DF matrix.
- demonstrate the ^{137}Cs decontamination of Envelope A tank waste sample AP-101DF using SL-644 columns and provide a Cs-decontaminated sample for downstream process testing (i.e., ^{99}Tc removal, low activity waste (LAW) melter feed testing, and LAW vitrification).
- develop loading and elution breakthrough profiles for SL-644 columns.
- produce and characterize the Cs eluate solution from SL-644 columns 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.[5]

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[4] 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.

[5] 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 and Test Plan "Actual Waste Ion Exchange Testing for the RPP-WTP Project," CHG-TP-41500-013, Rev. 0, D. L. Blanchard, Jan. 24, 2000.

2.0 Experimental

2.1 SL-644 Resin

Initial AP-101DF batch contacts were conducted with SL-644 resin batch 981112YK-N3-16/18. This material batch had a distinct salt and pepper appearance with a significant light gray fraction. Initial batch distribution studies with this SL-644 batch on AW-101 simulant indicated it performed well and it was used for AP-101DF batch distribution testing. Unfortunately, later simulant column testing proved this material inadequate (Fiskum, Blanchard, and Arm 2002). Additional SL-644 was obtained from IBC production batch number 010319SMC-IV-73. This material batch exhibited a black-red appearance peppered with light brown specs.

Before sampling SL-644 from the IBC-supplied plastic bottle, the bulk resin bottle was turned several times to produce a well-mixed material. Sub-samples were removed immediately after mixing. The sieve results of the two resin batches were previously reported (Fiskum, Blanchard, and Arm 2002) and are reproduced in Table 2.1. The 212- to 425-µm particle size dry-sieved fraction was used in the ion exchange columns.[6] The average particle size corresponded to 540-µm in diameter, expanded in 3 M NaOH-2 M $NaNO_3$-0.1 M KNO_3 (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 (Korkisch). Given the diameter of the column at 1.46 cm, the column diameter was 27 times the average diameter of the 212- to 425-µm diameter dry-sieved resin particles expanded in caustic solution.

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

Sieve Size$^{(1)}$	Particle Size (µm)	981112YK-N3-16/18 wt %	010319SMC-IV-73 wt %
18	**>1000**	1.78	0.06
30	**600-1000**	55.25	37.27
40	**425-600**	20.12	38.23
50	**300-425**	13.76	18.01
70	**212-300**	7.93	6.08
100	**150-212**	1.11	0.26
140	**106 -150**	0.02	0.06
>140	**<106**	0.03	0.03
(1) U. S. standard sieve size corresponds to ASTM E-11 specification.			

Properties of the 981112YK-N3-16/18 and 212- to 425-µm 010319SMC-IV-73 SL-644 resins have been previously reported (Fiskum, Blanchard, 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

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[6] This particle size distribution was used successfully in AW-101 simulant testing.

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 (hydrogen form or acid form) and the as-received form of the resin. The F-factor for the Na-form resin was performed differently because of stability problems observed in prior tests on this resin form (Steimke et al. 2001). Drying to constant mass under vacuum at ambient temperature was considered adequate for removing water from the Na-form resin.[7] The L-factor represents the fractional mass remaining after washing the as-received resin form with 0.5 M HNO_3 and DI water, 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	**981112YK-N3-16/18**	**010319SMC-IV-73**
Sieve fraction	As-received	212- to 425-μm
Bulk density, g/mL(1)	0.80	0.74
F-factor, as-received	0.891	0.8773
L-factor, conversion to H-form, fractional mass remaining	0.493	0.538
F-factor, H-form	0.939	0.932
I_{Na}, fractional mass gain from H-form to Na-form	1.29	1.25

(1) Bulk density based on resin as-supplied by IBC, no drying, washing, or conditioning.

2.2 Crystalline Silicotitanate

Crystalline silicotitanate IE-911, Lot 2081000009, was obtained from UOP LLC, Des Plaines, IL. The UOP particle size specification of this material was 30-60 mesh. The material was provided in the hydrogen form. A 5-g aliquot was converted to the sodium form by soaking in 40 mL 1 M NaOH for two days. The NaOH solution was then decanted and the IE-911 was rinsed with five successive volumes of DI water. The resin was then dried at 95°C overnight. The F-factor, 0.9630 was determined at the same time the batch contact samples were weighed. This was obtained by drying approximately 0.5 g at 95°C to constant mass.

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[7] 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.

2.3 AP-101 Diluted Feed

Samples of tank 241-AP-101 (Envelope A) waste were received in the High Level Radiation Facility (HLRF) in 2000. The homogenization, dilution, subsampling, and analysis of this material have been previously described (Goheen et al. 2002). The total volume of AP-101DF available for Cs ion exchange processing was about 1.2 L. The feed composition is summarized in Table 2.3.

Table 2.3. Composition of AP-101DF (Envelope A)

Cations, M	
Na^+	4.97 E+0
K^+	7.61 E-1
Cs^+	3.68 E-5 (1)
Uranyl	1.7 E-4
Mole Ratios	
Na/Cs mole ratio	1.4 E+5
K/Cs mole ratio	2.1 E+4
Anions, M	
AlO_2^- (2)	2.59 E-1
Cl^-	4.1 E-2
F^-	< 1.2 E-1(3)
CO_3^{2-}	4.46 E-1
CrO_4^{-2} (2)	2.92 E-3
NO_2^-	7.1 E-1
NO_3^-	1.68 E+0
OH^-	1.94 E+0
PO_4^{-3} (2)	1.2 E-2
SO_4^{-2}	4.8 E-2
Oxalate	< 1 E-2
TOC, g/L	1.64 g/L
Radionuclides, (μCi/mL)	
Total alpha	2.6 E-04
^{60}Co	2.5 E-03
^{90}Sr	7.1 E-02
^{99}Tc	3.93 E-2(4)
^{99}Tc as pertechnetate	3.46 E-2
^{137}Cs	1.26 E+2
^{154}Eu	< 7 E-4
$^{239+240}Pu$	1.10E-4
$^{243+244}Cm$	2.12E-6
Solution density, g/mL	1.256

(1) Total Cs reported by Goheen et al. 2002 may be biased low. Reported value does not agree with ^{137}Cs determination by GEA and application of isotopic distribution (^{133}Cs 60.6 wt%, ^{135}Cs 15.4 wt%, ^{137}Cs 24.0 wt%) where a total Cs concentration of 6.04 μg/mL or 4.5 E-5 M is calculated.
(2) Al, Cr, and P determined by ICP-AES. The anionic form is assumed on the basis of waste chemistry.
(3) F concentration is an upper bound; co-eluting anions positively interfered with peak integration.
(4) Uncertainty, including standards bias, ranged from -10% to +30%

2.4 Batch Contacts

The batch contacts were performed with the H-form of SL-644 batch 981112YK-N3-16/18. The Na-form of IE-911 was tested in parallel with the SL-644. Initial batch contacts were performed using AP-101DF at three different Cs concentrations. Aliquots of the AP-101DF subsamples were tested without spiking, additional aliquots were spiked with 0.1 M $CsNO_3$ to obtain stock solutions of nominally 1E-3 M and 5 E-3 M Cs. The initial Cs concentrations in the AP-101DF stock contact solutions and the corresponding Na/Cs and K/Cs mole ratios are given in Table 2.4. After discerning the SL-644 batch 981112YK-N3-16/18 was insufficient to remove Cs under column loading conditions (Fiskum, Blanchard, and Arm 2002), another batch contact with AP-101DF was performed using the H-form of SL-644 batch 010319SMC-IV-73, 212- to 425-μm dry particle size. The scope of this second test was much reduced in order to conserve AP-101DF feed for use in subsequent column ion exchange and vitrification tests. Thus only the mid-range Cs concentration was tested in duplicate.

Table 2.4. Initial Cs Concentrations in the AP-101DF Solutions Used for the Batch Distribution Tests

Solution	Target Initial Cs Conc. [M]	Target Nominal Na/Cs$^{(1)}$mole ratio	Target Nominal K/Cs$^{(1)}$ mole ratio
SL-644 Batch 981112YK-N3-16/18 and IE-911			
Un-spiked	3.68E-5	1.4 E+5	2.1 E+4
Cs Spike 1	1.0 E-3	5.0 E+3	7.6 E+2
Cs Spike 2	5.0 E-3	1.0 E+3	1.5 E+2
SL-644 Batch 010319SMC-IV-73			
Cs Spike 3	1.0 E-3	5.0 E+3	7.6 E+2
(1) Na^+ and K^+ are the primary cations that compete with Cs^+ for ion exchange with SL-644 and IE-911.			

The batch-contact tests were performed in duplicate at a phase ratio of approximately 100 mL/g (liquid volume to exchanger mass). Typically, 0.1 g of SL-644 exchanger was contacted with 10 mL of AP-101DF, and 0.05 g of IE-911 and SL-644 for Cs Spike 3 were contacted with 5 mL AP-101DF. The exchanger mass was determined to an accuracy of 0.0002 g. The waste volume was transferred by pipet and the actual volume was determined by mass difference with an accuracy of 0.0002 g and the solution density. Samples were agitated in an orbital shaker for approximately 24 hours for SL-644 and 72 hours for IE-911. The temperature was not controlled but was generally constant at 26 ± 1°C during the 3 days of contact.

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

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

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

where C_0 = initial ^{137}Cs concentration
C_1 = final ^{137}Cs concentration
V = volume of the liquid sample (mL)
M = 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 = dried resin mass divided by the initial resin mass.

The dry bed resin density, ρ, was obtained according to the following equation:

$$\rho = \frac{m * L * F * I_{Na}}{BV} \qquad \textbf{(2)}$$

where ρ = dry Na-form g of resin per mL of resin
m = resin mass in the 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 in the feed (discussed in section 3.2.7).

The Cs λ value (column distribution ratio) is a function of the feed condition batch distribution coefficient and the dry bed density, and is obtained as shown in Equation 3.

$$\lambda = K_d * \rho \qquad \textbf{(3)}$$

2.5 Column Run Experimental Conditions

2.5.1 Ion Exchange Column System

A schematic of the ion exchange column system is shown in Figure 2.1. The system consisted of two small columns containing the 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 for obtaining 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 Savannah River Technology Center 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 each 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. A decal millimeter scale affixed to the column allowed for measurement of resin bed height and thus shrinkage and swelling. The upper section contained four entry ports and a taper joint with screw cap that securely fitted the column. The lead column assembly used a pressure relief valve (10 psi trigger), pressure gauge, and sample inlet; the remaining port was plugged. The lag column assembly used one port for sample entry, and the other three ports were plugged. In both columns, the inlet sample lines extended through the port opening to the top of the column. The connecting tubing was 1/8-in OD, 1/16-in ID polyethylene. Valved quick-disconnects (Cole Parmer, Vernon Hills, IL) were installed in-line to allow for ease of column switching. A FMI QVG50 pump (Fluid Metering, Inc., Syosset, New York) equipped with a ceramic and Kynar® coated low-flow piston pump head was used for all fluid introduction. The flow rate 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.

2.5.2 Resin Bed Preparation

The initial SL-644 resin and bed preparations were performed using a procedure similar to that recommended by Hassan et al. (1999). Before packing the resin beds, aliquots of SL-644 resin were placed in separate beakers. The two resin aliquots (4.17 and 4.20 g lead and lag columns, respectively) were soaked in about 40 mL of 1 M NaOH (~10:1 solution to resin volume ratio) with light agitation for 2 h. The NaOH was decanted, and the SL-644 was slurried with about 40 mL of DI water. The resin was then quantitatively transferred into the columns using additional DI water to aid the transfer. Both of the resin beds were individually washed with 0.5 M HNO_3. After resin washing, the corrected sodium form SL-644 mass was calculated to be 2.46 g and 2.48 g (dried at ambient temperature under vacuum) in the lead and lag columns, respectively, according to the following equation:

$$M_{bed} = M_{AR} * F_{AR} * L * I_{Na} \quad \textbf{(4)}$$

where M_{bed} = resin bed mass
M_{AR} = resin mass loaded in the column, as-received form, 4.17 g lead and 4.20 g lag columns
F_{AR} = F-factor for the as-received resin, 0.877
L = fractional mass remaining after conversion to the H-form, 0.538
I_{Na} = fractional mass gain on conversion from the H-form to the Na-form, 1.25.

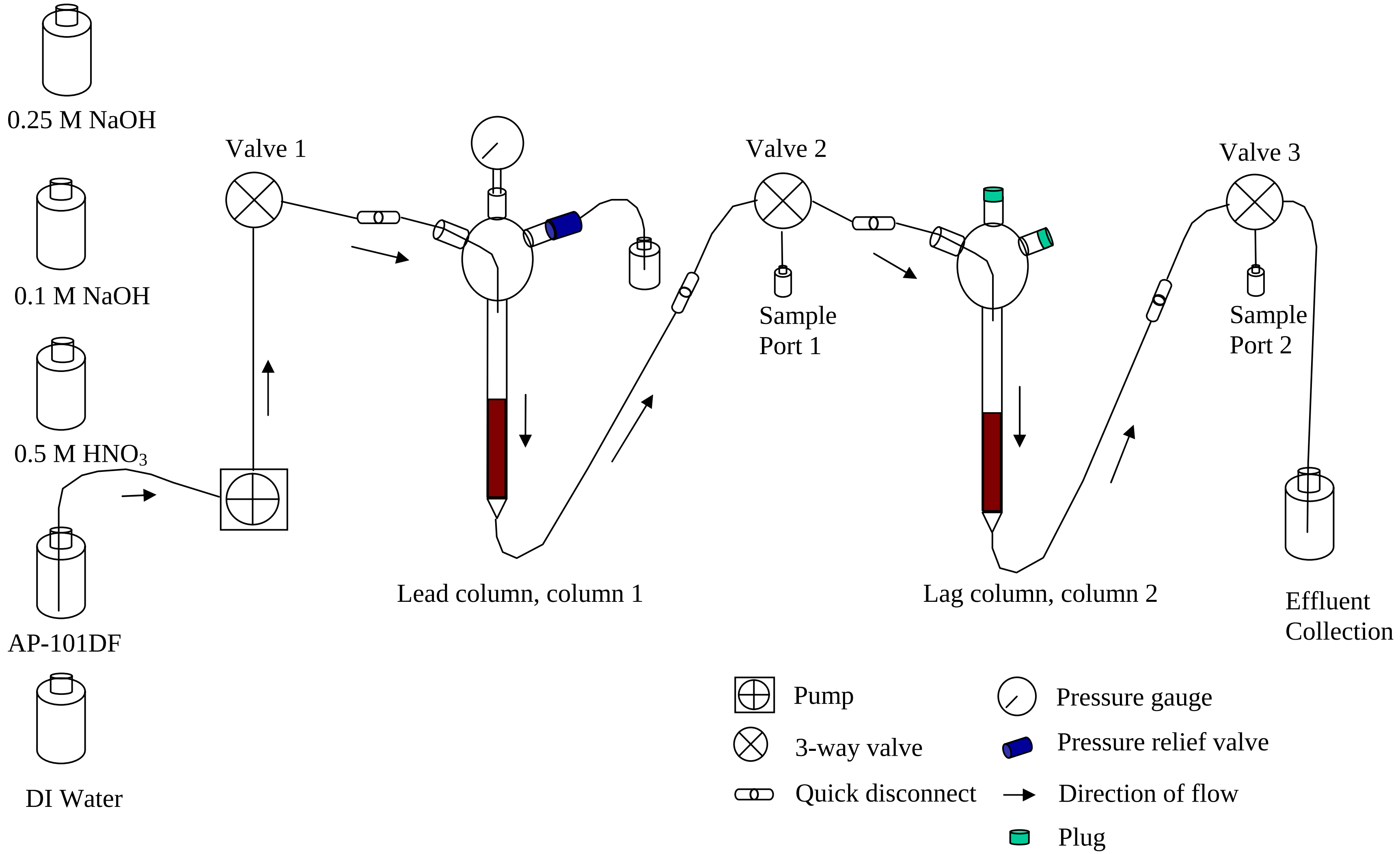

Figure 2.1. Cesium Ion Exchange Column System

The entire ion exchange system was rinsed with DI water, regenerated with 0.25 M NaOH, and then used for a full shakedown experiment with AW-101 simulant (Fiskum, Blanchard, and Arm 2002). Both columns were individually eluted and rinsed. A summary of the shakedown test operations is provided in Table 2.5.

Table 2.5. Bed Conditioning and Simulant Shakedown Testing Parameters

Process Step	Solution	Average Volume			Flow Rate, BV/h
		BV[(1)]	AV[(2)]	mL	
Pre-Conditioning, Lead and Lag Columns Separate					
Wash	0.5 M HNO_3	13.5		150	0.87
DI water rinse	DI water		3.0	67	2.1
Regeneration	0.25 M NaOH	5.1		56	2.0
Loading, Feed Displacement, and Rinse, Columns in Series					
Regeneration (cont.)	0.25 M NaOH	3.7		41	1.9
Load	AW-101 simulant	143		1576	2.6
Feed Displacement	0.1 M NaOH		2.9	121	2.5
Rinse	DI water		2.0	84	2.5
Elution, Lead and Lag Columns Separate					
Elution	0.5 M HNO_3	12		132	0.92
Eluant rinse	DI water		2.7	60	2.6

(1) BV = bed volume (11 mL in 0.25 M NaOH regeneration condition)
(2) AV = apparatus volume (42 mL for columns in series; 23 mL for lead column processing and 21 mL for lag column processing)

2.5.3 Ion Exchange Column Test with AP-101DF

The beds had been stored approximately 3 weeks in the H-form in DI water since the end of the simulant AW-101 column run. The resin beds were fluidized with DI water. Then the ion exchange system was loaded into the Shielded Analytical Laboratory (SAL) hot cell with the resin in the H-form. All subsequent processing was performed in the hot cells at temperatures ranging from 25 to 29°C. The entire system was initially cycled in the hot cell with 0.25 M NaOH, DI water, 0.5 M HNO_3, and DI water sequentially through the lead and lag columns. The bed conditioning, AP-101DF loading,[8] feed displacement, and DI water rinse steps were conducted by passing these solutions through both resin beds connected in series. The elution, elution rinse, and the regeneration steps were conducted on the lead column only. The experimental conditions for each process step are shown in Table 2.6. The resin bed volume (BV) is the volume in 0.25 M NaOH. For the AP-101DF test, the BV was measured at 10.6 mL in the regeneration condition just prior to waste loading. The AP-101DF effluent was collected in four effluent bottles. The first bottle collected 64 mL, slightly over one apparatus volume (AV), and consisted primarily of the displaced regeneration solution. The following effluent collections captured nominally

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[8] AP-101DF processing represents the second Cs load and elute cycle for the resin beds.

350-mL AP-101DF fractions. After ascertaining ^{137}Cs activity was low, the three 350-mL fractions were combined into one composite for follow-on Tc ion exchange.

The solution above the lead resin bed was drained to within about 1 cm of the resin surface when switching to the next process step. Then nominally 5-mL of the solution were allowed to build up above the resin bed prior to commencement of flow. This helped minimize reagent mixing thus speeding the reagent transition through the column system.

Table 2.6. Experimental Conditions for AP-101DF Ion Exchange

Process step	Solution	Total Volume			Flow rate		Time, h	T, °C
		BV$^{(1)}$	AV$^{(2)}$	mL	BV/h	mL/min		
Bed conditioning	0.25 M NaOH	9.8	2.5	104	1.9	0.33	5.2	25
Bed conditioning	DI water	5.7	1.4	60.6	2.7	0.48	2.1	25
Bed conditioning	0.5 M HNO_3	7.8	2	82.2	2.5	0.44	3.1	25
Bed conditioning	DI water	6.9	1.8	74	2.8	0.49	2.5	25
Regeneration	0.25 M NaOH	9.6	2.4	103	1	0.18	9.6	25
Loading lead column	AP-101 Feed	112.2	-	1189	2.8	0.50	37.8	26-28
Loading lag column$^{(3)}$	AP-101 Feed	109.4	-	1159	2.8	0.50	37.8	26-28
Feed displacement	0.1 M NaOH	13.2	3.3	140	2.74	0.48	5.1	28
DI water rinse	DI water	8.7	2.2	92	2.9	0.51	3.2	28
Elution of lead column								
Elution	0.5 M HNO_3	19.3	-	204	0.88	0.16	24.6	28-29
Eluant rinse	DI water	6.9	3.19	73.7	2.9	0.51	4.5	28-29
Regeneration	0.25 M NaOH	4.7	2.17	50	0.9 - 1.5	0.16 - 0.27	6.2	23
Rinse	DI water	5	2.31	53.1	2.59	0.46	1.9	24

(1) BV in 0.25 M NaOH regeneration condition (10.6 mL),
(2) AV (42 mL for sequential column format, 23 mL for lead column only)
(3) The feed volume through the lag column is reduced because of sampling from the lead column.

The sampling and analysis protocol is shown in Table 2.7. During the loading phase, small samples (about 2 mL) were collected from the lead column starting at 7 BVs, then at 15 BVs and continuing in nominal 10-BV increments of feed from both the lead and lag columns. The flow rate ranged from 2.99 to 3.23 BV/h in the feed condition, which was equivalent to 2.74 to 2.96 BV/h when the resin bed was expanded in the regeneration solution. Flow rate doubled during sample collection from the lead column because of the strong siphoning effect at the sample port. Thus, for the 13 samples collected from the lead column, and for 2 minutes each sample, AP-101DF fed at a nominal flow rate of 6 BV/h. The feed displacement, DI water rinse, elution, and elution rinse samples were taken at 1-BV increments at flow rates shown in Table 2.6.

Table 2.7. Sampling Interval and Analyses

Process Step	Lead Column	Lag Column	Approximate Sample Size (mL)	Analyses
Bed conditioning	–	–	–	–
Bed conditioning	–	–	–	–
Bed conditioning	–	–	–	–
Loading	Every 10 BVs	Every 10 BVs	2	GEA
Feed displacement	–	Every 1 BVs	10	GEA
DI water rinse	–	Every 1 BVs	10	GEA
Elution	Every 1 BVs	–	10	GEA
Eluant rinse	Every 1 BVs	–	10	GEA
Regeneration	1 composite	–	50	ICP-AES, GEA, OH-
Composite Samples				
Effluent – 1	–	–	2	GEA
Effluent – 2	–	–	2	GEA
Effluent – 3	–	–	2	GEA
Effluent – 4	–	–	2	GEA
Effluent composite (effluents 2 - 4)	–	–	10	GEA, ICP-AES, IC, U, ICP-MS (Tc), pertechnetate
Eluate	1 composite	-	10	ICP-AES, GEA, TOC, IC, U, ICP-MS, and various radioisotopes

2.5.4 Sample Analysis

The ^{137}Cs concentration was determined using a bench-top GEA spectrometer. This allowed for rapid sample analysis. Selected sample results were later confirmed by GEA analysis in the analytical laboratory. Because of the high dose rate from ^{137}Cs, the feed sample and eluate samples required dilution before removal from the hot cell. These samples were diluted with 0.25 M NaOH and 0.5 M HNO_3, respectively. The extent of dilution was determined by mass difference. Composite samples from each of the effluent bottles were taken for analysis by GEA. Another composite sample was taken for analysis once the contents of effluent bottles 2 through 4 were combined for Tc ion exchange. The regeneration solution was composited and a sample taken for ICP-AES and OH^- determination. Once the GEA results were confirmed for the eluate samples, the eluate samples were composited and a sample of the composite was submitted for various analyses; GEA, ICP-AES, total organic carbon (TOC), ion chromatography (IC), U, pertechnetate, and ICP-MS for ^{99}Tc.

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 IC. TOC and total inorganic carbon (TIC) were determined by silver-catalyzed hot persulfate oxidation and furnace oxidation methods.

3.0 Results and Discussion

3.1 Batch Contact Results

The K_d values were calculated using Equation 1 and are based on the ^{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 AP-101DF. The equilibrium Cs concentration was based on the measured ^{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 is a reasonable assumption. In these experiments the waste solutions were estimated to have 19.4 meq of OH^- and 49.7 meq of Na^+, while the resin contained 0.22 meq of H^+ for the 0.1 g resin used in the batch distribution tests.[9]

The calculated ^{137}Cs K_d values are plotted in Figure 3.1 for SL-644 and IE-911 in contact with AP-101DF. The IE-911 feed condition K_d value is interpolated at 710 mL/g. The SL-644, 981112YK-N3-16/18 resin batch K_d values are defined by the following equation (linear least squares fit): $K_d = 142.44 * \text{Ln}([\text{Na}]/[\text{Cs}]) - 997.49$, $R^2 = 0.998$.

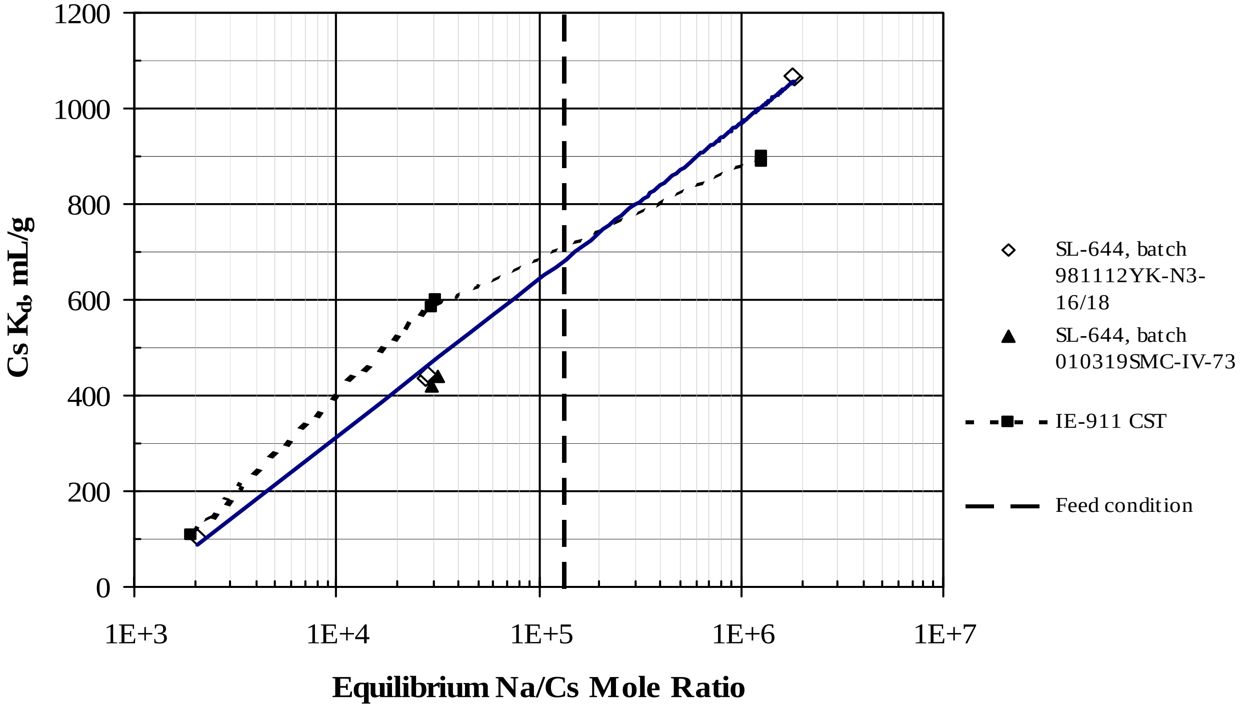

Figure 3.1. ^{137}Cs Distribution Coefficients (K_d) for AP-101DF (T = 26°C)

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[9] The H-form resin contains 2.2 meq H^+ per gram. (Rapko, et al. 2002)

The experimental data from the single Cs concentration tested (Cs spike 3) shows the K_d value, represented by the 010319SMC-IV-73 SL-644 resin batch (212- to 425-µm particle size), is equivalent to the 981112YK-N3-16/18 material. However the slope could not be evaluated. Assuming equivalent slope, the K_d value at the AP-101DF feed condition can be estimated at 690 mL/g from the point at which the K_d curve crosses the Na/Cs mole ratio (1.4 E+5) for the AP-101DF sample.

The calculated dry-bed densities determined using the resin masses and volumes in the ion exchange column system are given in Table 3.1. Good agreement was obtained between the lead and lag columns. The 0.25 M NaOH dry-bed density was 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 equivalent to the decrease in volume upon contracting from the Na-form to the H-form.

Table 3.1. Dry Bed Density

Property	**010319SMC-IV-73, Lead Column**	**010319SMC-IV-73, Lag Column**
Column resin mass,(1) g	4.17	4.20
Corrected column Na-form resin mass, g	2.46	2.48
Bed volume, 0.25 M NaOH, mL	10.7	10.5
Bed volume, AP-101DF, mL	9.7	9.7
Bed volume, 0.5 M HNO_3, mL	7.7	not performed
Dry bed density, ρ, in given matrix		
Na-form resin		
0.25 M NaOH, g/mL	0.230	0.236
AP-101DF, g/mL	0.254	0.255
H-form resin		
0.5 M HNO_3, g/mL	0.256	not performed
(1) The 212- to 425-µm particle-size resin mass measured in the as-received form.		

The calculated Cs λ values, as a function of Na/Cs mole ratio, are shown in Figure 3.2. Two Cs λ curves are plotted for SL-644. One curve represents the measured K_d values from SL-644 batch 981112YK-N3-16/18 multiplied by the calculated resin bed density of 0.15 g/mL (Fiskum, Blanchard, and Arm 2002). The other SL-644 Cs λ curve is estimated for the 010319SMC-IV-73 resin, applying the resin bed density of 0.23 g/mL to the K_d values generated for the 981112YK-N3-16/18 resin. The Cs λ value is estimated to be 170 BVs in the AP-101DF feed condition, and 160 BVs in the 0.25 M NaOH regeneration condition for 010319SMC-IV-73 SL-644. This value is the approximate point at which the Cs breakthrough curve was predicted to pass through C/C_o = 50% if sufficient feed was available for processing.

The calculated Cs λ values for IE-911 are also shown in Figure 3.2. The IE-911 bed density was determined to be 1.04 g/mL. The feed condition Cs λ value for IE-911 is estimated to be 740 BVs.

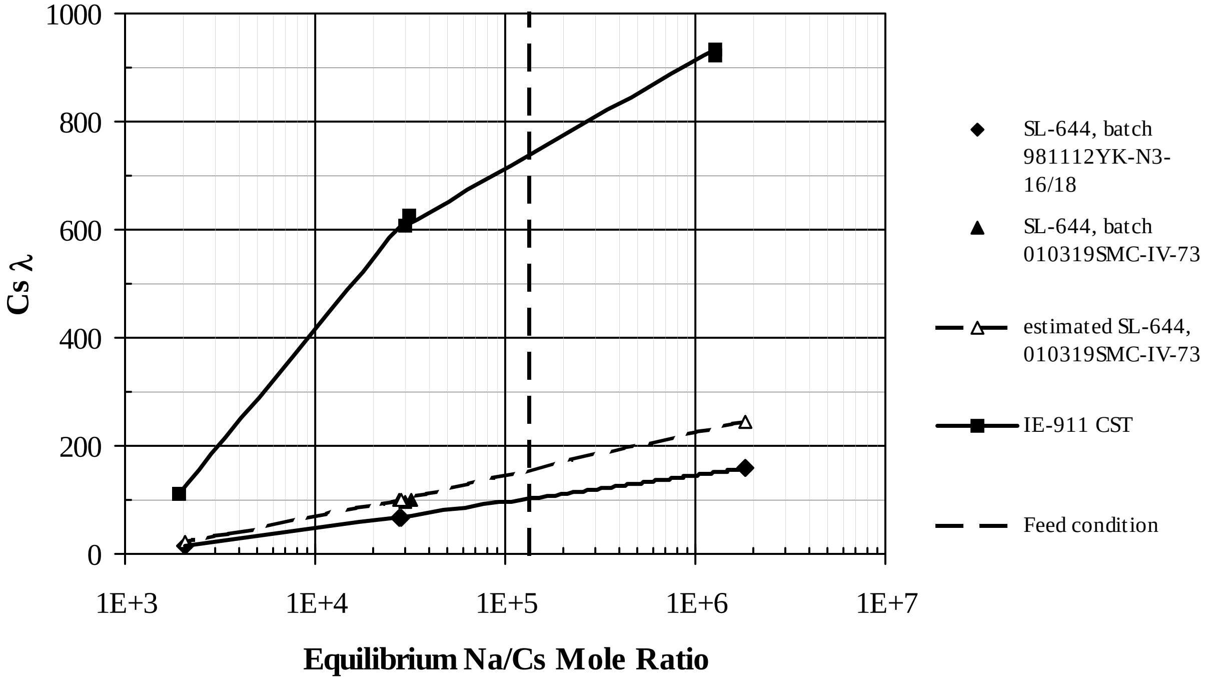

Figure 3.2. Estimated ^{137}Cs λ Values for AP-101DF (T = 26°C)

3.2 Column Test

3.2.1 Initial Resin Conditioning

The resin bed conversion progress from Na-form to H-form and vice versa could be monitored visually according to the color of the resin bed. The Na-form appeared black in the columns whereas the H-form appeared brown. Initial conversion to the Na-form in the sequential column format required nominally 104 mL (9.8 BVs fully expanded) or 2.5 AVs. During this time the BVs increased from 7.5 mL to 11 mL. Conversion to the H-form required 82 mL of 0.5 M HNO_3 (7.8 BV fully expanded) or 2.0 AVs. During this time the resin bed shrank from 11 mL to 7.8 mL. The second regeneration was performed at half the flow rate (1 BV/h) of the first regeneration. The same volume of 0.25 M NaOH was required to complete the conversion, as evidenced by visual examination.

3.2.2 Loading

After the resin beds were conditioned, the loading phase was initiated with AP-101DF. Approximately 1.5 AVs (64 mL) of effluent were initially collected in a separate collection bottle. Most of this effluent was 0.25 M NaOH from the regeneration step mixed with some AP-101DF in the ion exchange apparatus. This solution was maintained separately and not mixed with the final Cs-decontaminated AP-101DF effluent composite; thus most of the apparatus 0.25 M NaOH solution was prevented from mixing with the AP-101DF effluent. The resin shrank about 8.5% to 9.7 mL in the AP-101DF feed.

The Cs effluent concentrations from the columns 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 10.6 mL. The C_o value for ^{137}Cs was determined to be 126 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 a bench-top GEA spectrometer, were generally in good agreement with selected samples independently analyzed by the analytical laboratory. Raw analytical results and calculations are located in the appendix.

The contract ^{137}Cs removal limit is also shown in Figure 3.3. The C/C_o value of 0.099% 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 is determined using the Na concentration of 5.0 M in the AP-101DF, a ^{137}Cs feed concentration of 126 µCi/mL, a 14 wt% waste Na_2O loading in the glass, and a glass product density of 2.66 g/mL.[10]

The 50% Cs breakthrough value is the point at which the C/C_o is 50% (0.5) and is normally a direct indicator of the effective capacity of the resin. A straight-line extrapolation to 50% breakthrough from 0.2% C/C_o is inherently prone to error. With this caveat, the 50% Cs breakthrough extrapolated to about 190 BVs (0.25 M NaOH condition) for the lead column. This value is in good agreement with the predicted Cs λ value of 160 BVs determined with batch-contact tests. There is insufficient data for an estimate of the 50% Cs breakthrough for the lag column.

The decontamination factors (DFs) were calculated on composites from each of the three effluent collection bottles and are summarized in Table 3.2. These may be compared to the contractual limit of C/C_o = 0.099% (DF= 1,010). In all cases, sufficient Cs was removed that the contract limit for Cs removal was met. The final lag column sample C/C_o and DF are also shown. The DF result is equivalent to the maximum DF within experimental error.

Table 3.2. Decontamination Factors for ^{137}Cs from AP-101DF

Sample	Volume, BV (mL)	^{137}Cs Concentration µCi/mL$^{(1)}$	C/C_o, %	DF
Final lag column sample	109 (1160)	7.4 E-05	7.3 E-05	1.4 E+06
Effluent composite bottle-2	34 (356)	9.8 E-05	7.7 E-05	1.3 E+06
Effluent composite bottle-3	36 (377)	1.1 E-04	8.6 E-05	1.2 E+06
Effluent composite bottle-4	40 (424)	2.3 E-04	1.8 E-04	5.5 E+05
Composite effluent	104 (1103)	1.08 E-04	8.6 E-05	1.2 E +06
(1) The ^{137}Cs uncertainty ranges from 3% to 8% relative error, 1-σ.				

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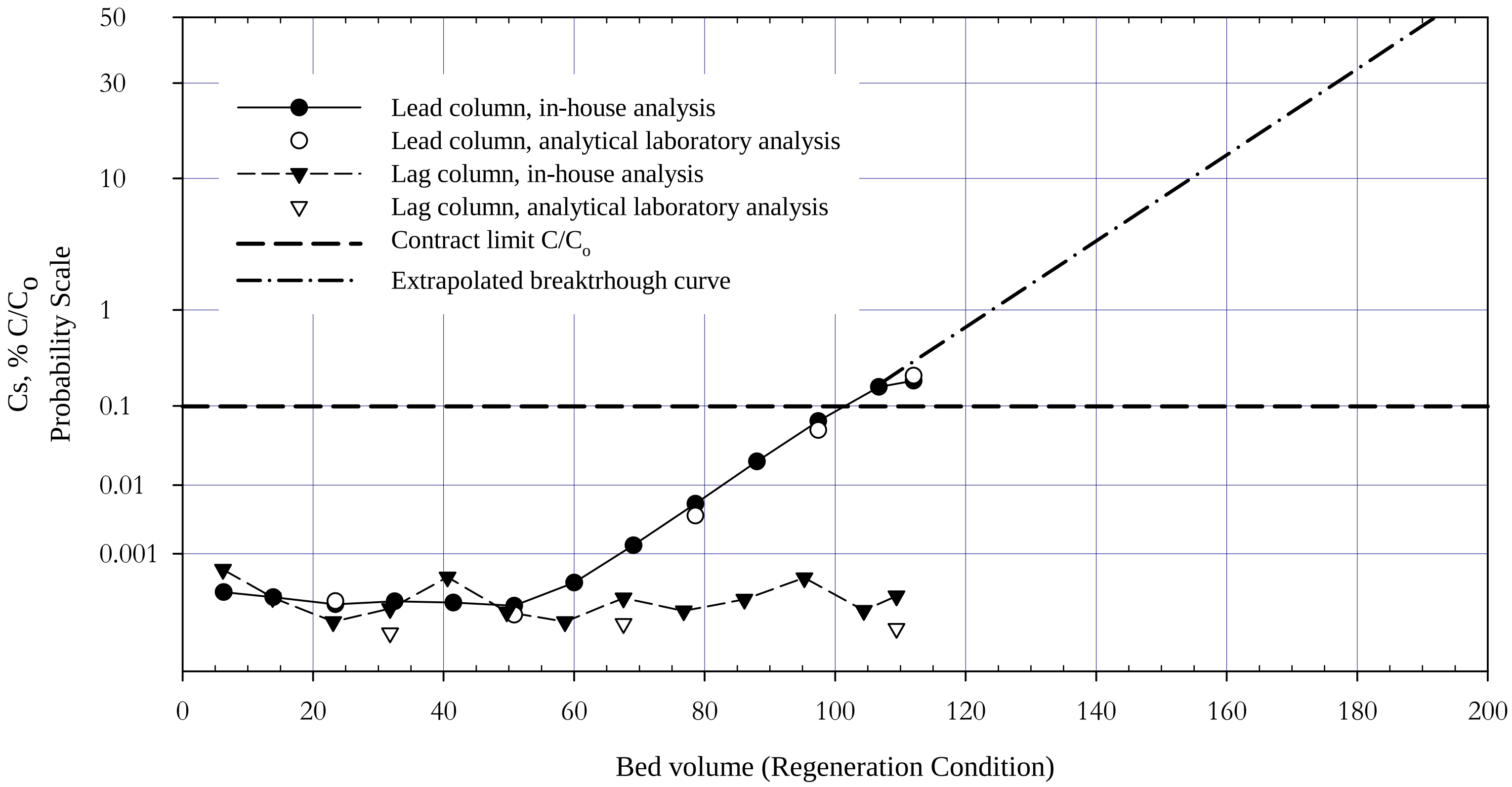
[10] Applying a glass loading of 20 wt% would reduce the contract limit % C/C_o to 0.069 %.



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425-μm dry particle size
Process temperature = 28 °C
BV in 0.25 M NaOH feed condition = 10.6 mL
Flow Rate = 2.8 BV/h
^{137}Cs C_o = 126 μCi/mL
Na concentration = 4.97 M.

Figure 3.3. ^{137}Cs Breakthrough Curves for AP-101DF Sample, Probability Plot

The three composite effluent fractions were combined into a single composite for subsequent processing. This Cs-decontaminated effluent was characterized and the results are summarized in Table 3.3. Major constituents (Na, K, Al, NO_3^-, and NO_2^-) in the effluent were similar in concentration to the feed within experimental error. The authors have no explanation for the 24% increase measured for OH other than experimental/analytical error. The pertechnetate remained constant within experimental error. The total Tc determined by ICP-MS appeared to be biased high in this analysis consistent with post-analysis standards bias (see ICP-MS report ASR 6121 in Appendix E). Notably, nearly all U and Pu[11] were removed from the AP-101DF by SL-644. The F, PO_4^{3-} and SO_4^{2-} also resulted in low effluent recovery. The low recovery is thought to be due to analytical error/uncertainty.

3.2.3 Feed Displacement and Rinse

There was a delay between the introduction of the feed displacement and the collection of these samples from the effluent line. Approximately 42 ml of Cs-decontaminated AP-101DF were collected as effluent prior to starting the feed displacement sampling. The relative Cs concentration, as C/C_o, was < 4 E-3 % for all feed displacement and DI water rinse samples.

3.2.4 Elution and Eluant Rinse

The elution proceeded similarly as found in the simulant test with respect to the Cs elution profile and resin bed shrinkage. However, the characteristic color change from black in the Na-form to tan in the H-form was not visible. The resin appeared virtually black for the entire elution and rinse cycles.

The lead column C/C_o[12] values for ^{137}Cs are shown in Figure 3.4 for the elution and the eluant rinse steps. 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. The majority of the ^{137}Cs was contained in elution BVs 3-5. The peak value of C/C_o was found to be 64. The elution cutoff of $C/C_o = 0.01$ was reached at 12 BVs but elution was continued beyond this due to the lag between sample collection and the determination of the Cs concentration. At 18.5 BVs the C/C_o visibly rises. At this point, the columns were allowed to sit in 0.5 M HNO_3 for about two hours while samples were counted. Cesium continued to elute from the resin and when flow was re-started, showed a slight rise in relative Cs concentration. The C/C_o values for the eluant rinse with DI water dropped rapidly in ^{137}Cs concentration indicating DI water did not continue Cs elution.

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[11] Pu concentration in the effluent was estimated from the Pu concentration in the Tc ion exchange effluent. This assumes that the Tc ion exchange processing did not remove Pu.

[12] The C_o refers to the ^{137}Cs concentration in the AP-101 DF 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.

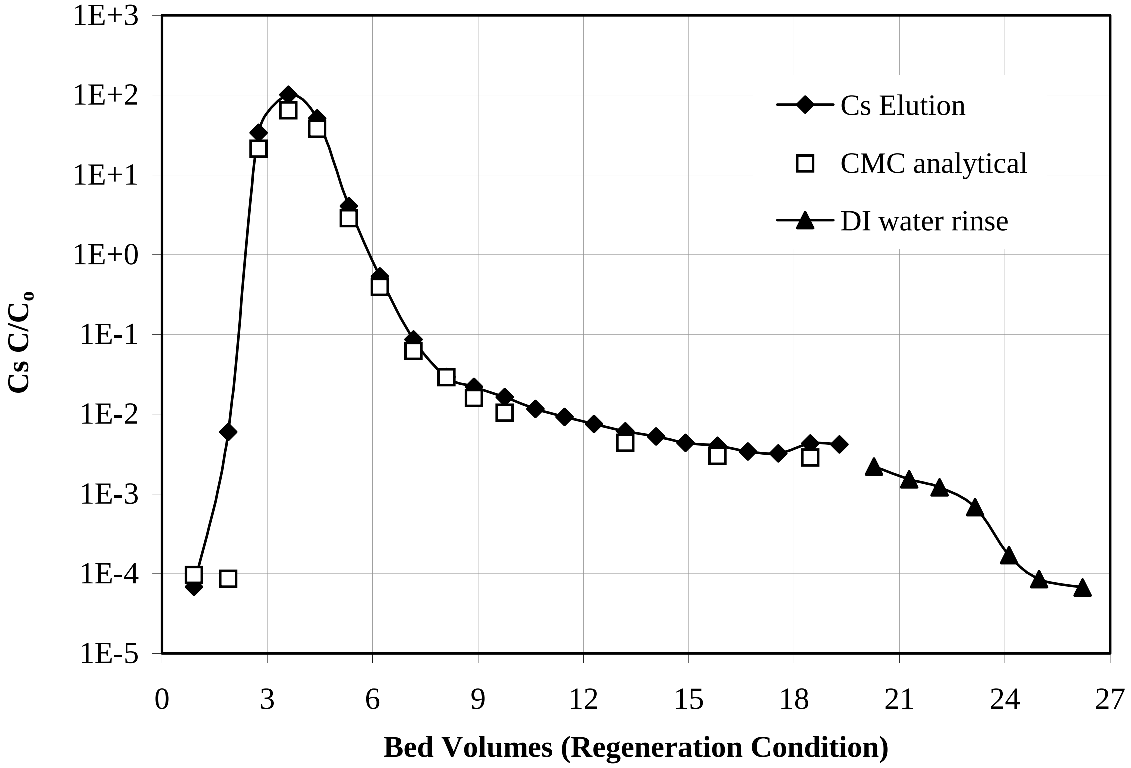
Table 3.3. AP-101DF Cs-Decontaminated Product Effluent

Component	Concentration, M	% Change from Feed
Cations		
Na^+	4.81 E+0	-3.0
K^+	7.44 E-1	-2.2
Uranyl	3.8 E-6	-98
Anions		
AlO_2^-	2.45 E-1	-5.3
F^-	9.74 E-2	-19.
Cl^-	4.09 E-2	-1.2
CrO_4^{-2}	2.70 E-3	-7.5
NO_2^-	7.78 E-1	10.
NO_3^-	1.88 E+0	12.
OH^-	2.40 E+0	24
PO_4^{-3} (IC)	7.3 E-3	-27.
P (ICP-AES)	1.06 E-2	-15.
SO_4^{-2}	3.25 E-2	-33.
Oxalate	1.1 E-2	-2.7
Radionuclides	**μCi/mL**	**% Change from Feed**
^{60}Co	2.23 E-3	-12.
^{99}Tc	4.99 E-02(2)	27.
^{99}Tc as pertechnetate	3.95 E-2	14.
^{137}Cs	1.08 E-4	>-99
^{154}Eu	< 3.0 E-5	NA
$^{239+240}$Pu	1.74E-5 (estimated) (1)	-84(1)
Solution density, g/mL	1.257	0.08

NA = not applicable, not detected in feed or effluent

(1) Pu removal estimated based on feed Pu concentration and effluent Pu concentration from Tc ion exchange. The $^{239+240}$Pu concentration in the Tc-removed AP-101DF effluent was 1.74E-5 μCi/mL. The Pu removal estimate is supported by the eluate analysis where 72% Pu was recovered.

(2) Uncertainty, including standard bias, ranged from -10% to +30%.



Conditions: SL-644 batch number 010319SMC-IV-73 212- to 425-µm dry particle size
Process temperature = 28-29 °C
BV in 0.25 M NaOH feed condition = 10.6 mL
Flow Rate = 0.88 BV/h
^{137}Cs C$_o$ = 126 µCi/mL
Eluant is 0.5 M HNO_3.

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

The eluate samples from the lead column were composited and a sub-sample taken for analysis. The remainder of the composite was saved for vitrification testing. The analytical results are shown in Tables 3.4 and 3.5. Sodium was the dominant component detected using ICP-AES with some Ca, Cd, Cr, Fe, K, Pb, U, and Pu eluting as well. The relative fractions of these analytes recovered in the eluate are given in the tables. The U recovered in the eluate (~42 mg) represents 87% of the U loaded in the AP-101DF feed (48 mg). Plutonium (based on $^{239+240}$Pu) is also largely recovered (72%) in the eluate.[13] A significant fraction (57%) of the Cm was also recovered in the eluate. The other analytes recovered to a much

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[13] The sum of ^{239}Pu and ^{240}Pu measured by ICP-MS (8.98E-4 µCi/mL) was a factor of two higher than the radiochemistry determination of the $^{239+240}$Pu (4.63E-4 µCi/mL). The ICP-MS determination was considered biased high.

smaller percentage in the eluate (Na: 0.14%; Ca: 2%; Cr: 1%; Fe: 29%; K: 0.03%; Pb: Sr: 7%). Because the feed concentrations were “less than” values, the Cd and Pb recovered at >10% and >8%, respectively. The B and Si were probably leached from the glass vials used during the sample preparation process; the preparative blank sample was nearly as high in B and Si concentrations as the eluate sample. Thus the reported B and Si concentrations are not indicative of the eluate composition. As expected ^{137}Cs was the dominant radionuclide detected. The only anion detected was nitrate, which is not surprising since the eluant was 0.5 M HNO_3.

Most of the specified minimum reportable quantity (MRQ) levels were met with some exceptions. The large amount of ^{137}Cs prevented the detection limits for ^{241}Am, ^{154}Eu, and ^{155}Eu from meeting the MRQ levels.   Relatively high ^{137}Cs activity increased the gamma background level in the detectors due to Compton scattering, thereby making it difficult to detect lower concentrations of other gamma emitters. The large amount of NO_3 prevented the detection limit for Cl from meeting the MRQ level of 3 μg/mL. The large NO_3 concentration required large sample dilutions and increased the method detection limit for the other components. The anion concentrations other than nitrate are expected to be small. The TIC analysis was not completed because carbonate is known to evolve as CO_2 in acidic solutions.

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

Analyte	**Method**	**MRQ μg/mL**	**Cs eluate μg/mL**	**% recovered**	**Analyte**	**Method**	**MRQ μg/mL**	**Cs eluate μg/mL**	**% recovered**
Ag	ICP-AES	NMRQ	< 0.63	ND	Rh	ICP-MS	NMRQ	5.7 E-2	NM
Al	ICP-AES	75	18.6	0.05	Ru	ICP-MS	NMRQ	3.2 E-3	NM
As	ICP-AES	NMRQ	< 6.3	ND	Sb	ICP-AES	NMRQ	< 13	ND
B	ICP-AES	NMRQ	194	HB	Se	ICP-AES	NMRQ	< 6.3	ND
Ba	ICP-AES	2.3	[0.29]	[>4](1)	Si	ICP-AES	170	145	HB
Be	ICP-AES	NMRQ	< 0.25	ND	Sn	ICP-AES	1500	< 38	ND
Bi	ICP-AES	NMRQ	< 2.5	ND	Sr	ICP-AES	NMRQ	< 0.38	ND
Ca	ICP-AES	150	[25]	[2.0]	Ta	ICP-MS	NMRQ	2.7E-3	NM
Cd	ICP-AES	8	[1.2]	[>10](1)	Te	ICP-AES	NMRQ	< 38	ND
Ce	ICP-AES	NMRQ	< 5.0	ND	Th	ICP-AES	NMRQ	< 25	ND
Co	ICP-AES	30	< 1.3	ND	Ti	ICP-AES	17	< 0.63	ND
Cr	ICP-AES	15	12	1.4	U	ICP-AES	600	[160]	[67]
Cs(2)	GEA	1.5	37	130(3)	U	KPA	600	206(4)	87
Cu	ICP-AES	17	[1.2]	[> 6] (1)	V	ICP-AES	NMRQ	< 1.3	ND
Dy	ICP-AES	NMRQ	< 1.3	ND	Y	ICP-AES	NMRQ	< 1.3	ND
Eu	ICP-AES	NMRQ	< 2.5	ND	Zn	ICP-AES	17	< 1.3	< 2.8
Fe	ICP-AES	150	[6.1]	[29]	Zr	ICP-AES	NMRQ	< 1.3	ND
K	ICP-AES	75	[57]	[0.03]	TOC	Hot Pers.	1500	< 30	< 0.3
La	ICP-AES	35	< 1.3	ND	TOC	Furnace	1500	180	0.4

Analyte	Method	MRQ μg/mL	Cs eluate μg/mL	% recovered	Analyte	Method	MRQ μg/mL	Cs eluate μg/mL	% recovered
Li	ICP-AES	NMRQ	< 0.75	ND	Br^-	IC	NMRQ	< 63	ND
Mg	ICP-AES	300	< 2.5	ND	Cl^-	IC	3	< 63	< 0.7
Mn	ICP-AES	150	< 1.3	ND	F^-	IC	150	< 63	< 0.5
Mo	ICP-AES	150	< 1.3	ND	NO_2^-	IC	3000	< 125	< 0.1
Na	ICP-AES	75	912	0.14	NO_3^-	IC	3000	29250	NA
Nd	ICP-AES	NMRQ	< 2.5	ND	PO_4^{-3}	IC	2500	< 125	ND
Ni	ICP-AES	30	[1.6]	[6]	SO_4^{-2}	IC	2300	< 125	< 0.5
P	ICP-AES	600	< 2.5	< 0.1	Oxalate	IC	NMRQ	< 125	ND
Pb	ICP-AES	300	[4.7]	[> 6] (1)	OH	Titration	17	NM	NM
Pd	ICP-MS	NMRQ	5.9 E-3	NM	Wt% dried solids		0.1	0.34%	NA
Pr	ICP-MS	NMRQ	5.2 E-4	NM	Wt% oxides		NMRQ	0.40%	NA
Pt	ICP-MS	NMRQ	3.0E-2	NM	Density, g/mL		NMRQ	1.011	NA

ASR 6192, sample ID 01-1842

Notes: MRQ is minimum reportable quantity requested by Bechtel. NMRQ is no minimum reportable quantity requested. 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%.

NA = not applicable
ND = analyte not detected in the feed or product
NM = analyte not measured in the eluate
HB = high preparative blank concentration, recovery cannot be calculated

(1) Analyte was not detected in the feed.

(2) Calculated based on ^{137}Cs concentration in the eluate and applied Cs isotopic distribution ratio (^{133}Cs 60.6 wt%, ^{135}Cs 15.4 wt%, ^{137}Cs 24.0 wt%).

(3) Calculated based on Cs feed concentration of 4.89 μg/mL. If the feed Cs concentration is actually 6.04 μg/mL, then Cs recovery is calculated to be 105%. See footnote (1) of Table 2.3.

(4) Total U measured by ICP-MS was in good agreement at 190 μg/mL.

Table 3.5. Radionuclides in the Lead Column Eluate Composite(1)

Analyte	Method	MRQ(2) µCi/mL	Cs eluate µCi/mL	Error %	% recovered	Analyte	Method	MRQ(2) µCi/mL	Cs eluate µCi/mL	Error %	% recovered
^{3}H	Radchem	NMRQ	1.72 E-4	10	NM	^{155}Eu	GEA	9.00 E-2	<3 E-1	–	ND
^{14}C	Radchem	NMRQ	2.4 E-5	30	NM	^{232}Th	GEA	NMRQ	<2 E-1	–	NM
^{51}Cr	GEA	NMRQ	< 2 E-2	–	NM	^{233}U	ICP-MS	NMRQ	1.78 E-4	1	NM
^{54}Mn	GEA	NMRQ	<3 E-2	–	NM	^{234}U	ICP-MS	NMRQ	9.89 E-5	4	NM
^{59}Fe	GEA	NMRQ	<3 E-2	–	NM	^{235}U	ICP-MS	NMRQ	3.53 E-6	1	NM
^{60}Co	GEA	NMRQ	<5 E-3	–	<34	^{236}U	ICP-MS	NMRQ	7.17 E-6	2	NM
^{63}Ni	Radchem	NMRQ	8.83 E-4	5	NM	^{238}U	ICP-MS	NMRQ	6.31 E-5	0.5	NM
^{79}Se	Radchem	NMRQ	3.52 E-6	25	NM	^{236}Pu	Radchem	NMRQ	< 4 E-7	–	NM
^{88}Y	GEA	NMRQ	<2 E-2	–	NM	^{237}Np	ICP-MS	NMRQ	6.25 E-7(5)	13	ND
^{90}Sr	Radchem	1.50 E-1	2.95 E-2	3	7	^{238}Pu	Radchem	NMRQ	6.00 E-5	4	74
^{95}Nb	GEA	NMRQ	<2 E-2	–	NM	$^{239+240}$Pu	Radchem	NMRQ	4.63 E-4	2	72
^{99}Tc	ICP-MS	3.00 E-3	[1.36 E-4] (3)	10	0.06	^{239}Pu	ICP-MS	NMRQ	7.64 E-4 (6)	3	NM
^{103}Ru	GEA	NMRQ	<2 E-1	–	NM	^{240}Pu	ICP-MS	NMRQ	1.34 E-4 (6)	13	NM
^{106}Ru	GEA	NMRQ	<7 E-1	–	NM	^{241}Pu	Radchem	NMRQ	3.06 E-3	8	NM
^{113}Sn	GEA	NMRQ	<2 E-1	–	NM	^{241}Am	Radchem	7.20E-04	3.04 E-5	3	3.8
^{125}Sb	GEA	NMRQ	<4 E-1	–	NM	^{241}Pu, ^{241}Am	ICP-MS	NMRQ	<1.6 E-4 µg/mL(7)		NM
^{126}Sn/Sb	GEA	NMRQ	2.69 E-1	19	NM	^{242}Am	Radchem	NMRQ	< 3 E-7	–	NM
^{129}I	ICP-MS	NMRQ	< 6 E-6	–	NM	^{242}Cm	Radchem	NMRQ	<2 E-7	–	ND
^{134}Cs	GEA	NMRQ	1.56 E-1	6	NM	^{242}Pu	ICP-MS	NMRQ	<1.6 E-6	–	NM
^{137}Cs	GEA	5.00 E-2	7.65 E+2	2	104	$^{243+244}$Cm	Radchem	NMRQ	7.30 E-6	6	57
^{144}Ce	GEA	NMRQ	<6 E-1	–	NM	Alpha sum(8)	Radchem	NMRQ	5.60 E-4	2	36
^{151}Sm(4)	Radchem	NMRQ	1.16 E-4	6	NM	Total alpha	Radchem	2.30E-01	9.13 E-4	11	
^{152}Eu	GEA	NMRQ	<1 E-2	–	NM	Total beta	Radchem	NMRQ	7.97 E+2	4	NM

Analyte	Method	MRQ(2) µCi/mL	Cs eluate µCi/mL	Error %	% recovered	Analyte	Method	MRQ(2) µCi/mL	Cs eluate µCi/mL	Error %	% recovered
^{154}Eu	GEA	2.00 E-3	<3 E-2	–	ND						

Notes:

NM = not measured in the feed

ND = not detected in feed or eluate

(1) ASR 6192, Sample 01-1842

(2) MRQ is minimum reportable quantity requested by Bechtel. NMRQ is no minimum reportable quantity requested.

(3) Uncertainty, including standards bias, for ^{99}Tc ranged from -10% to +30%.

(4) The ^{151}Sm is an upper bound because other beta-emitters interfered with the analysis.

(5) Uncertainty, including standards bias, was -20% to +10%.

(6) Uncertainty, including standards bias, was -10% to +20%.

(7) The AMU-241 is reported in units of µg/mL. Applying the specific activity of ^{241}Pu, sample activity is <1.6E-2 µCi/mL; applying the specific activity of ^{241}Am, sample activity is <5.4E-4 µCi/mL. The uncertainty, associated with bias standards, ranged from -10% to +20%.

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

3.2.5 Regeneration

The lead column was regenerated with 50 mL 0.25 M NaOH, collected in 1 composite fraction. The composition of this regeneration effluent solution is shown in Table 3.6. Sodium was the only cation found above the blank concentration. The charge balance between Na and OH are not equal. The counter-ion for the extra Na is predicted to be residual NO_3 from the elution step.

Table 3.6. Composition of Regeneration Solution

Analyte	Concentration, µg/mL	Concentration, M
Na^+	1020	0.044
K^+	< 40	< 1E-3
OH^-	500	0.030
Total Cs(1)	2.8 E-3	2.1 E-8
^{137}Cs	6.69 E-4	5.85 E-2 µCi/mL
Density, g/mL	1.006	
(1) Total Cs is calculated from the isotopic ratio and measured ^{137}Cs concentration		

3.2.6 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 3.7). As expected, virtually all ^{137}Cs was found in the eluate, recovering >99 % of the ^{137}Cs present in the initial AP-101DF feed. The lag column remained loaded with 35 µCi ^{137}Cs based on the integration of the lead column breakthrough curve.

Table 3.7. Activity Balance for ^{137}Cs

Solution	^{137}Cs, µCi	^{137}Cs in Feed Sample, %
Feed Sample	1.50 E+5	1.00 E+2
Effluent	1.3 E-1	8.4 E-5
Load samples	1.2 E+0	8.2 E-4
Feed displacement	2.6 E-1	1.7 E-4
DI Water Rinse	2.1 E-1	1.4 E-4
Column #1 Eluate	1.56 E+5	1.04 E+2
Column #1 DI water rinse	7.6 E+0	5.1 E-3
Column #1 regeneration	3.0 E+0	2.0 E-3
Lag column Cs loading	3.5 E+1	2.3 E-2
Total ^{137}Cs Recovery	1.56 E+5	1.04 E+2

3.2.7 SL-644 Resin Volume Changes

The SL-644 resin is known to change in volume as a function of the solution pH and ionic strength (Hassan et al., 1999). The resin BV change history is shown in Table 3.8. The volume contraction after each subsequent 0.5 M HNO_3 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 3.5 a and b. In Figure 3.5a, the BVs are normalized to the volume in the 0.25 M NaOH regeneration condition just prior to AP-101DF loading. Two process cycles are shown with the first cycle consisting of the bed conditioning steps and the second cycle consisting of the actual process test. Each process step is denoted with a number given in Table 3.8. In Figure 3.5b, the observed volume changes show clearly the greater contraction and slightly reduced expansion. Fluidizing the bed in the H-form resulted in tighter resin packing, yet the subsequent expansion in the Na-form appears to have been unaffected in that the regeneration volume is equivalent to that obtained prior to fluidization. Overall, there appears to be a general decline in resin volume in both the H-form and the Na-form. The change in resin volume may be caused in part by small losses of the resin during processing. Resin volume changes may also be attributed to compaction while swelling during the regeneration step.

Table 3.8. SL-644 Bed Volume Changes

Feed	Symbol	Process Step	BV 010319SMC-IV-73 212-425 µm PSD	
			Lead column, mL(1)	Lag column, mL(2)
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	—

(1) Calculated resin mass after washing, dry Na-form: 2.46 g.
(2) Calculated resin mass after washing, dry Na-form: 2.48 g.
Note: The inside diameter of each column was 1.46 cm.

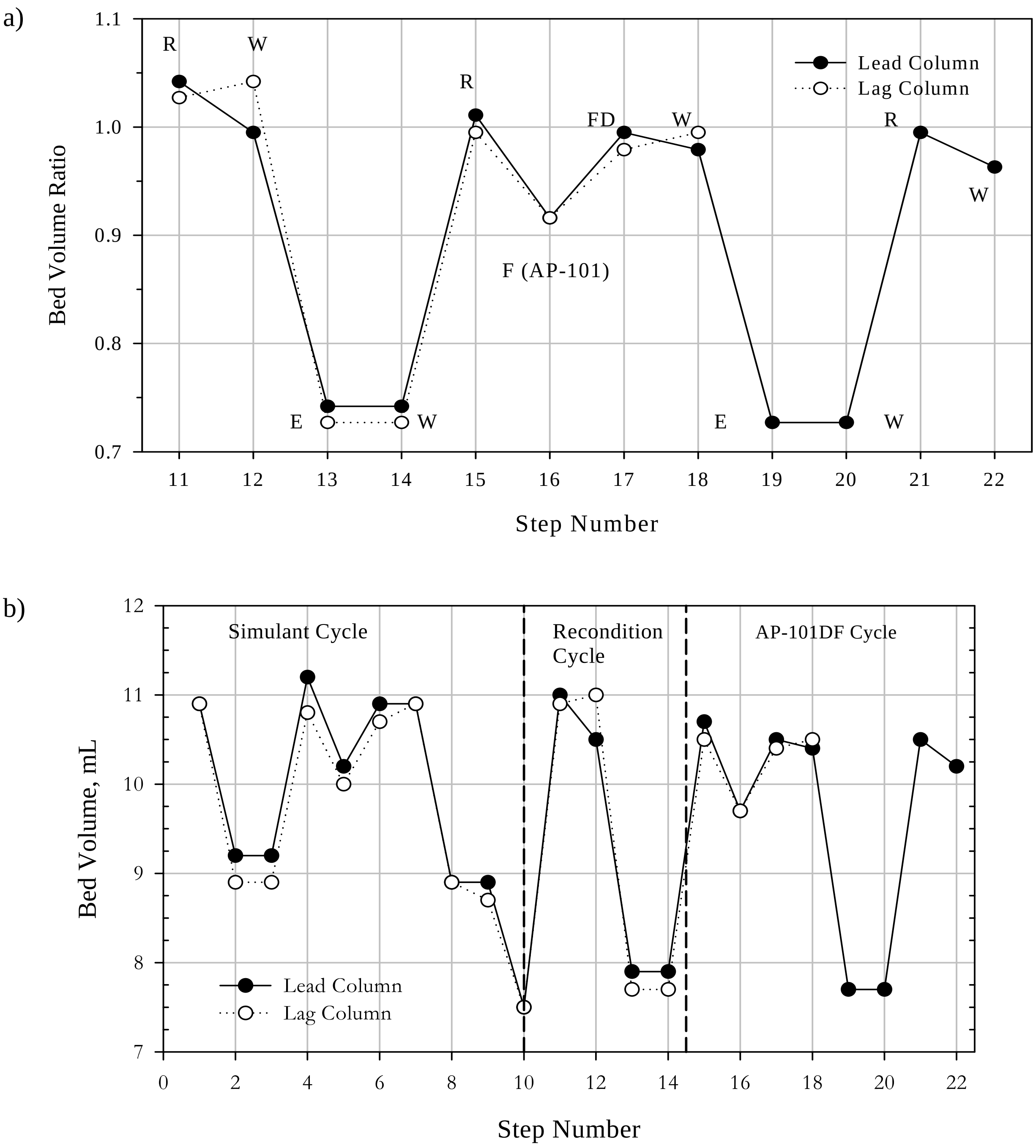

Figure 3.5. (a and b) Comparison of Bed Volumes of the Lead and Lag Columns (SL-644 Batch 010319SMC-IV-73, 212- to 425-µm)

a) Relative BV for reported testing cycle; baseline BV = 10.6 mL in the 0.25 M NaOH Regeneration Condition.

b) Actual BVs for all tested cycles, including initial simulant run.

4.0 Conclusions

The objectives of the testing were met.

Batch distribution values were developed as a function of Na/Cs mole ratios for both SL-644 and IE-911 in AP-101DF.

- The SL-644 equilibrium data resulted in a K_d value of 690 mL/g, corresponding to a predicted Cs λ of 160 BVs (0.25 M NaOH condition), at a Na/Cs mole ratio of 1.4 E+5, 25^{o}C.
- The IE-911 CST equilibrium data resulted in a K_d value of 710 mL/g, corresponding to a Cs λ value of 740 BVs, at a Na/Cs mole ratio of 1.4E+5, 25 ^{o}C.

Cs decontamination from AP-101DF (Envelope A) was successfully demonstrated.

- An overall DF of 7.8 E+5 was obtained after processing 112 BVs providing a Cs-decontaminated effluent with a ^{137}Cs concentration of 1.1 E-4 μCi/mL. This represents 0.09% of the contract limit of 1.25 E-1 μCi/mL in the treated effluent (based on 14 wt% waste Na_2O loading).[14]

Cs load and elution breakthrough profiles were developed.

- The estimated 50% Cs breakthrough for the lead ion exchange column, extrapolated from 0.2% C/C_o to 50% C/C_o, was about 190 BVs (0.25 M NaOH regeneration condition). This is a huge extrapolation and as such is inherently uncertain, however it is nearly equivalent to the predicted 160 BVs based on batch-contact studies.
- The Cs-loaded lead column was efficiently eluted with 0.5 M HNO_3. Over 94% of the ^{137}Cs was eluted from the column in 2.5 BVs of eluate. A total of 12 BVs eluate were required to reach the elution end point of $C/C_o = 0.01$. The peak C/C_o value for ^{137}Cs was 64 for the lead column. The activity balance accounted for 104% of the feed ^{137}Cs, which is essentially equivalent to 100% within experimental error.

The Cs eluate solution was composited and characterized in preparation for subsequent evaporation and HLW vitrification testing.

- A high percentage of the U (87%) and Pu (72%) was exchanged onto the resin and recovered in the acid eluate.

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[14] The effluent ^{137}Cs concentration was 0.13% of the contract limit of 0.0874 μCi/mL in the treated effluent based on the maximum waste loading of 20 wt% waste Na_2O.

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

- The SL-644 was adequately regenerated with 4.7 BVs or 2.2 AVs of 0.25 M NaOH. The average ^{137}Cs concentration in the regeneration solution was 5.8 E-2 μCi/mL, equivalent to C/C_o of 4.6 E-4.

- An activity balance for ^{137}Cs indicated 104% of the ^{137}Cs present in the feed sample was accounted for in the samples and process streams (mostly in the eluate) indicative of good experimental integrity.

- No fouling, or other off-normal conditions, of the resin bed was observed.

5.0 References

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Appendix A

Calculations

Cs-137 Contractual Limit in AP-101 Env. A Vit feed

Assumptions

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

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

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

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

5) AP-101DF actual waste and AW-101 simulant Na concentration = 5 M

6) AP-101DF actual waste ^{137}Cs concentration = 126 µCi / mL

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
(2.5E-5 Ci ^{137}Cs / mole Na) * (5 mole Na / L feed) = 1.25 E-4 Ci ^{137}Cs / L
= 125 µCi ^{137}Cs / L
= 0.125 µCi ^{137}Cs / mL

AP-101DF actual waste Cs fraction remaining (C/Co) Contractual Limit

(0.125 µCi ^{137}Cs/ mL) / (126 µCi ^{137}Cs / mL) = 9.92E-4 C/C_o
= 0.0992 % C/C_o

Decontamination Factor (DF) Contract Limit

1 / (9.92E-4 C/C_o) = 1008 C_o/C

Appendix B

Sample Identification

Table B.1. Sample Identification

Sample Description	ASR	RPL ID	Sample Identification	Extended Sample Description
Batch Contact Testing				
First batch contact with SL-644 981112YK-N3-16/18	6048	01-0575 -- 01-0576	AP101-644a and duplicate	Batch contact with SL-644, no spike
		01-0577 -- 01-0578	AP101-S1-644a and duplicate	Batch contact with SL-644, 1E-3 M Cs spike
		01-0579 -- 01-0580	AP101-S2-644a and duplicate	Batch contact with SL-6444, 5E-3 M Cs spike
Batch contact with IE-911		01-0581 -- 01-0582	AP101-CSTa and duplicate	Batch contact with IE-911 no spike
		01-0583 -- 01-0584	AP101-S1-CSTa and duplicate	Batch contact with IE-911, 1E-3M Cs spike
		01-0585 -- 01-0586	AP101-S2-CSTa and duplicate	Batch contact with IE-911, 5E-3M Cs spike
Controls		01-0587	AP101-S1a	Batch contact control (no exchanger) 1E-3M Cs
		01-0588	AP101-S2a	Batch contact control (no exchanger) 5E-3M Cs
		01-0589	AP101-Ca	Batch contact control (no exchanger), unspiked
Second batch contact with SL-644 010319SMC-IV-73	6130	01-1336--01-1337	AP101-S3-644-F/A and duplicate	Batch contact samples with SL-644, 1E-3M Cs
		01-1338	AP101- S3C-F/A	Batch contact control (no exchanger) Spike 3
		01-1339 -- 01-1340	AP101-S2a and S1a	Re-analysis of controls, Spike 1 and Spike 2
Column Run				
Initial Feed Sample, AP-101DF	6031	01-0520	AP-101DF	Initial feed sample
Load - Elution samples	6097	01-0780	AP-101-F0	Initial feed sample
		01-0781 -- 01-0785	AP-101L-F3 through F-13	Lead column load samples
		01-0786 -- 01-0788	AP-101P-F4 through F-13	Lag column load samples
		01-0789 -- 01-0791	AP101-Fcomp2-A through Fcomp4-A	Composite effluent samples
		01-0792 -- 01-0794	AP101-FD2-A through FD6-A	Feed displacement samples
		01-0795 -- 01-0796	AP101-DI2-A and DI5-A	Water rinse samples
		01-0797 -- 01-0809	AP101L-E1-DA	Lead column elution analytical samples
		01-0810	AP101L-RGN-A	Regeneration solution analytical sample
AP-101DF effluent composite	6121	01-1203	AP1-Tc-0-C	AP-101DF Cs IX effluent composite
AP-101 Cs eluent	6192	01-1842	AP101L-E-Comp	AP-101DF Cs eluant

Notes: 1) The suffix letter "a" after the sample ID indicates the sample was loaded out of the hot cell into a clean analytical vial; the suffix letter "D" after the sample ID indicates the sample was diluted prior to submission for analysis. Unidentified samples are associated with other tests unrelated to this work.

Appendix C

AP-101DF Batch Contact Calculations and Results

TI-PNNL-WTP-041, Rev. 0 Data Reduction

Batch Contact of AP101 Waste with SuperLig 644 and IE-911

AP-101
Cs concentration determination

AP-101 as-received reported concentration was 5.4 ug/mL Cs-133
The AP-101 as-received contained 144 uCi/mL Cs-137, corresponding to 1.66 ug/mL or 6.9 ug/mL total Cs
Dilution from 5.62 M Na to 4.97M Na, a dilution factor of 1.13, results in 4.8 ug/mL Cs-133
Dilution from 6.9 ug/mL total Cs by 1.13, results in 6.1 ug/mL total Cs.

AP101	Diluted uCi/mL	Sp Act	ug/mL	calc. Iso. Ratio	M	isotopic ratio	calculated total Cs
Cs-133			2.96	0.573	2.23E-05	0.6058	**4.89**
Cs-134	0.0287	1294	2.22E-05	4.30E-06	1.66E-10		
Cs-135	nm	0.0009	0.755	0.146	5.59E-06	0.1543	4.89
Cs-137	126	87	1.45	0.280	1.06E-05	0.2399	6.04
Cs Sum			5.16		3.84E-05		1.24
Na					4.97		
K			31200		0.665		

AP101	as-received uCi/mL	Sp Act	ug/mL	calc. Iso. Ratio	M	isotopic ratio	calculated total Cs	Dilute 1.13X total Cs
Cs-133			5.4	0.665	4.06E-05	0.6058	8.91	7.82
Cs-134	0.0473	1294	3.66E-05	4.50E-06	2.67E-10			
Cs-135	nm	0.0009	1.06	0.131	nm	0.1543		
Cs-137	144	87	1.66	0.20	1.21E-05	0.2399	6.90	6.05
Cs Sum			8.12		5.27E-05			

4.89 µg/mL Cs reported in AP101 DF report

AP-101 Denstiy 1.256 g/mL

Cs spiking solution
3.6696 g $CsNO_3$ 194.91 g/mole into 0.025 L 7.531E-01 M Cs See preparation in TI-41500-020, Rev. 0, Addendum A
100,085 µg/mL Cs

ID	bottle tare, g	bottle + AP101, g	AP101, g	AP101, mL	Cs spike vol., mL	Cs in AP101, µg	Cs spike conc.	Cs-133 added, µg	Final Cs conc., µg/mL	Final Cs molarity	Measured Cs ug/mL	Dilution Corrected Cs, ug/mL	Ratio found/ prepared*
unspiked	pipet directly from "DF D"		44	35.0000	0	171		0	4.89	3.68E-5			
S1	12.5603	56.8076	44.2473	35.2287	0.0497	172	100,085	4974	145.9	1.10E-3	9.33E+0	192	1.314
S2	12.7352	56.86	44.1248	35.1312	0.25	172	100,085	25021	712.0	5.36E-3	4.54E+1	953	1.338

*A positive 32% bias was found in the LCS
therefore, the as-prepared [Cs] is used

Cs spiking solution for S3
1.4672 g $CsNO_3$ 194.91 g/mole into 0.01 L 7.528E-01 M Cs See preparation in TI-RPP-WTP-082, Rev. 0
100,041 µg/mL Cs

ID	bottle tare, g	bottle + AP101, g	AP101, g	AP101, mL	Cs spike vol., mL	Cs in AP101, µg	Cs spike conc.	Cs-133 added, µg	Final Cs conc., µg/mL	Final Cs molarity	Measured Cs ug/mL	Dilution Corrected Cs, ug/mL	Ratio found/ prepared*
S3	17.2815	32.7131	15.4316	12.2863	0.0155	60	100,085	1551	130.989	9.86E-4	124	124	0.95

Na molarity =	4.97
Feed Na/Cs mole ratio:	1.35E+05

Batch Contact of AP-101DF Waste with SuperLig 644 and IE-911

AP-101
Batch contact

									Equilibrium Cs Concentration			
Sample ID	Resin	Resin, g (m)	F-factor	I_{Na} factor increase	corrected resin mass, g	AP101, mL (V)	Cs-137, uCi/mL (Aeq)	Fraction of original Cs-137	M	ug/mL	Na/Cs mole ratio	Kd
AP101-644	16/18	0.1009	0.9392	1.25	0.1185	10.0819	9.29E+0	7.41E-2	2.73E-6	3.62E-1	1.82E+6	1062
AP101-644-D	16/18	0.1004	0.9392	1.25	0.1179	10.1102	9.32E+0	7.43E-2	2.73E-6	3.64E-1	1.82E+6	1066
AP101-S1-644	16/18	0.1016	0.9392	1.25	0.1193	10.0796	2.04E+1	1.62E-1	1.78E-4	2.37E+1	2.79E+4	436
AP101-S1-644-D	16/18	0.1012	0.9392	1.25	0.1188	10.0132	2.01E+1	1.60E-1	1.76E-4	2.34E+1	2.83E+4	442
AP101-S2-644	16/18	0.101	0.9392	1.25	0.1186	9.9890	5.66E+1	4.51E-1	2.42E-3	3.21E+2	2.06E+3	102
AP101-S2-644-D	16/18	0.101	0.9392	1.25	0.1186	10.0373	5.64E+1	4.50E-1	2.41E-3	3.20E+2	2.06E+3	104
AP101-S3-644	73	0.0515	0.9321	1.25	0.0600	4.9494	2.18E+1	1.58E-1	1.56E-4	2.07E+1	3.19E+4	440
AP101-S3-644D	73	0.0504	0.9321	1.25	0.0587	5.0549	2.34E+1	1.70E-1	1.67E-4	2.22E+1	2.97E+4	422
AP101-CST	CST	0.0493	0.9630	1.00	0.0475	4.9953	1.33E+1	1.06E-1	3.89E-6	5.18E-1	1.28E+6	888
AP101-CST-D	CST	0.0492	0.9630	1.00	0.0474	5.0162	1.32E+1	1.05E-1	3.88E-6	5.16E-1	1.28E+6	899
AP101-S1-CST	CST	0.0496	0.9630	1.00	0.0478	4.9939	1.91E+1	1.52E-1	1.67E-4	2.22E+1	2.98E+4	584
AP101-S1-CST-D	CST	0.0508	0.9630	1.00	0.0489	4.9693	1.81E+1	1.45E-1	1.59E-4	2.11E+1	3.13E+4	601
AP101-S2-CST	CST	0.0511	0.9630	1.00	0.0492	4.9743	6.07E+1	4.84E-1	2.59E-3	3.45E+2	1.92E+3	108
AP101-S2-CST-D	CST	0.0518	0.9630	1.00	0.0499	4.9765	6.07E+1	4.84E-1	2.59E-3	3.45E+2	1.92E+3	106
							Ao					
AP101-S1		0				4.9717	1.26E+2	1.00E+0	1.10E-3	1.46E+2	4.53E+3	
AP101-S2		0				4.9726	1.25E+2	9.99E-1	5.36E-3	7.12E+2	9.28E+2	
AP101-C		0				4.9766	1.25E+2	9.98E-1	3.68E-5	4.89E+0	1.35E+5	
AP101-S3							1.38E+2	1.00E+0	9.86E-4	1.31E+2	5.04E+3	

Na molarity = 4.97

Kd = (Ao-Aeq)/Aeq x V/(m*F*I_{Na})

where F = F factor for water loss

Average Ao for S1, S2 and C
1.25E+2

Batch Contact of AP101 Waste with SuperLig 644 and IE-911

F-Factors

Vial ID	Vial Mass (g) incl. cap	Vial + cap + Resin mass, g	Resin Mass (g)	mass change	mass % change	F factor	Grand Average	+/-
SL-644-F1	16.9748	17.4750	0.5002					
Batch 16/18		17.4439	0.4691	0.0311	6.22	0.9378		
		17.4468	0.4720	-0.0029	-0.62	0.9436		
average						0.9407		
SL-644-F2	16.6938	17.1945	0.5007					
Batch 16/18		17.1634	0.4696	0.0311	6.21	0.9379		
		17.1631	0.4693	0.0003	0.06	0.9373		
average						0.9376	0.9392	0.0016
CST-F1	16.6125	17.1032	0.4907					
		17.0848	0.4723	0.0184	3.75	0.9625		
		17.0845	0.4720	0.0003	0.06	0.9619		
average						0.9622		
CST-F2	16.6869	17.2052	0.5183					
		17.1850	0.4981	0.0202	3.90	0.9610		
		17.1879	0.5010	-0.0029	-0.58	0.9666		
average						0.9638	0.9630	0.0008
SL-644-F1-082	17.0993	17.4046	0.3053					
Batch -73		17.3892	0.2899	0.0154	5.04	0.9496		
		17.3820	0.2827	0.0072	2.48	0.9260		
		17.3843	0.2850	-0.0023	-0.81	0.9335		
SL-644-F2-082	17.1339	17.4371	0.3032					
Batch -73		17.4211	0.2872	0.0160	5.28	0.9472		
		17.4142	0.2803	0.0069	2.40	0.9245		
		17.4161	0.2822	-0.0019	-0.68	0.9307	0.9321	0.0014

Appendix D

AP-101DF Batch Contact Analytical Results

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

Project / WP#:	42042 / F20907
ASR#:	6046 & 6048
Client:	S. Fiskum
Total Samples:	18

	From	To
RPL#:	01-00562	01-00564
Client ID:	SAW101-feed	SAN105-feed
RPL#:	01-00575	01-00589
Client ID:	AP101-644a	AP101-Ca

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

Analyst: D.R. Sanders

Analysis Date (File): 02-20-01 (A0647) & 02-21-01 (A0648)

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)

Jerry Wagner 6-7-2001
Reviewed by

6-25-01
Concur

A total of eighteen samples received as prepared liquids (AW101 simulant, AN105 simulant and AP-101 Batch Contacts), were analyzed by ICPAES. Simulant samples required an analytical dilution of 2-fold because of high sodium concentration.

Analytes of interest include Al, K and Na. Other analytes measured are for information only. All samples were prepared (PNL-ALO-106) in one batch and included a process blank, blank spike, duplicates and matrix spike. The matrix spike submitted for analysis did not appear to have any spiked analytes in it. The blank spike did have spiked analytes in it (27 analytes that included the analytes of interest Al, Na, and K).

Al, K and Na were present in all samples submitted at relatively high concentration, generally above EQL. The process blank did not have any substantial amount of Al, K, or Na greater than EQL. A few other analytes present in the process blank were either below EQL or less than 5% of sample concentration for that analyte. Boron and silicon were present at concentrations both below and above EQL with large differences noted for duplicate samples. The source of boron and silicon is likely from glassware used to process the samples.

See attached ICPAES Data Report for final results. All results reported have been corrected for sample digestion (PNL-ALO-106) according to sample preparation bench sheets supplied by the client. Bench sheets indicate that each sample aliquot (0.25mL) was weighed, digested to remove organic material, and diluted to a final volume of 25mL (also weighed).

Quality control check-standard results, relative to ICPAES instrument performance, met tolerance requirements for all analytes of interest reported except where noted below. In general sample RPD between duplicates agreed well for analytes of interest and within tolerance limits for most other analytes. Potassium measurement of the quality control check samples indicated a slightly higher than tolerance limit of up to 12%. Measurement of single element standards for potassium did not exceed about 9% above the expected concentration.

Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan.

Five fold serial dilution:

All analytes of interest were within tolerance limit of ± 10% after correcting for dilution.

Duplicate RPD (Relative Percent Difference):

All analytes of interest were within tolerance limit of ± 20%.

Post-Spiked Samples (Group A):

All analytes were recovered within tolerance of 75% to 125% recovery.

Post-Spiked Samples (Group B):

All analytes were recovered within tolerance of 75% to 125% recovery.

Blank Spike:

Other than boron, all analytes were recovered within tolerance limit of 80% to 120%.

Matrix Spiked Sample:

A sample was submitted for analysis and designated as a matrix spike but did not contain any measurable amount of spike material.

Quality Control Check Standards:

Concentration of all analytes of interest is within tolerance limit of ± 10% accuracy in the check standards: QC_MCVA, QC_MCVB, and QC_SSTMCV except potassium. One measurement of QC_SSTMCV about 11% high.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except potassium. Potassium tended to measure slightly high, from about 8% to 12%. Measurement of QC_SSTMCV for potassium (100 ug/mL) is within tolerance limits.

Process Blank:

Analytes of interest are either below EQL or less than 5% of sample concentration.

Laboratory Control Standard (LCS):

A blank-spike was prepared and analyzed as the LCS (See Blank Spike above)

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.

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

	Multiplier=	100.0	200.0	200.0	200.0	200.0
	RPL/LAB #=	BLK 0562	01-0562 @2	01-0562-DUP @2	01-0563 @2	01-0564 @2
	Client ID=	*Process Blank (ALO-106)*	*SAW101 feed*	*SAW101 feed (dup)*	*SL-Cs-COMP*	*SAN-105-feed*
Det. Limit	*Run Date=*	2/20/2001	2/20/2001	2/20/2001	2/20/2001	2/20/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	[27]	13,900	14,900	13,300	19,600
0.250	As	--	--	--	--	--
0.050	B	382	437	423	220	279
0.010	Ba	--	[12]	[12]	[11]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	[52]	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	--	--	705
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[10]	[22]	[6.1]	[14]	[6.7]
2.000	K	--	14,700	16,300	13,900	4,430
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	[51]	[53]	[47]	--
0.050	Mo	--	[28]	[29]	[28]	[44]
0.150	Na	413	110,000	121,000	105,000	135,000
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	[6.1]	--
0.100	P	--	[71]	[69]	[78]	[99]
0.100	Pb	--	[28]	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[160]	[200]	[170]	[110]	[130]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

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

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

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

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

	Multiplier=	100.0	100.0	100.0	100.0	100.0
	RPL/LAB #=	01-0575	01-0575 DUP	01-0576	01-0577	01-0578
	Client ID=	*AP-101-644a*	*AP-101-644a (dup)*	*AP-101-644a-D*	*AP-101-S1-644a*	*AP-101-S1-644a-D*
Det. Limit	***Run Date=***	2/21/2001	2/21/2001	2/21/2001	2/21/2001	2/21/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	386	392	390	385	385
0.250	As	--	--	--	--	--
0.050	B	237	353	233	241	235
0.010	Ba	[1.9]	[1.8]	[1.1]	[1.5]	[1.4]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	[6.1]	[6.4]	[6.0]	[6.2]	[6.1]
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[7.6]	[4.3]	[6.6]	[6.7]	[6.0]
2.000	K	[1,800]	[1,800]	[1,700]	[1,800]	[1,800]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	17,500	17,900	18,300	18,100	17,900
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	[24]	[22]	[20]	[22]	[23]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[150]	[170]	[130]	[120]	[120]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
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).

	Multiplier=	100.0	100.0	100.0	100.0	100.0
	RPL/LAB #=	01-0579	01-0580	01-0581	01-0582	01-0583
	Client ID=	*AP-101-S2-644a*	*AP-101-S2-644a-D*	*AP-101-CSTa*	*AP-101-CSTa-D*	*AP-101-S1-CSTa*
Det. Limit	*Run Date=*	2/21/2001	2/21/2001	2/21/2001	2/21/2001	2/21/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	393	389	385	379	372
0.250	As	--	--	--	--	--
0.050	B	241	230	384	401	407
0.010	Ba	[1.4]	[1.5]	[1.1]	--	[1.2]
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	[5.8]	[5.9]	[7.5]	[7.4]	[7.3]
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[3.9]	[6.1]	[5.9]	[5.3]	--
2.000	K	[1,800]	[1,800]	[1,700]	[1,600]	[1,600]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	19,600	19,800	19,600	19,000	18,900
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	[20]	[19]	[19]	[19]	[18]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[74]	[56]	--	[62]	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

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

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

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

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

	Multiplier=	100.0	100.0	100.0	100.0	100.0
	RPL/LAB #=	01-0584	01-0585	01-0586	01-0587	01-0588
	Client ID=	AP-101-S1-CSTa-D	AP-101-S2-CSTa	AP-101-S2-CSTa-D	AP-101- S1a	AP-101- S2a
Det. Limit	Run Date=	2/21/2001	2/21/2001	2/21/2001	2/21/2001	2/21/2001
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
0.025	Ag	--	--	--	--	--
0.060	Al	376	382	390	401	390
0.250	As	--	--	--	--	--
0.050	B	389	384	392	357	390
0.010	Ba	[1.3]	[1.1]	[1.5]	[1.6]	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	[7.4]	[7.5]	[7.7]	[7.7]	[7.8]
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	[5.9]	[20]	--	[14]
2.000	K	[1,700]	[1,600]	[1,700]	[1,800]	[1,700]
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	19,700	19,800	20,700	21,200	20,400
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	[18]	[18]	[18]	[16]	[17]
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[52]	[57]	[57]	--	[58]
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

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

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

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

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

	Multiplier=	100.0				
	RPL/LAB #=	01-0589				
	Client ID=	*AP-101- Ca*				
Det. Limit	*Run Date=*	2/21/2001				
(ug/mL)	(Analyte)	(ug/mL)				
0.025	Ag	--	--	--	--	--
0.060	Al	388	--	--	--	--
0.250	As	--	--	--	--	--
0.050	B	379	--	--	--	--
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	--	--	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	[7.4]	--	--	--	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	--	--	--	--
2.000	K	[1,700]	--	--	--	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	--	--	--
0.150	Na	20,600	--	--	--	--
0.100	Nd	--	--	--	--	--
0.030	Ni	--	--	--	--	--
0.100	P	[17]	--	--	--	--
0.100	Pb	--	--	--	--	--
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	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	--	--	--	--
0.050	Zr	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Group

2/22/01

Client : Sandy Fiskum

Cognizant Scientist: L R Greenwood Date : 2/22/01

Concur : T Trang-le Date : 2/22/01

Procedures
PNL-ALO-450 - Gamma Energy Analysis

Measured Activities (uCi/ml)

ALO ID Client ID	Co-60 Error %	Cs-134 Error %	Cs-137 Error %
01-0562 SAW 101- Feed	<3.E-5	<3.E-5	6.74E-2 2%
01-0563 SL-Cs-Comp	<2.E-5	<2.E-5	3.57E-2 5%
01-0575 AP101-644a	1.11E-4 13%	<8.E-5	4.60E-1 2%
01-0576 AP101-644a-D	<8.E-5	<8.E-5	4.57E-1 2%
01-0577 AP101-SI-644a	1.01E-4 13%	<2.E-4	9.85E-1 2%
01-0578 AP101-SI-644a-D	<7.E-5	<2.E-4	9.80E-1 2%
01-0579 AP101-S2-644a	<2.E-4	<5.E-4	2.76E+0 2%
01-0580 AP101-S2-644a-D	<9.E-5	7.74E-4 8%	2.72E+0 2%
01-0581 AP101-CSTa	1.21E-4 12%	<1.E-4	6.29E-1 2%
01-0582 AP101-CSTa-D	<8.E-5	<1.E-4	6.27E-1 2%
01-0583 AP101-SI-CSTa	<8.E-5	<1.E-4	9.00E-1 2%
01-0584 AP101-SI-CSTa-D	1.15E-4 11%	<2.E-4	8.59E-1 2%
01-0585 AP101-S2-CSTa	<9.E-5	<3.E-4	2.86E+0 2%

Measured Activities (uCi/ml)

ALO ID Client ID	Co-60 Error %	Cs-134 Error %	Cs-137 Error %
01-0586 AP101-S2-CSTa-D	1.40E-4 13%	<3.E-4	2.91E+0 2%
01-0587 AP101-S1a	<9.E-5	1.64E-3 7%	6.12E+0 2%
01-0588 AP101-S2a	<9.E-5	1.46E-3 9%	5.97E+0 2%
01-0589 AP101-Ca	<1.E-4	1.61E-3 9%	6.00E+0 2%

File: 01-0562

Battelle Pacific Northwest National Laboratory
Radiochemical Processing Group

3/7/01

Client : Sandy Fiskum

Cognizant Scientist: L R Greenwood Date : 3/7/01

Concur : T Trang-le Date : 3/7/01

Procedures
RPG-CMC-476, -408 (Sr-90)

Measured Activities (uCi/ml)

ALO ID Client ID	Sr-90 Error +/-
01-0581 AP101-CSTa	7.46E-4 36%
01-0582 AP101-CSTa-D	1.34E-3 20%
01-0583 AP101-SI-CSTa	1.02E-3 27%
01-0584 AP101-SI-CSTa-D	1.06E-3 26%
01-0585 AP101-S2-CSTa	2.52E-3 21%
01-0586 AP101-S2-CSTa-D	1.58E-3 34%
01-0586 DUP AP101-S2-CSTa-D	1.24E-3 43%
01-0587 AP101-S1a	3.82E-3 15%
01-0588 AP101-S2a	3.46E-3 17%
01-0589 AP101-Ca	4.22E-3 13%
Matrix Spike	99%
Blank Spike	106%
Blank	<5.E-6

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).

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

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

7/25/01

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

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

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

tmP Thomas 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%.

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

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Reviewed by: 4MPD 11-26-01

Analyst: James Bramson
11/27/01

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

Log-In Number	Sample ID	ICP/MS ID	MDL µg/ml	Cs-133 µg/ml	±	Cs-133 1SD	Rec/RPD %
	1%HNO3	ICB		< 1.85E-06			
	1%HNO3	CCB1		2.44E-06	±	2.88E-07	
	1%HNO3	CCB2		2.61E-06	±	3.29E-07	
	1%HNO3	CCB3		< 1.85E-06			
	1%HNO3	CCB4		2.40E-06	±	3.73E-07	
	1%HNO3	CCB5		2.32E-06	±	7.13E-07	
	1%HNO3	ICB		< 1.54E-06			
	1%HNO3	CCB3		< 2.13E-06			
	1%HNO3	CCB4		< 2.17E-06			
	1%HNO3	ICB		< 8.00E-06			
	1%HNO3	CCB1		< 8.42E-06			
	1%HNO3	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

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Appendix E

AP-101DF Column Processing Calculations and Results

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

Bed volume in 0.25 M NaOH	10.6	mL
Apparatus volume	42	mL

	Densities	
HNO3	1.012	g/mL
0.1M NaOH	0.999	
0.25M NaOH	1.006	
simple sim.	1.2069	
AP101 waste	1.256	

	apparatus volume
total system	42
column 1	23
column 2	21

Conditioning 4/23/01
Lead column + Lag column

start	end	Δt	g mass	mL volume	mL/min flow rate	BV/hr	BV	AV	
0.25M NaOH wash									
8:34	13:45	5:11	104.3	103.6779	0.33	1.89	9.8	2.47	
DI water wash									
13:45	15:50	2:05	60.6	60.6	0.48	2.74	5.7	1.44	
HNO3 wash									
15:58	19:05	3:07	83.2	82.21	0.44	2.49	7.8	1.96	
DI water wash									
19:08	22:03	2:29	73.6	73.6	0.49	2.80	6.9	1.75	
0.25M NaOH wash									
4/23/2001 23:30	4/24/2001 9:07	9:37	102.7	102.087	0.18	1.00	9.6	2.43	Volume was increased because the resin in column 2 was not completely converted.

AP101 actual waste loading starting on 4/24/01

Initial effluent collection
1 apparatus volume of feed

start	end	Δt	mass	cumul. volume	flow rate	BV/hr	apparatus volume	Sample ID	Lead column sampling start	end	Vial tare	Vial + smpl	mass	volume	total vol.	BV	C/Co	Running total sample vol
9:15	11:23	2:08	64.1	63.7	0.498	2.82	1.5	AP101L-F1	11:23	11:27	16.9634	20.3799	3.4165	2.720	66.549	6.3	2.43E-6	2.7201

Feed flow

start	end	Δt	mass	cumul. volume	flow rate	BV/hr	cumulat. BV	Sample ID	Lead column sampling start	end	Vial tare	Vial + smpl	mass	volume	total vol.	BV	C/Co	Running total sample vol
11:34	14:15	2:41	98.8	142.4	0.489	2.77	13.4	AP101L-F2	14:15	14:18	16.9839	20.0049	3.021	2.405	147.603	13.9	2.01E-6	5.1254
14:26	17:36	3:10	218.5	237.7	0.502	2.84	22.4	AP101L-F3	17:36	17:37	16.9654	19.1673	2.202	1.753	248.501	23.4	1.53E-6	6.8785
17:55	20:55	3:00	331.9	328.0	0.502	2.84	30.9	AP101L-F4	20:55	20:57	16.9213	19.7282	2.807	2.235	344.196	32.5	1.72E-6	9.1133
4/24/2001 21:06	4/25/2001 0:15	3:09	447.3	419.8	0.486	2.75	39.6	AP101L-F5	0:15	0:17	16.8744	19.6263	2.752	2.191	439.875	41.5	1.63E-6	11.3043
0:25	3:35	3:10	119.0	514.6	0.499	2.82	48.5	AP101L-F6	3:35	3:38	16.9911	19.9990	3.008	2.395	538.682	50.8	1.44E-6	13.6991
3:45	6:54	3:09	235.5	607.3	0.491	2.78	57.3	AP101L-F7	6:54	6:57	16.9758	19.9055	2.930	2.333	635.768	60.0	3.48E-6	16.0317
7:03	10:15	3:12	352.2	700.3	0.484	2.74	66.1	AP101L-F8	10:15	10:17	16.9345	19.8250	2.891	2.301	732.711	69.1	1.35E-5	18.3330
10:23	13:34	3:11	473.5	796.8	0.506	2.86	75.2	AP101L-F9	13:34	13:36	16.9052	19.3418	2.437	1.940	832.920	78.6	5.50E-5	20.2730
13:43	16:54	3:11	120.7	892.9	0.503	2.85	84.2	AP101L-F10	16:54	16:55	16.9269	19.5473	2.620	2.086	932.776	88.0	2.07E-4	22.3593
17:04	20:15	3:11	241.2	988.9	0.502	2.84	93.3	AP101L-F11	20:15	20:17	16.7901	19.3045	2.514	2.002	1032.785	97.4	6.64E-4	24.3612
20:23	23:34	3:11	359.7	1083.2	0.494	2.80	102.2	AP101L-F12	23:34	23:36	16.9869	19.5084	2.522	2.008	1130.874	106.7	1.65E-3	26.3688
4/25/2001 23:43	4/26/2001 1:22	1:39	424.7	1135.0	0.523	2.96	107.1	AP101L-F13	1:28	1:31	16.9073	20.8904	3.983	3.171	1187.713	112.0	1.93E-3	29.5400
					average	**2.82**												
ΔT, total	37:48:00																	29.5

Lag column sampling

This solution was added to the INIT-RGN bottle

Sample ID	start	end	Vial tare	Vial + sample	sample mass	volume	total vol.	BV	C/Co	Running total sample volume
AP101P-F1	11:31	11:34	17.2058	19.9555	2.750	2.189	65.91	6.22	5.6E-6	2.189
AP101P-F2	14:21	14:25	17.2799	19.4785	2.199	1.750	146.32	13.80	2.0E-6	3.940
AP101P-F3	17:50	17:55	17.2179	20.4102	3.192	3.173	244.80	23.09	7.7E-7	7.113
AP101P-F4	21:03	21:05	17.2003	19.2222	2.022	1.610	336.69	31.76	1.3E-6	8.723
AP101P-F5	0:21	0:25	17.1807	19.2736	2.093	1.666	430.24	40.59	4.3E-6	10.389
AP101P-F6	3:41	3:45	17.1585	19.6688	2.510	1.999	526.98	49.72	1.1E-6	12.388
AP101P-F7	6:59	7:03	17.2147	19.3844	2.170	1.727	621.46	58.63	7.7E-7	14.115
AP101P-F8	10:20	10:23	17.2662	19.3924	2.126	1.693	716.07	67.55	1.9E-6	15.808
AP101P-F9	13:38	13:41	17.1642	19.2634	2.099	1.671	814.32	76.82	1.2E-6	17.479
AP101P-F10	16:59	17:03	17.2030	19.7993	2.596	2.067	912.48	86.08	1.8E-6	19.547
AP101P-F11	20:19	20:23	17.1547	19.3334	2.179	1.735	1010.16	95.30	4.1E-6	21.281
AP101P-F12	23:39	23:43	17.1809	19.5873	2.406	1.916	1106.42	104.38	1.2E-6	23.197
AP101P-F13	1:36	1:40	17.0099	18.4433	1.433	1.141	1159.31	109.37	2.1E-6	24.338

24.3

0.1M NaOH feed displacement

Sample ID		Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	cumul. BV	AV	Cumul. AV	C/Co
		1:41	3:05	1:24	483.6	532.0	48.4	38.5	0.46	2.60	3.64	3.64	0.92	0.92	
AP101-FD-1		3:14	3:34	0:20	17.1797	29.4498	12.3	12.3	0.61	3.48	1.16	4.79	0.29	1.21	1.85E-5
AP101-FD-2		3:35	3:55	0:20	17.1226	26.9840	9.9	9.9	0.49	2.79	0.93	5.73	0.24	1.44	1.66E-5
AP101-FD-3		3:56	4:16	0:20	17.2508	27.1071	9.9	9.9	0.49	2.79	0.93	6.66	0.23	1.68	2.70E-5
AP101-FD-4		4:17	4:40	0:23	17.1142	28.4410	11.3	11.3	0.49	2.79	1.07	7.73	0.27	1.95	2.51E-5
AP101-FD-5		4:41	5:01	0:20	17.1802	26.7305	9.6	9.6	0.48	2.71	0.90	8.63	0.23	2.18	2.43E-5
AP101-FD-6		5:01	5:22	0:21	17.2648	26.8727	9.6	9.6	0.46	2.59	0.91	9.53	0.23	2.41	2.47E-5
AP101-FD-7		5:23	5:43	0:20	16.9496	27.0879	10.1	10.1	0.51	2.87	0.96	10.49	0.24	2.65	2.48E-5
AP101-FD-8		5:44	6:04	0:20	17.1927	26.6840	9.5	9.5	0.48	2.69	0.90	11.39	0.23	2.87	2.74E-5
AP101-FD-9		6:05	6:25	0:20	17.4383	27.3385	9.9	9.9	0.50	2.80	0.93	12.32	0.24	3.11	2.87E-5
AP101-FD-10		6:26	6:46	0:20	17.4177	27.2543	9.8	9.8	0.49	2.79	0.93	13.25	0.23	3.34	2.94E-5
	ΔT, total		5:05			total vol.	101.9	average	0.48	2.74					

The initial rinse was collected in the AP-101DF effluent bottle.

DI water rinse

Sample ID		Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	cumul. BV	AV	Cumul. AV	C/Co
AP101-DI-1		6:53	7:13	0:20	17.4118	27.1571	9.745	9.745	0.49	2.76	0.9	0.9	0.23	0.23	3.06E-5
AP101-DI-2		7:14	7:34	0:20	17.4666	27.2705	9.804	9.804	0.49	2.77	0.9	1.8	0.23	0.47	3.11E-5
AP101-DI-3		7:36	7:56	0:20	17.3339	26.6221	9.288	9.288	0.46	2.63	0.9	2.7	0.22	0.69	3.79E-5
AP101-DI-4		7:57	8:17	0:20	17.5949	27.9263	10.331	10.331	0.52	2.92	1.0	3.7	0.25	0.93	3.53E-5
AP101-DI-5		8:19	8:39	0:20	17.4213	28.2286	10.807	10.807	0.54	3.06	1.0	4.7	0.26	1.19	2.37E-5
AP101-DI-6		8:41	9:02	0:21	17.2843	28.1969	10.913	10.913	0.52	2.94	1.0	5.7	0.26	1.45	1.66E-5
AP101-DI-7		9:03	9:23	0:20	17.5350	27.9468	10.412	10.412	0.52	2.95	1.0	6.7	0.25	1.70	1.36E-5
AP101-DI-8		9:25	9:46	0:21	17.4760	28.3187	10.843	10.843	0.52	2.92	1.0	7.7	0.26	1.96	1.14E-5
AP101-DI-9		9:47	10:07	0:20	17.4884	27.8184	10.330	10.330	0.52	2.92	1.0	8.7	0.25	2.20	1.22E-5
	ΔT, total		3:14					average	0.51	2.86					

density	1 g/mL

Cs elution from lead column,
Separate columns

	Sample ID	start	end	Δt	Vial tare	Vial + sample	mass colleccted	volume	flow rate	BV/hr	cumul. BV	C/Co
	HNO3 wash--lead column											
	AP101L-E1	10:19	11:20	1:01	17.4281	27.2318	9.8037	9.69	0.16	0.90	0.91	6.80E-5
	AP101L-E2	11:32	12:32	1:00	17.5184	27.9966	10.4782	10.35	0.17	0.98	1.89	5.99E-3
	AP101L-E3	12:33	13:33	1:00	17.3130	26.5583	9.2453	9.14	0.15	0.86	2.75	3.36E+1
	AP101L-E4	13:34	14:35	1:01	17.3870	26.4352	9.0482	8.94	0.15	0.83	3.60	1.01E+2
	AP101L-E5	14:37	15:37	1:00	17.4172	26.2987	8.8815	8.78	0.15	0.83	4.42	5.10E+1
	AP101L-E6	15:38	16:38	1:00	17.4236	27.0074	9.5838	9.47	0.16	0.89	5.32	4.05E+0
	AP101L-E7	16:39	17:39	1:00	17.5534	27.0399	9.4865	9.37	0.16	0.88	6.20	5.31E-1
	AP101L-E8	17:45	18:45	1:00	17.4160	27.6899	10.2739	10.15	0.17	0.96	7.16	8.60E-2
	AP101L-E9	18:46	19:50	1:04	17.2890	27.3419	10.0529	9.93	0.16	0.88	8.10	2.92E-2
	AP101L-E10	19:51	20:46	0:55	17.3951	25.8487	8.4536	8.35	0.15	0.86	8.88	2.20E-2
	AP101L-E11	20:50	21:50	1:00	17.2808	26.6313	9.3505	9.24	0.15	0.87	9.76	1.63E-2
	AP101L-E12	21:52	22:52	1:00	17.3601	26.7641	9.4040	9.29	0.15	0.88	10.63	1.16E-2
	AP101L-E13	22:53	23:53	1:00	17.4525	26.3552	8.9027	8.80	0.15	0.83	11.46	9.26E-3
	AP101L-E14	23:54	0:54	1:00	17.4432	26.4375	8.9943	8.89	0.15	0.84	12.30	7.52E-3
	AP101L-E15	0:56	1:56	1:00	17.4823	27.1091	9.6268	9.51	0.16	0.90	13.20	6.06E-3
	AP101L-E16	1:57	2:57	1:00	17.3776	26.7528	9.3752	9.26	0.15	0.87	14.07	5.24E-3
	AP101L-E17	2:58	3:58	1:00	17.4015	26.3984	8.9969	8.89	0.15	0.84	14.91	4.38E-3
	AP101L-E18	4:00	5:01	1:01	17.4323	27.1386	9.7063	9.59	0.16	0.89	15.82	4.00E-3
	AP101L-E19	5:04	6:04	1:00	17.2560	26.5876	9.3316	9.22	0.15	0.87	16.69	3.40E-3
	AP101L-E20	6:05	7:05	1:00	17.4017	26.6698	9.2681	9.16	0.15	0.86	17.55	3.22E-3
	AP101L-E21	8:56	9:56	1:00	17.3078	27.1111	9.8033	9.69	0.16	0.91	18.46	4.29E-3
+drained bed	AP101L-E22	9:57	10:56	0:59	17.3822	26.2543	8.8721	8.77	0.15	0.84	19.29	4.16E-3
	ΔT, total		24:37:00			**sum**	**206.94**	**204.49**				
								average	**0.15**	**0.88**		

DI water

Sample ID	Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV
AP101-EDI-1	11:02	11:23	0:21	17.3964	27.8227	10.426	10.426	0.50	2.81	1.0	1.0	0.45	0.45
AP101-EDI-2	11:24	11:45	0:21	17.4730	28.1401	10.667	10.667	0.51	2.88	1.0	2.0	0.46	0.92
AP101-EDI-3	13:45	14:04	0:19	17.4785	26.6420	9.164	9.164	0.48	2.73	0.9	2.9	0.40	1.32
AP101-EDI-4	14:05	14:25	0:20	17.3782	28.0442	10.666	10.666	0.53	3.02	1.0	3.9	0.46	1.78
AP101-EDI-5	14:26	14:46	0:20	17.3632	27.6428	10.280	10.280	0.51	2.91	1.0	4.8	0.45	2.23
AP101-EDI-6	14:48	15:06	0:18	17.4474	26.4792	9.032	9.032	0.50	2.84	0.9	5.7	0.39	2.62
AP101-EDI-7	15:07	15:33	0:26	17.4391	30.6266	13.188	13.188	0.51	2.87	1.2	6.9	0.57	3.19
	ΔT, total		4:31				**average**	**0.51**	**2.86**				
							73.422						

density 1 g/mL

Regeneration with 0.25M NaOH

Sample ID	Start	End	Δt	Bottle tare	Bottle + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV	
AP101L-RGN	9:19	10:57	1:38	24.7	40.2	15.5	15.5	0.16	0.90	1.5	1.5	0.67	0.67	
	11:05	11:45	0:40	40.2	51.0	10.8	10.8	0.27	1.53	1.0	2.5	0.47	1.14	
	11:51	14:45	2:54	51.0	63.3	12.3	12.3	*0.07*	*0.40*	1.2	3.6	0.53	1.68	The pump was inadvertently turned off during this event
	14:55	15:38	0:43	63.3	74.7	11.4	11.4	0.27	1.50	1.1	4.7	0.50	2.17	thus flow rate cannot be calculated.
ΔT, total		6:19				**sum**	**50.0**							

Rinse with DI water

Sample ID	Start	End	Δt	Vial tare	Vial + sample	Sample mass	Sample Volume	mL/min	BV/hr	BV	Cumul. BV	AV	Cumul. AV	
AP101-DIRinse-Final	15:44	16:24	0:40	74.7	93.6	18.9	18.9	0.47	2.67	1.8	1.8	0.82	0.82	This sample was added to the AP101L-RGN
	8:13	8:50	0:37	14.7	31.9	17.2	17.2	0.46	2.63	1.6	1.6	0.75	1.57	
	8:53	9:32	0:39	31.9	48.9	17.0	17.0	0.44	2.47	1.6	1.6	0.74	2.31	
					sum		**53.1**							

Counting Data for TI-RPP-WTP-055, Rev. 0

Background Counts

Date	Net counts	Jncertainty	ime (sec)	cpm	uncertainty %	FileName
3/27/2001	123	21	56059	0.13	17%	BKG1
3/28/2001	104	20	43074	0.14	19%	BKG2
4/2/2001	11	3	3000	0.22	27%	BKG3
			average	**0.14**		

3000 sec MDL
0.29 cpm
1.13E-7 C/Co
0.118 cpm/g

AP-101Comparitor Standard density **1.256** g/mL

0.5058g AP101 brought to 5.0150g then 0.1001 g pipetted into a 2-mL geometry (diluent is 0.1 M NaOH). 0.01010 g in counting geometry

Date	Net counts	Error	Time (sec)	cpm	net cpm	mass	net cpm/g	mL	cpm/mL	FileName
4/25/01	17301	236	100	10381	10380.5	0.0101	1.03E+6	0.0080	1.29E+6	A101F01
4/26/01	17256	217	100	10353.6	10353.5	0.0101	1.03E+6	0.0080	1.29E+6	A101F02
4/26/01	16977	203	100	10186.2	10186.1	0.0101	1.01E+6	0.0080	1.27E+6	A101F03
4/27/01	17571	218	100	10542.6	10542.5	0.0101	1.04E+6	0.0080	1.31E+6	A101F04
4/27/01	17255	221	100	10353	10352.9	0.0101	1.03E+6	0.0080	1.29E+6	A101F05
4/30/01	18199	208	100	10919.4	10919.3	0.0101	1.08E+6	0.0080	1.36E+6	A101F07
	17688	218	100	10612.8	10612.7	0.0101	1.05E+6	0.0080	1.32E+6	A101F08
	17400	204	100	10440	10439.9	0.0101	1.03E+6	0.0080	1.30E+6	A101F09
5/2/01	17253	220	100	10351.8	10351.7	0.0101	1.02E+6	0.0080	1.29E+6	A101F10
	17301	216	100	10380.6	10380.5	0.0101	1.03E+6	0.0080	1.29E+6	A101F11
	17682	206	100	10609.2	10609.1	0.0101	1.05E+6	0.0080	1.32E+6	A101F12
5/3/01	17208	215	100	10324.8	10324.7	0.0101	1.02E+6	0.0080	1.28E+6	A101F13
5/3/01	17359	212	100	10415.4	10415.3	0.0101	1.03E+6	0.0080	1.30E+6	A101F14
5/16/01	55490	353	300	11098	11097.9	0.0101	1.10E+6	0.0080	1.38E+6	A101F18
5/16/01	53479	369	300	10695.8	10695.7	0.0101	1.06E+6	0.0080	1.33E+6	A101F19
						Average	**1.04E+6**		**1.31E+6**	
						standard deviation	**1.94E+4**		**2.44E+4**	
						% standard deviation	**1.9**		**1.9**	

1.554E+09 total cpm

ROI is slightly larger than samples
so that the entire peak can be integrated.
Sample peaks are much smaller and exibhit
another small peak close to Cs-137.

AP-101 run starting 4/24/01
Lead column, loading phase

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	FileName	C/Co	BV	% C/Co	DF	total net cpm
AP101L-F1	4/25/01	319	37	3000	6.24	2.4747	1.9703	2.52	3.17	A101LF1	2.43E-6	6.3	2.43E-4		6
AP101L-F2	4/25/01	267	38	3000	5.20	2.4872	1.9803	2.09	2.63	A101LF2	2.01E-6	13.9	2.01E-4	4.97E+5	5
AP101L-F3	4/25/01	176	39	3000	3.38	2.1284	1.6946	1.59	2.00	A101LF3	1.53E-6	23.4	1.53E-4	6.54E+5	3
AP101L-F4	4/25/01	254	73	3000	4.94	2.7638	2.2005	1.79	2.25	A101LF4	1.72E-6	32.5	1.72E-4	5.81E+5	5
AP101L-F5	4/25/01	236	42	3000	4.58	2.7046	2.1533	1.69	2.13	A101LF5	1.63E-6	41.5	1.63E-4	6.14E+5	5
AP101L-F6	4/25/01	226	43	3000	4.38	2.935	2.3368	1.49	1.88	A101LF6	1.44E-6	50.8	1.44E-4	6.96E+5	4
AP101L-F7	4/25/01	530	48	3000	10.46	2.8884	2.2997	3.62	4.55	A101LF7	3.48E-6	60.0	3.48E-4	2.87E+5	10
AP101L-F8	4/25/01	1879	60	3000	37.44	2.6742	2.1291	14.00	17.59	A101LF8	1.35E-5	69.1	1.35E-3	7.42E+4	37
AP101L-F9	4/25/01	6633	91	3000	132.52	2.3192	1.8465	57.14	71.77	A101LF9	5.50E-5	78.6	5.50E-3	1.82E+4	133
AP101L-F10	4/26/01	5417	76	600	541.56	2.5148	2.0022	215.3	270.5	A101LF10	2.07E-4	88.0	2.07E-2	4.83E+3	542
AP101L-F11	4/26/01	15867	136	600	1586.56	2.3005	1.8316	689.7	866.2	A101LF11	6.64E-4	97.4	6.64E-2	1.51E+3	1587
AP101L-F12	4/26/01	8354	105	120	4176.86	2.4369	1.9402	1714.0	2152.8	A101LF12	1.65E-3	106.7	1.65E-1	6.06E+2	4177
AP101L-F13	4/26/01	12514	173	120	6256.86	3.1203	3.9199	2005.2	1596.2	A101LF13	1.93E-3	112.0	1.93E-1	5.18E+2	6257
														% Cs cpm (sum)	**8.22E-4**

AP-101 actual waste 4/24/01
Lag column, loading phase

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	mass	mL	net cpm/g	net cpm/mL	FileName	C/Co	BV	C/Co, %	DF	total net cpm
AP101P-F1	4/26/01	742	38	3000	14.702	2.5134	2.0011	5.85	7.35	A101PF1	5.63E-6	6.2	5.63E-4		15
AP101P-F2	4/26/01	224	31	3000	4.342	2.1352	1.7000	2.03	2.55	A101PF2	1.96E-6	13.8	1.96E-4	5.11E+5	4
AP101P-F3	4/26/01	132	39	3000	2.502	3.1356	2.4965	0.80	1.00	A101PF3	7.68E-7	23.1	7.68E-5	1.30E+6	3
AP101P-F4	4/26/01	138	36	3000	2.622	1.9781	1.5749	1.33	1.66	A101PF4	1.28E-6	31.8	1.28E-4	7.84E+5	3
AP101P-F5	4/26/01	431	40	3000	8.482	1.9157	1.5252	4.43	5.56	A101PF5	4.26E-6	40.6	4.26E-4	2.35E+5	8
AP101P-F6	4/26/01	150	38	3000	2.862	2.4262	1.9317	1.18	1.48	A101PF6	1.13E-6	49.7	1.13E-4	8.81E+5	3
AP101P-F7	4/26/01	92	37	3000	1.702	2.1304	1.6962	0.80	1.00	A101PF7	7.69E-7	58.6	7.69E-5	1.30E+6	2
AP101P-F8	4/26/01	211	36	3000	4.082	2.0266	1.6135	2.01	2.53	A101PF8	1.94E-6	67.6	1.94E-4	5.16E+5	4
AP101P-F9	4/25/01	131	37	3000	2.482	2.0211	1.6092	1.23	1.54	A101PF9	1.18E-6	76.8	1.18E-4	8.46E+5	2
AP101P-F10	4/26/01	238	41	3000	4.622	2.4134	1.9215	1.92	2.41	A101PF10	1.84E-6	86.1	1.84E-4	5.43E+5	5
AP101P-F11	4/26/01	451	38	3000	8.882	2.0649	1.6440	4.30	5.40	A101PF11	4.14E-6	95.3	4.14E-4	2.42E+5	9
AP101P-F12	4/26/01	147	40	3000	2.802	2.2695	1.8069	1.23	1.55	A101PF12	1.19E-6	104.4	1.19E-4	8.42E+5	3
AP101P-F13	4/27/01	159	33	3000	3.042	1.3962	1.1116	2.18	2.74	A101PF13	2.10E-6	109.4	2.10E-4	4.77E+5	3
AP101FEcomp1	5/1/01	245	18	3000	4.762	2.1272	1.6936	2.24	2.81	Fcomp1	2.15E-6		2.15E-4	4.64E+5	
AP101FEcomp2	5/1/01	228	32	3000	4.422	2.5005	1.9908	1.77	2.22	Fcomp2	1.70E-6		1.70E-4	5.88E+5	
AP101FEcomp3	5/1/01	198	41	3000	3.822	2.5129	2.0007	1.52	1.91	Fcomp3	1.46E-6		1.46E-4	6.83E+5	
AP101FEcomp4	5/1/01	379	39	3000	7.442	2.4539	1.9537	3.03	3.81	Fcomp4	2.92E-6		2.92E-4	3.43E+5	
														% Cs cpm (sum)	**4.062E-06**

Counting Data

Data Reduction TI-RPP-WTP-055, Rev. 0

Feed Displacement

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	Tare	Tare + sample	mass	mL	net cpm/g	net cpm/mL	File Name	C/Co	BV	C/Co, %	Cum BV	total net cpm
AP101-FD-1	4/27/01	2048	49	3000	40.82	17.550	19.672	2.121	2.1212	19.24	19.24	A101FD1	1.85E-5	4.8	1.85E-3	114.2	236
AP101-FD-2	4/27/01	1740	43	3000	34.66	17.3756	19.3816	2.006	2.0060	17.28	17.28	A101FD2	1.66E-5	5.7	1.66E-3	119.9	171
AP101-FD-3	4/27/01	2801	54	3000	55.88	17.497	19.4854	1.988	1.9884	28.10	28.10	A101FD3	2.70E-5	6.7	2.70E-3	126.5	277
AP101-FD-4	4/27/01	2620	52	3000	52.26	17.4183	19.4236	2.005	2.0053	26.06	26.06	A101FD4	2.51E-5	7.7	2.51E-3	134.3	295
AP101-FD-5	4/27/01	2538	51	3000	50.62	17.1808	19.1819	2.001	2.0011	25.30	25.30	A101FD5	2.43E-5	8.6	2.43E-3	142.9	242
AP101-FD-6	4/30/01	2572	51	3000	51.30	17.3666	19.3676	2.001	2.0010	25.64	25.64	A101FD6	2.47E-5	9.5	2.47E-3	152.4	247
AP101-FD-7	4/30/01	2596	51	3000	51.78	17.3926	19.3977	2.005	2.0051	25.83	25.83	A101FD7	2.48E-5	10.5	2.48E-3	162.9	262
AP101-FD-8	4/30/01	2845	54	3000	56.76	17.4741	19.4663	1.992	1.9922	28.49	28.49	A101FD8	2.74E-5	11.4	2.74E-3	174.3	271
AP101-FD-9	4/30/01	2985	55	3000	59.56	17.4584	19.4561	1.998	1.9977	29.82	29.82	A101FD9	2.87E-5	12.3	2.87E-3	186.6	295
AP101-FD-10	4/30/01	3069	55	3000	61.24	17.249	19.2547	2.006	2.0057	30.53	30.53	A101FD10	2.94E-5	13.3	2.94E-3	199.9	301
																% cpm	1.67E-4

DI Rinse

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	Tare	Tare + sample	mass	mL	net cpm/g	net cpm/mL	FileName	C/Co	BV	C/Co, %	Cum BV	total net cpm
AP101-FDI-1	5/1/01	3197	58	3000	63.80	17.236	19.243	2.008	2.0075	31.78	31.78	A101DI1	3.06E-5	14.2	3.06E-3	214.1	310
AP101-FDI-2	5/1/01	3248	57	3000	64.82	17.3381	19.3442	2.006	2.0061	32.31	32.31	A101DI2	3.11E-5	15.1	3.11E-3	229.2	317
AP101-FDI-3	5/1/01	3949	64	3000	78.84	17.1902	19.1921	2.002	2.0019	39.38	39.38	A101DI3	3.79E-5	16.0	3.79E-3	245.1	366
AP101-FDI-4	5/2/01	3683	61	3000	73.52	17.1719	19.1773	2.005	2.0054	36.66	36.66	A101DI4	3.53E-5	16.9	3.53E-3	262.1	379
AP101-FDI-5	5/2/01	2479	50	3000	49.44	17.281	19.2884	2.007	2.0074	24.63	24.63	A101DI5	2.37E-5	18.0	2.37E-3	280.0	266
AP101-FDI-6	5/2/01	1691	41	3000	33.68	17.3827	19.3384	1.956	1.9557	17.22	17.22	A101DI6	1.66E-5	19.0	1.66E-3	299.0	188
AP101-FDI-7	5/2/01	1388	37	3000	27.62	17.2286	19.1837	1.955	1.9551	14.13	14.13	A101DI7	1.36E-5	20.0	1.36E-3	319.0	147
AP101-FDI-8	5/2/01	1187	34	3000	23.60	17.332	19.3223	1.990	1.9903	11.86	11.86	A101DI8	1.14E-5	21.0	1.14E-3	340.0	129
AP101-FDI-9	5/2/01	1265	35	3000	25.16	17.3218	19.3105	1.989	1.9887	12.65	12.65	A101DI9	1.22E-5	22.0	1.22E-3	362.0	131
				total	1982	cpm in 90 mL of DI water rinse solution			17.9181							% cpm	1.44E-4

AP-101 run starting 4/24/01.
Lead column, eluting phase

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	Mass to dilute	Dilution mass	Mass to dilute	Dilution mass	mass for counting	counted mass	mL	net cpm/g	net cpm/mL	FileName	C/Co	BV
AP101L-E1	4/27/01	9	3	1830	0.16	0.0944	5.2896	1	1	0.1	1.78E-3	1.76E-3	8.79E+1	8.89E+1	A101LE1A	6.80E-5	0.9
AP101L-E2	4/27/01	4	2	925.6	0.1	0.0861	5.0544	0.0932	10.1419	0.1	1.57E-5	1.55E-5	7.73E+3	7.82E+3	A101LE2A	5.99E-3	1.9
AP101L-E3	4/27/01	3,809	62	605.32	377.4	0.0958	10.1239	0.0929	10.1212	0.1	8.69E-6	8.58E-6	4.35E+7	4.40E+7	A101LE3	3.36E+1	2.8
AP101L-E4	4/27/01	5777	78	300	1155	0.097	10.1252	0.0938	10.1397	0.1	8.86E-6	8.76E-6	1.30E+8	1.32E+8	A101LE4	1.01E+2	3.6
AP101L-E5	4/27/01	5987	79	300	1197.3	0.0965	10.1316	0.0966	5.0644	0.1	1.82E-5	1.80E-5	6.59E+7	6.67E+7	A101LE5	5.10E+1	4.4
AP101L-E6	4/27/01	939	30	300	187.7	0.0931	5.0589	0.0982	5.0425	0.1	3.58E-5	3.54E-5	5.24E+6	5.30E+6	A101LE6	4.05E+0	5.3
AP101L-E7	4/27/01	6417	82	300	1283.3	0.0945	5.055	1	1	0.1	1.87E-3	1.85E-3	6.86E+5	6.95E+5	A101LE7	5.31E-1	6.2
AP101L-E8	4/27/01	1011	32	300	202.1	0.0914	5.0226	1	1	0.1	1.82E-3	1.80E-3	1.11E+5	1.12E+5	A101E8	8.60E-2	7.2
AP101L-E9	4/30/01	3198	57	3000	64	0.0853	5.044	1	1	0.1	1.69E-3	1.67E-3	3.77E+4	3.82E+4	A101E9	2.92E-2	8.1

Counting Data

Data Reduction TI-RPP-WTP-055, Rev. 0

AP101L-E10	4/30/01	2798	54	3000	56	0.0994	5.0491	1	1	0.1	0.0020	1.95E-3	2.84E+4	2.87E+4	A101E10	2.20E-2	8.9
AP101L-E11	4/30/01	2015	45	3000	40	0.0963	5.0441	1	1	0.1	0.0019	1.89E-3	2.10E+4	2.13E+4	A101E11	1.63E-2	9.8
AP101L-E12	5/3/01	5813	77	600	581	0.0967	5.0619	1	1	2.0325	0.0388	0.0384	14968	15147	LE12D	1.16E-2	10.6
AP101L-E13	5/3/01	5273	74	700	452	0.0939	5.0541	1	1	2.0343	0.0378	0.0373	11955	12098	LE13D	9.26E-3	11.5
AP101L-E14	5/3/01	5030	72	800	377	0.0962	5.0598	1	1	2.0429	0.0388	0.0384	9709	9826	LE14D	7.52E-3	12.3
AP101L-E15	5/8/01	5493	74	1100	299	0.0953	5.0675	1	1	2.0345	0.0383	0.0378	7827	7921	LE15D	6.06E-3	13.2
AP101L-E16	5/3/01	4363	67	1000	262	0.0961	5.0539	1	1	2.0349	0.0387	0.0382	6762	6843	LE16D	5.24E-3	14.1
AP101L-E17	4/27/01	963	31	300	192	0.0864	5.0455	1	1	1.9857	0.0340	0.0336	5660	5728	LE17	4.38E-3	14.9
AP101L-E18	4/27/01	811	29	300	162	0.0788	5.0241	1	1	2.0009	0.0314	0.0310	5164	5226	LE18	4.00E-3	15.8
AP101L-E19	4/27/01	835	28	300	167	0.0956	5.0485	1	1	2.0087	0.0380	0.0376	4387	4439	LE19	3.40E-3	16.7
AP101L-E20	4/27/01	779	27	300	156	0.0934	5.035	1	1	2.0149	0.0374	0.0369	4165	4215	LE20	3.22E-3	17.6
AP101L-E21	5/8/01	5880	77	1700	207	0.0926	5.0677	1	1	2.047	0.0374	0.0370	5545	5611	LE21D2	4.29E-3	18.5
AP101L-E22	5/3/01	5907	78	1700	208	0.0968	5.086	1	1	2.0391	0.0388	0.0383	5368	5433	LE22D	4.16E-3	19.3
					sum	2.0516	g										
					sum	2.021	mL										

Deionized water rinse following elution

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	Tare	Tare + sample	mass	mL	net cpm/g	net cpm/mL	FileName	C/Co	BV	BV post Elution	total net cpm
AP101-EDI-1	5/16/01	28825	207	300	5765	17.1953	19.2215	2.0262	2.0000	2845	2882	EDI1	2.21E-3	1.0	20.3	30053
AP101-EDI-2	5/16/01	19985	158	300	3997	17.5255	19.5530	2.0275	2.0000	1971	1998	EDI2	1.53E-3	2.0	21.3	21317
AP101-EDI-3	5/16/01	15812	142	300	3162	17.2598	19.2555	1.9957	2.0000	1585	1581	EDI3	1.21E-3	2.9	22.1	14489
AP101-EDI-4	5/16/01	8898	101	300	1779	17.3038	19.2953	1.9915	2.0000	894	890	EDI4	6.81E-4	3.9	23.2	9490
AP101-EDI-5	5/16/01	5221	73	700	447	17.3641	19.3512	1.9871	2.0000	225	224	EDI5	1.71E-4	4.8	24.1	2299
AP101-EDI-6	5/16/01	6324	83	1700	223	17.4638	19.4564	1.9926	2.0000	112	112	EDI6	8.53E-5	5.7	25.0	1007
AP101-EDI-7	5/16/01	5848	78	2000	175	17.4013	19.4029	2.0016	2.0000	88	88	EDI7	6.71E-5	6.9	26.2	1156
															% Cs in DI water rinse	**5.14E-3**

Regeneration with 0.25 M NaOH

Sample ID	Date	Net counts	Error	Time (sec)	net cpm	Tare	Tare + sample	mass	mL	net cpm/g	net cpm/mL	FileName	C/Co
AP101-RGN	5/8/01	5161	74	200	1548	17.5401	19.5629	2.0228	2.0107	765	770	RGN	5.89E-4

Regeneration with 0.25 M NaOH Analytical Lab Data

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co
AP101-RGN	01-00810	5.85E-02	2.55	4.64E-04

1.95E-3 % Cs in Regeneration solution

AP-101 run starting 4/24/01
Lead column, loading phase

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	% C/Co	Ratio analytical/ lab GEA C/Co	total Cs-137, µCi/mL
AP101-F0A	01-00780	126	1.32	1.00E+0			1.50E+05
AP101L-F3	01-00781	2.18E-04	3.5	1.73E-6	1.73E-04	1.13	
AP101L-F6	01-00782	1.28E-04	3.76	1.02E-6	1.02E-04	0.71	
AP101L-F9	01-00783	4.69E-03	2.44	3.72E-5	3.72E-03	0.68	
AP101L-F11	01-00784	6.49E-02	2.51	5.15E-4	5.15E-02	0.78	
AP101L-F13	01-00785	2.76E-01	1.33	2.19E-3	2.19E-01	1.14	

AP-101 actual waste 4/24/01
Lag column, loading phase

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	% C/Co	Ratio analytical/ lab GEA C/Co	DF
AP101P-F1							
AP101P-F2							
AP101P-F3							
AP101P-F4	01-00786	6.17E-05	7.02	4.90E-7	4.90E-05	3.84E-1	
AP101P-F5							
AP101P-F6							
AP101P-F7							
AP101P-F8	01-00787	9.00E-05	7.38	7.14E-7	7.14E-05	3.69E-1	
AP101P-F9							
AP101P-F10							
AP101P-F11							
AP101P-F12							
AP101P-F13	01-00788	7.40E-05	9.83	5.88E-7	5.88E-05	2.80E-1	1.70E+06
AP101FEcomp2	01-00789	9.79E-05	4.84	7.77E-7	7.77E-05	4.57E-1	1.29E+06
AP101FEcomp3	01-00790	1.09E-04	7.98	8.61E-7	8.61E-05	5.88E-1	1.16E+06
AP101FEcomp4	01-00791	2.31E-04	3.08	1.83E-6	1.83E-04	6.28E-1	5.46E+05

Feed Displacement

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	% C/Co	Ratio analytical/ lab GEA C/Co
AP101-FD-1						
AP101-FD-2	01-00792	5.93E-03	1.51	4.70E-5	4.70E-03	2.83E+0
AP101-FD-3	01-00793	2.08E-03	2.67	1.65E-5	1.65E-03	6.10E-1
AP101-FD-4						
AP101-FD-5						
AP101-FD-6	01-00794	1.61E-03	2.73	1.28E-5	1.28E-03	5.18E-1
AP101-FD-7						
AP101-FD-8						
AP101-FD-9						
AP101-FD-10						

DI Rinse

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	% C/Co	Ratio analytical/ lab GEA C/Co
AP101-FDI-1						
AP101-FDI-2	01-00795	2.05E-03	2.56	1.63E-5	1.63E-03	5.23E-1
AP101-FDI-3						
AP101-FDI-4						
AP101-FDI-5	01-00796	1.82E-03	2.67	1.45E-5	1.45E-03	6.11E-1
AP101-FDI-6						
AP101-FDI-7						
AP101-FDI-8						
AP101-FDI-9						

AP-101 run starting 4/24/01.

Lead column, eluting phase

Sample ID	CMC ID	Cs-137, µCi/mL	error, %	C/Co	Ratio analytical/ lab GEA C/Co		total Cs-137, µCi	
AP101L-E1	01-00797	1.21E-2	2.9	9.58E-5	1.41E+0		0.12	
AP101L-E2	01-00798	1.08E-2	2.98	8.57E-5	1.43E-2		0.11	
AP101L-E3	01-00799	2.66E+3	2	2.11E+1	6.28E-1		24337	
AP101L-E4	01-00800	8.11E+3	2	6.43E+1	6.37E-1		72484	
AP101L-E5	01-00801	4.75E+3	2	3.77E+1	7.39E-1		41687	
AP101L-E6	01-00802	3.61E+2	2	2.87E+0	7.07E-1		3423	
AP101L-E7	01-00879	4.94E+1	1.36	3.92E-1	7.38E-1		463	
AP101L-E8	01-00803	7.83E+0	2.65	6.21E-2	7.23E-1		79.48	
AP101L-E9	01-00804	3.66E+0	2.5	2.91E-2	9.95E-1		36.39	
AP101L-E10	01-00805	2.01E+0	2.52	1.60E-2	7.27E-1		16.79	
AP101L-E11	01-00806	1.32E+0	2.48	1.04E-2	6.41E-1		12.15	
AP101L-E12								
AP101L-E13								
AP101L-E14								
AP101L-E15	01-00807	5.50E-1	2.49	4.37E-3	7.21E-1		5.24	
AP101L-E16								
AP101L-E17								
AP101L-E18	01-00808	3.76E-1	2.59	2.98E-3	7.45E-1		3.60	
AP101L-E19								
AP101L-E20								
AP101L-E21	01-00809	3.59E-1	2.43	2.85E-3	6.63E-1		3.47	
AP101L-E22						sum	1.43E+5	
AP-101L-E-comp	01-1842	7.97E+2	2	6.33E+0	NA		1.63E+5	from total beta analysis
AP101L-E-comp	01-1842	7.65E+02	2	6.07E+0	NA		1.56E+5	from Cs-137 report
							104	% recovery in Cs eluent

AP-101DF Lead column loading with Cs-137

Co = 126 uCi/mL

C/Co	C/Co midpoint	C, uCi/mL	mL processed	mL	area, uCi
2.43E-06		3.06E-04	66.50	66.5	2.03E-02
2.01E-06	2.22E-06	2.80E-04	147.60	81.10	2.27E-02
1.53E-06	1.77E-06	2.23E-04	248.50	100.90	2.25E-02
1.72E-06	1.62E-06	2.05E-04	344.20	95.69	1.96E-02
1.63E-06	1.68E-06	2.11E-04	439.88	95.68	2.02E-02
1.44E-06	1.53E-06	1.93E-04	538.68	98.81	1.91E-02
3.48E-06	2.46E-06	3.10E-04	635.77	97.09	3.01E-02
1.35E-05	8.48E-06	1.07E-03	732.71	96.94	1.04E-01
5.5E-05	3.42E-05	4.31E-03	832.92	100.21	4.32E-01
0.000207	1.31E-04	1.65E-02	932.78	99.86	1.65E+00
0.000664	4.35E-04	5.49E-02	1032.78	100.01	5.49E+00
0.001649	1.16E-03	1.46E-01	1130.87	98.09	1.43E+01
0.001929	1.79E-03	2.25E-01	1187.71	56.84	1.28E+01

3.49E+01 uCi Cs-137 loaded onto lag column
2.33E-02 % loaded onto the lag column

Total Cs loaded 1.50E+05 uCi Cs-137

Appendix F

AP-101DF Column Processing Analytical Results

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

Project / WP#: 42365 ~~42040~~ / F22626
ASR#: 6031
Client: M. Urie
Total Samples: 1

	From	To
RPL#:	01-00520	01-00520
Client ID:	AP-101 DF *	AP-101 DF *

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

Analyst: D.R. Sanders

Analysis Date (File): 03-08-01 (A0652)

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)

Jerry Wagner 7-9-01
Reviewed by

7-12-01
Concur

One sample, "AP-101-DF A" (RPL ID# 01-00520), was processed by 325 Shielded Analytical Laboratory (SAL) using PNL- ALO-128 acid digestion of the diluted feed material. Approximately 1 mL of the diluted feed material (density 1.258 g/mL) prepared in duplicate; duplicate digestion process blanks (reagents only) and a blank-spike were processed and analyzed by ICPAES.

ICPAES results are reported as μg/mL and have been corrected for dilution resulting from acid digestion of the supernatant material. Sample material was transferred to digestion vessels using a 1 mL pipette then weighed. Sample volume was determined by dividing sample weight by sample density. After digestion, samples were diluted to a final volume of approximately 26 mL.

Analytes of interest and MRQ's listed in Table 1, Supernatant Analyses of Special Instructions include the following: Al, Ba, Ca, Cd, Cr, Fe, K, La, Mg, Na, Ni, P, Pb, and U. Detection limits, adjusted for sample preparation dilution and analytical dilution, exceeded MRQ's for the nearly all analytes listed in Table 1 except: Fe, Mg, P, and Pb. A 5-fold analytical dilution resulted in the poor detection limit. Concentration of the following analytes was above MRQ: Al, Cr, K, and Na. The remaining analytes requested were below MRQ.

See attached ICPAES Data Report for final results.

Quality control check-standard results met tolerance requirements for all analytes of interest reported except where noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan.

Five fold serial dilution:

All analytes of interest were within tolerance limit of 10% after correcting for dilution except potassium. Potassium differed by 18%. Other analytes such as aluminum, chromium, and phosphorous differed by less than 9%. No explanation can be given for the discrepancy.

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 20% RPD for all analytes of interest greater than EQL.

Post-Spiked Samples (Group A):

All analytes of interest were recovered within tolerance of 75% to 125% post-spike recovery except for potassium. Recovery for potassium was about 57%. The concentration of potassium spike added was about 1/5th of the sample concentration tested.

Post-Spiked Samples (Group B):

All analytes of interest were recovered within tolerance of 75% to 125%.

Blank Spike:

All analytes of interest were recovered within tolerance of 80% to 120% except silver. Silver recovery was low (about 22%). Low recovery may be due to the amount of hydrochloric acid used to process the sample (or from chloride present in the original sample) causing silver to precipitate. Silver was not detected in any of the samples.

Matrix Spiked Sample:

All analytes of interest were recovered within tolerance of 75% to 125% except silver. Silver recovery was low (about 22%). Low recovery may be due to the amount of hydrochloric acid used to process the sample (or from chloride present in the original sample) causing silver to precipitate. Silver was not detected in any of the samples.

Quality Control Check Standards:

Concentration of all analytes of interest is within tolerance limit of ± 10% accuracy in the check standards: QC_MCVA, QC_MCVB, and QC_SSTMCV with the following exceptions. Sodium and potassium measurements of QC_MCVA were slightly higher than 10% (11% to 17% for potassium and 11% to 14% for sodium). In QC_SSTMCV potassium (15% & 18% high) and sodium (15% & 16%) were high. The high concentration of these two analytes from the samples may have resulted in analyte carry-over in the instrument spray chamber resulting in higher than expected results.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except for one of two measurements of potassium. One of the two potassium measurements was 13% high. All other analytes measured were within tolerance.

Process Blank:

Concentration of all analytes of interest is within tolerance limit of ≤EQL or less than ≤5% of the sample.

Laboratory Control Standard (LCS):

A blank-spike containing analytes of interest was prepared and analyzed instead of an LCS.

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.

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

	Multiplier=	27.0	27.3	136.8	135.4	
	RPL/LAB #=	01-520-PB1	01-520-PB2	01-520 @5	01-520-DUP @5	
	Client ID=	Process Blank	Process Blank	AP-101 DF A	AP-101 DF B	
Det. Limit	Run Date=	3/8/2001	3/8/2001	3/8/2001	3/8/2001	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	
0.025	Ag	--	--	--	--	--
0.060	Al	[4.7]	[4.7]	6,930	7,030	--
0.250	As	--	--	--	--	--
0.050	B	47.3	42.6	[54]	[56]	--
0.010	Ba	--	--	--	--	--
0.010	Be	--	--	--	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	[47]	[210]	[210]	--
0.015	Cd	--	--	--	--	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	--	151	153	--
0.025	Cu	--	--	--	--	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	[1.2]	[0.73]	[3.8]	--	--
2.000	K	--	--	29,800	29,700	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	--	[14]	[14]	--
0.150	Na	76.5	61.2	113,000	115,000	--
0.100	Nd	--	--	--	--	--
0.030	Ni	[2.2]	[1.7]	[4.4]	--	--
0.100	P	--	--	381	389	--
0.100	Pb	--	--	--	--	--
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	[120]	[130]	[420]	[300]	--
1.500	Sn	--	--	--	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	U	--	--	--	--	--
0.050	V	--	--	--	--	--
2.000	W	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	[2.7]	[7.6]	[8.4]	--
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).

ASR-6031 from `A0652 I.Burgeson ASR-6057 (01-623 ... 01-626) and M.Urie ASR-6031 (01-520) ICP98 hi.XLS

7/9/2001 @ 5:22 PM

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

PO Box 999, Richland, WA 99352

Client:	WTP (M. Urie)	**Charge Code/Project:**	W57922 / 42365
RPL Numbers:	01-0520	**ASR Number:**	6031
Analyst:	MJ Steele	**Analysis Date:**	7/24, 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).

AP-101 Diluted Feed Results

RPL Number	Sample ID	TOC MDL µgC/mL	TOC µgC/mL	TC MDL µgC/mL	7/24/01 TC µgC/mL	TC MDL µgC/mL	8/23/01 TC µgC/mL	TIC (a) MDL µgC/mL	TIC (b) µgC/mL
01-00520 PB	Hot Cell Blank		138		23		58		n.d.
01-00520 A	AP101 DF-A	220	8,000	15	7,400	170	8,200	170	n.d.
01-00520 A Dup	AP101 DF-A	220	7,800	15	7,400	170	7,700	170	n.d.
	RPD		3%		0%		6%		
	RSD		2%		0%		4%		
01-00520 B	AP101 DF-B	220	8,100	30	7,500	170	7,900	170	n.d.
01-00520 B Dup	AP101-DF-B	220	7,900	30	7,600	170	7,800	170	n.d.
	RPD		3%		1%		1%		
	RSD		2%		1%		1%		
01-00520 B MS	AP101-DF-B		96%		101%		101%		
BS/LCS	Blank Spike		99%		99%		102%		

(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 6031 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 Ricca, JT Baker, Aldrich, Sigma, Mallinckrodt and VWR. The identification of the standards and their Chemical Management System (CMS) numbers and expiration dates 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 each of the three analysis days was 92%, 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 average blank of 15 µC. The TC determination produced average blanks of 4 and 54 µC. The 54 µC blank level is unusually high; however, the reproducibility of the blank was reasonably good (i.e., 49 to 59 µC).

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 client's Test Specification for both the TC and TOC analysis of the supernatant and solids.

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%). Also, the relative standard deviations (RSD) meet the acceptance criteria of <15% established by the client's Test Specification.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. At a TOC recovery of 96% and a TC recovery of 101%, the matrix spike demonstrates recoveries well within the acceptance criteria of 75% to 125%.

Furnace Results Compared to Hot Persulfate Results

		TIC		TOC		TC		
RPL Number	Sample ID	Persul. µgC/mL	Furn. (a) µgC/mL	Persul. µgC/mL	Furn. µgC/mL	Persul. (b) µgC/mL	Furn. 7/24 µgC/mL	Furn. 8/23 µgC/mL
01-00520A	AP101 DF-A	5,350	nd	1,650	8,000	6,990	7,400	8,200
01-00520A Dup	AP101 DF-A	5,370	nd	1,650	7,800	7,010	7,400	7,700
01-00520B	AP101 DF-B	5,450	nd	1,630	8,100	7,080	7,500	7,900
01-00520B Dup	AP101-DF-B	5,240	nd	1,610	7,900	6,850	7,600	7,800

'nd' = not detected
Persul. = Results from hot persulfate method
Furn. = Results from furnace combustion method – 750 °C for TOC and 1000 °C for TC
(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 carbonate, is being combusted at 750 °C (as TOC) with the furnace method. No organic analyses were performed as part of the AP-101 diluted feed analyses. However, TOC from acetate, formate, oxalate, and citrate analysis of the AP-101 as-received material is about 1500 µgC/mL. Although there may be more organic compounds present than were analyzed for on the AP-101 as-received material, the sum from these major organic species more closely fits the hot persulfate results than the furnace results.

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: MW Urie **Date** 27 Nov 01

Review/Approval by: MJ Steele **Date** 12/4/01

Raw Data Calculation/Archive Information: File: ASR 6019L&S 6025L 6031L 6107S.xls

Battelle PNWD
PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 3

Project / WP#: 42040 / W57922
ASR#: 6031
Client: Mike Urie
Total Samples: 1

RPL #	Client ID
01-00520	AP-101-DF A

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: 3/27/01, 4/4/01, 4/12/01

Analysis Files: Experiment - 27MAR01, 4APR01, 12APR01
Procedure - 010327a, 010404a, 010412a
Element Menu - CsRbTc, actinides

Revised Report Date: 3 February 2004

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

CmP Thomas — 3 Feb04
Revised By — Date

3Feb04
Reviewed By — Date

Revision	**Effective Date**	**Description of Change**
1	August 2001	Format of the narrative report was redone for consistency with reports associated with other analytical techniques within the ASO.
2	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).
3	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).

One sample, AP-101 Diluted Feed submitted for analysis, was analyzed on a radioactive-material-contained ICP/MS for the requested analytes.

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 laboratory dilutions performed on the sample during analysis. Two preparation blanks, a blank spike, duplicate, and matrix spike was submitted with the samples. In addition, replicate and post spike (PS) analyses were also performed.

2. Quality Control

Duplicate (DUP). The duplicate and replicate analyses met the QC criterion of ± 20%for the Cs, Rb, and Tc analysis. RPDs were not calculated for the actinides since their concentration was below detection level.

Matrix Spike (MS). MS and PS recoveries met the QC criterion of 70-130% for the Cs, Rb, and Tc analysis. The actinides did not appear to be spiked into the sample, however the PS met the MS QC criterion of 75-125%.

Preparation Blank (PB). The PBs were below detection level for all analytes and met the QC criterion.

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 criterion of less than the estimated quantitation limit (< EQL) was met.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). The ICV/CCV standards met the QC criteria of ± 10% for the Cs, Tc, and Rb analysis. Some of the QC criteria were not met during the actinide analysis of the PBs: Pu-239 (87%), Pu-240 (82%, 81%), and Pu/Am-241 (89.8%, 89.8%). Some of the QC criteria were not met during the actinide analysis of the samples: Np-237 (113%), Pu-240 (115%, 83%, 83%), and Pu/Am-241 (112%, 116%)

Internal Standard (IS). The ISs met the QC criteria 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 AMU-241 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 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%.

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Project No. 42040

Internal Distribution
File/LB

Date May 3, 2001

To Mike Urie

From LMP Thomas 5.3.01

Subject ICPMS Analysis of ASR 6031 samples
{ALO# 01-(00520)}

Pursuant to ASR 6031, the samples submitted for analysis were analyzed by ICPMS for the requested analytes. The results are tabulated on the attached report forms. The concentrations are reported in μCi/ml of original sample for ^{237}Np, ^{239}Pu, ^{240}Pu and ^{99}Tc; ~μg/ml of original sample for Cs, Rb and ^{241}Pu/^{241}Am.

Standards
Dilutions of Isotope Products standards for ^{237}Np and ^{241}Am, an Amersham ^{99}Tc standard, an NIST $^{239/240}$Pu standard, and CPI single element standards for Cs and Rb were used to generate the calibration curves. Independent standards of each analyte were used for the independent calibration verification (ICV) standards. The analyses of the CCV standards for the Cs, Rb, and ^{99}Tc analysis were within ±10%. The analyses of the CCV standards for the ^{237}Np, ^{239}Pu, ^{240}Pu and ^{241}Pu/^{241}Am analysis were outside ±10% (but within ±20%). However, since there were non-detectable levels of the analyte in the samples, no reruns were performed. The reagent blank and diluent for the standards and samples was a 1% high purity nitric acid solution.

Analysis
Inter-laboratory (AIA) spikes and duplicates were prepared and analyzed in addition to the duplicate and spike samples submitted. These are denoted in lower case as 'duplicate' and 'spike' (vs. DUP and MS for the samples submitted). The inter-laboratory spikes were within ±25% recovery and inter-laboratory duplicates were within ±5% RPD.

Technetium Isotopic Analysis. The ^{99}Tc values reported assume that the ruthenium present is exclusively fission product ruthenium and therefore does not have an isotope at m/z 99. The isotopic analysis observed for Ru is not a natural distribution and is consistent with previous tank waste analysis.

If you have questions regarding this analysis please call me at 376-5936 or Tom Farmer at 372-0700.

Client: Mike Urie
WP/Project: F22630/42040
ASR: 6031
Log-In: 01-00520
Report Date: 5/3/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: 5.3-01

Analyst: 5/3/01

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Unless otherwise specified; the results are reported in µg analyte/ml (ppm) of the original sample for Cs, Rb and µCi analyte/ml of the original sample for ^{99}Tc

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µg/ml	Rb µg/ml	±	1SD	MDL µCi/ml	^{99}Tc µCi/ml	±	1SD	MDL µg/ml	Cs µg/ml	±	1SD
	1%HNO3		ICB	<	3.2E+00			<	6.9E-08			<	2.5E-06		
	1%HNO3		CCB1	<	3.5E+00			<	7.7E-08			<	2.7E-06		
	1%HNO3		CCB2	<	3.6E+00			<	7.8E-08			<	2.8E-06		
	1%HNO3		CCB3	<	2.9E+00			<	6.4E-08			<	2.3E-06		
	1%HNO3		CCB4	<	3.0E+00			<	6.5E-08			<	2.3E-06		
	ICV		ICV1		2.0E-04	±	2.1E-06		3.3E-06	±	2.7E-08		1.9E-04	±	1.3E-06
	CCV		CCV1a		2.0E-04	±	3.1E-07		3.2E-06	±	3.7E-08		1.9E-04	±	2.6E-06
	CCV		CCV2a		2.1E-04	±	5.4E-06		3.3E-06	±	5.3E-08		1.9E-04	±	2.1E-06
	CCV		CCV3a		2.1E-04	±	2.7E-06		3.0E-06	±	4.4E-08		1.8E-04	±	6.4E-07
	CCV		CCV4a		2.1E-04	±	2.2E-06		3.0E-06	±	8.0E-08		1.8E-04	±	2.3E-06
True Value					**2.00E-04**				**3.39E-06**				**2.00E-04**		
01-00520-PB1	01-00520-PB1	PROCESS BLANK	PB3	1.06E-01 <	1.06E-01			2.32E-03 <	2.32E-03			8.22E-02 <	8.22E-02		
01-00520-PB1	01-00520-PB1 + spike	PROCESS BLANK	PBS2	9.65E-02	5.58E+00	±	4.63E-02	2.10E-03	8.94E-02	±	1.09E-03	7.46E-02	5.20E+00	±	6.25E-02
Spike Concentration					2.00E-04				3.18E-06				1.87E-04		
Spike Recovery					**100%**				**94%**				**94%**		
01-00520-PB2	01-00520-PB2	PROCESS BLANK	PB4	9.71E-02 <	9.71E-02			2.12E-03 <	2.12E-03			7.50E-02 <	7.50E-02		
01-00520-BS2	01-00520-BS2	BLANK SPIKE	BS2	8.75E-01	2.07E+01	±	5.26E-01	1.91E-02 <	1.91E-02			6.76E-01	2.77E+01	±	3.73E-01
Spike Concentration					7.78E-05								1.04E-04		
Spike Recovery					**103%**								**92%**		
01-00520	01-00520	AP-101-DF A	Sample9	7.39E-02	3.99E+00	±	3.83E-02	1.61E-03	4.00E-02	±	9.62E-04	5.71E-02	2.99E+00	±	1.47E-02
01-00520	01-00520 duplicate	AP-101-DF A	Sample11	7.59E-02	4.00E+00	±	2.50E-02	1.65E-03	3.86E-02	±	5.84E-04	5.87E-02	2.93E+00	±	3.31E-02
01-00520	01-00520 + spike	AP-101-DF A	Sample13	7.67E-02	9.61E+00	±	8.36E-02	1.67E-03	1.15E-01	±	1.72E-03	5.93E-02	7.68E+00	±	6.53E-02
Spike Concentration					2.05E-04				2.75E-06				1.71E-04		
Spike Recovery					**103%**				**81%**				**86%**		
01-00520-DUP	01-00520-DUP	AP-101-DF B	Sample10	7.34E-02	3.88E+00	±	6.23E-02	1.60E-03	3.83E-02	±	1.14E-03	5.67E-02	2.92E+00	±	3.61E-02
01-00520-MS2	01-00520-MS2	AP-101-DF B	Sample12	8.04E-01	2.45E+01	±	3.96E-01	1.75E-02	3.93E-02	±	4.51E-04	6.21E-01	2.89E+01	±	2.64E-02
Spike Concentration					9.25E-05								1.09E-04		
Spike Recovery					**123%**								**96%**		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 3/27/01
Instrument Filenames: Experiment (27MAR01), Procedure(010327a), Element Menu (CsRbTc)

Client: Mike Urie
WP/Project: F22630/42040
ASR: 6031
Log-In: 01-00520
Report Date: 5/2/01

Analytical Chemistry Group, Battelle PNNL
ICP/MS Analysis Data Report

Reveiwed by: tmPJ 5.3.01

Analyst: 5/3/01

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Unless otherwise specified; the results are reported in µg analyte/ml (ppm) of the original sample for ^{241}Pu/^{241}Am and µCi analyte/ml of the original sample for all others.

Log-In Number	Sample ID	Client ID	ICP/MS ID	MDL µCi/ml	^{237}Np µCi/ml	±	1SD	MDL µCi/ml	^{239}Pu µCi/ml	±	1SD	MDL µCi/ml	^{240}Pu µCi/ml	±	1SD	MDL µg/ml	^{241}Pu/^{241}Am µg/ml	±	1SD
	1%HNO3 (Blanks)		ICB		< 2.53E-10				< 6.05E-08				< 4.37E-08				< 2.46E-07		
	1%HNO3 (Blanks)		CCB1		< 3.12E-10				< 7.47E-08				< 5.40E-08				< 3.04E-07		
	1%HNO3 (Blanks)		CCB2		< 2.19E-10				< 5.24E-08				< 3.79E-08				< 2.13E-07		
	1%HNO3 (Samples)		ICB		< 8.70E-10				< 1.70E-07				< 1.56E-07				< 5.77E-07		
	1%HNO3 (Samples)		CCB1		< 9.08E-10				< 1.78E-07				< 1.63E-07				< 6.02E-07		
	1%HNO3 (Samples)		CCB2		< 9.05E-10				< 1.77E-07				< 1.63E-07				< 6.00E-07		
	1%HNO3 (Samples)		CCB3		< 7.26E-10				< 1.42E-07				< 1.30E-07				< 4.82E-07		
	ICV (Blanks)		ICV		3.24E-08	±	5.70E-10		3.22E-06	±	1.94E-07		1.90E-07	±	1.55E-08		4.49E-05	±	1.01E-06
	CCV (Blanks)		CCV1		3.26E-08	±	1.00E-09		3.04E-06	±	4.15E-08		2.25E-07	±	2.75E-08		4.49E-05	±	1.74E-06
	CCV (Blanks)		CCV2		3.18E-08	±	6.00E-10		2.64E-06	±	6.37E-08		1.89E-07	±	3.50E-09		4.03E-05	±	1.12E-06
	CCV (Blanks)		CCV3		3.77E-08	±	1.25E-09		3.17E-06	±	5.08E-08		2.19E-07	±	1.28E-08		4.78E-05	±	1.50E-06
True Value					**3.52E-08**				**3.04E-06**				**2.33E-07**				**5.00E-05**		
	ICV (Samples)		ICV		3.70E-08	±	3.93E-10		2.97E-06	±	2.84E-08		2.34E-07	±	2.92E-09		5.17E-05	±	9.44E-07
	CCV (Samples)		CCV1		3.55E-08	±	4.04E-10		3.19E-06	±	6.69E-08		2.68E-07	±	4.28E-08		5.10E-05	±	9.97E-07
	CCV (Samples)		CCV2		3.86E-08	±	2.46E-10		2.89E-06	±	1.66E-07		1.94E-07	±	5.76E-09		5.59E-05	±	1.12E-06
	CCV (Samples)		CCV3		3.97E-08	±	1.33E-09		2.89E-06	±	2.50E-08		1.94E-07	±	3.96E-08		5.81E-05	±	2.78E-06
True Value					**3.52E-08**				**3.04E-06**				**2.33E-07**				**5.00E-05**		
	20ppb U030 uranium	(Blanks)	U030a		< 2.45E-10				< 5.86E-08				< 4.23E-08				< 2.38E-07		
	10ppb 4321B uranium	(Samples)	4321b		< 1.11E-09				< 2.17E-07				< 1.99E-07				< 7.34E-07		
	30ppb 4321B uranium	(Blanks)	4321b1		< 2.79E-10				7.82E-08	±	2.87E-09		< 4.83E-08				< 2.72E-07		
	30ppb 4321B uranium	(Blanks)	4321b2		< 2.27E-10				< 5.43E-08				< 3.92E-08				< 2.21E-07		
01-00520-PB1	01-00520-PB1	PROCESS BLANK	PB7	1.33E-06	< 1.33E-06			3.18E-04	< 3.18E-04			2.29E-04	< 2.29E-04			1.29E-03	< 1.29E-03		
01-00520-PB1	01-00520-PB1 + spike	PROCESS BLANK	PBS4	1.49E-06	3.76E-04	±	9.46E-06	3.56E-04	3.20E-02	±	1.76E-03	2.57E-04	2.24E-03	±	2.37-04	1.45E-03	4.90E-01	±	2.86E-02
Spike Concentration					6.85E-08				5.79E-06				3.68E-07				8.93E-05		
Spike Recovery					**97%**				**93%**				**79%**				**89%**		
01-00520-PB2	01-00520-PB2	PROCESS BLANK	PB8	1.31E-06	< 1.31E-06			3.13E-04	< 3.13E-04			2.26E-04	< 2.26E-04			1.27E-03	< 1.27E-03		
01-00520-BS2	01-00520-BS2	BLANK SPIKE	BS4	2.40E-06	< 2.40E-06			5.75E-04	< 5.75E-04			4.16E-04	< 4.16E-04			2.34E-03	< 2.34E-03		
01-00520	01-00520	AP-101-DF A	Sample9	3.71E-06	< 3.71E-06			7.26E-04	< 7.26E-04			6.67E-04	< 6.67E-04			2.46E-03	< 2.46E-03		
01-00520	01-00520 duplicate	AP-101-DF A	Sample11	3.60E-06	< 3.60E-06			7.05E-04	< 7.05E-04			6.47E-04	< 6.47E-04			2.39E-03	< 2.39E-03		
01-00520	01-00520 + spike	AP-101-DF A	Sample19	3.63E-06	1.90E-04	±	7.68E-06	7.11E-04	2.15E-02	±	1.37E-04	6.53E-04	2.12E-03	±	1.17E-04	2.41E-03	2.69E-01	±	3.21E-03
Spike Concentration					3.41E-08				3.80E-06				2.66E-07				4.87E-05		
Spike Recovery					**97%**				**122%**				**114%**				**97%**		
01-00520-DUP	01-00520-DUP	AP-101-DF B	Sample10	3.91E-06	< 3.91E-06			7.65E-04	< 7.65E-04			7.03E-04	< 7.03E-04			2.59E-03	< 2.59E-03		
01-00520-MS2	01-00520-MS2	AP-101-DF B	Sample12	3.74E-06	< 3.74E-06			7.31E-04	< 7.31E-04			6.71E-04	< 6.71E-04			2.48E-03	< 2.48E-03		

Analytical Equipment: ICP/MS VG WB36913
Balance: Mettler 512-06-01-014
Procedure: PNL-ALO-280
Analysis Date: 4/4/01, 4/12/01
Instrument Filenames: Experiment (4APR01,12APR01), Procedure(010404a, 010412a), Element Menu (actinides)

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

Client: M. Urie

Charge Code/Project: F22630 / 42040
New W57922 / 42365

RPL Numbers: 01-00520
Analyst: MJ Steele

ASR Number: 6031
Analysis Date: July 19/20, 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 Number	Sample ID	TIC MDL ugC/mL	TIC Results ugC/mL	TIC RPD	TOC MDL ugC/mL	TOC Results ugC/mL	TOC RPD	TC Results ugC/mL	TC RPD
01-0520 PB	Hot Cell Blank	7	10		18	<18		10	
01-0520 A	AP101-DF A	13	5,350		36	1,650		6,990	
01-0520 A Dup	AP101-DF A	13	5,370	0%	36	1,650	0%	7,010	0%
01-0520 B	AP101-DF B	13	5,450		36	1,630		7,080	
01-0520 B Dup	AP101-DF B	13	5,240	4%	36	1,610	2%	6,850	3%
01-0520 B MS	Recovery		97%			92%		95%	
Blank Spike/LCS	Recovery		102%			105%			
Blank Spike/LCS	Recovery		103%			102%			

The TOC/TIC analyses of the sample submitted under ASRs 6031 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 numbers for the standards are included 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 as well as against the Table 3 "Quality Control Parameters for Liquid Samples" provided with the ASR.

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyze each day that the AP-101 supernatant samples were analyzed. The LCS/BSs 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 spike for the AP-101 DF B sample demonstrates 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 meet the acceptance criteria of <20% RPD for the QA Plan and <15% RSD from Table 3. It should be noted that AP-101 DF A and AP-101 DF B are identical samples but have been aliquotted from different bottles (i.e., Bottles DF A and DF B). The RPD between the averages is <2% for TIC, TOC, and TC.

Hot Cell Trip Blank: Water resident in the SAL hot cells was sub-sampled, handled, and transferred to the laboratory for TOC/TIC analysis. This 'trip' blank showed a very small TIC contamination level (i.e., about 2x the estimated method detection limit) and no TOC.

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: MW Urie **Date** 10-02-01

Review/Approval by: MJ Steele **Date** 10-20-01

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

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

Client:	M Urie	Charge Code/Project:	F22626/42040
ASR Number:	6031	Sample Receipt Date:	00/29/2001
Sample Prep Date:	N/A	Sample Analysis Date:	04/03-04/2001
Analyst:	MJ Steele	**Revision Date:**	**07/23/2001**

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.

Raw data, standards information, preparation benchsheets filed with ASR 6019 Urie: Project 42365

Sample 01-00520 Final Results: REVISION 1

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	0.28	0.28	0.55	0.28	0.55	0.55	0.55	0.55
01-00520 PB	Hot Cell Blank	< 0.28	< 0.28	< 0.55	< 0.28	< 0.55	< 0.55	< 0.55	< 0.55
	EQL	480	480	950	480	1,900	950	1,900	950
01-00520	AP-101-DF A	2,300	1,400	31,200	< 480	103,400	< 950	4,660	< 950
01-00520 Dup	AP-101-DF B	2,300	1,500	33,800	< 480	104,700	< 950	4,600	< 950
	RPD	0	5	6	n/a	1	n/a	1	n/a
01-00520 MS %Rec	AP-101-DF MS	97	93	102	97	102	97	91	105
LCS %Rec	60/160 Spike Solution	107	105	105	106	101	104	102	107

The fluoride results should be considered the upper bound concentration for the fluoride. Significant peak distortion of the fluoride peak suggests the presence of co-eluting anion(s), possibly formate or acetate.

The sample was prepared for ion chromatography anion analysis by dilution at 1900-fold and 3800-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 1900-fold. The estimated quantitation limits (EQL) which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in the table.

Q.C. Comments:

Duplicates: The duplicates were sub-sampled in the hot cells, the sample was aliquotted from container labeled AP-101-DF A and the duplicate from container labeled AP-101-DF B. The duplicate relative percent difference (RPD) meets the acceptance criteria of <20%.

Matrix Spike (HCV 010328): A matrix spike was prepared from the sample and all anion recoveries were within the 75% to 125% recovery acceptance criteria.

Laboratory Control Sample-LSC/BS (LSC 010328): 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 80% to 120% acceptance criteria.

System Blank/Processing Blanks: Eighteen system blanks were analyzed throughout the analysis runs. No anions were detected in the system blanks above the estimate quantitation level. Also, no anions were detected in the hot cell blank (01-00520 PB).

Quality Control Calibration Verification Check Standards: Nine mid-range verification standards were analyzed throughout the analysis runs. Except for one chloride measurement and two oxalate measurement, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The chloride measurement that failed produced a recovery of 89% and the oxalate measurements produced recoveries of 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: ________________ Date 7-23-01

Review/Approval: MJ Steele Date 7-23-01

Archive Information:

Files: ASR 6031 Urie.doc ASR 6014 6019 6025 6031 6057.xls

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Project No. 42040

Internal Distribution
File/LB

Date June 29, 2001

To M. W. Urie

From L. R. Greenwood LRGreenwood

Subject Radiochemical Analyses for AP-101 – ASR 6031,32

Samples of the supernates from tank AP-101 were analyzed for gamma emitters, ^{90}Sr, total alpha, U, Pu, Am/Cm, and ^{99}Tc according to ASRs 6031 and 6032. The samples were acid digested in the hot cells according to procedures PNL-ALO-128 and -129 and aliquots were delivered to the laboratory for analysis. The attached reports list measured analyte activities in the original sample material in units of μCi/ml for the supernates. 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.

The hot cell sample preparations were performed in two separate batches, as indicated by the horizontal lines separating the results in the attached tables. However, all of the samples were initially run together in one batch in the laboratory. The hot cell process blanks prepared with each batch of samples showed significant alpha contamination, especially for the AP-101 samples, as is discussed below for each analysis. The hot cell blanks also showed high contamination with ^{90}Sr for sample AP-101-DF-A and -B. Consequently, this sample was reprepared in the hot cell and reanalyzed for ^{90}Sr and the actinides. The reanalyses showed some contamination for ^{90}Sr; however, no contamination was seen for Pu and Am/Cm.

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. Initially, the samples were diluted prior to gamma analysis. However, in order to meet the requested detection limits, direct aliquots of the hot cell preparations were counted for periods of 4 to 14 hours. All of the samples showed the presence of significant ^{137}Cs activity. The AP-101 samples also showed ^{60}Co and ^{134}Cs. The MRQ values for extended counting time GEA were met in all cases except for the opportunistic analyte ^{241}Am for sample AP-101-AR, where the detection limit was twice the MRQ value of 1.E-2 μCi/ml. For sample AP-101-AR we detected the opportunistic analyte ^{134}Cs; however we did not see any of the others. Detection limits are listed in the tables except for ^{106}Ru/Rh (< 8.E-2 μCi/ml) and ^{125}Sb (< 5.E-2 μCi/ml). All of the hot cell process blanks showed the presence of ^{137}Cs and some of them also showed ^{134}Cs. However, in all cases, the activities in the blanks were negligible with respect to the samples. Sample duplicates showed excellent repeatability with RPD or RSD values less than 10%.

Total Alpha

The total alpha activity was determined by evaporating small aliquots of the acid-digested samples onto planchets according to RPG-CMC-4001. The samples were then counted on Ludlum detectors according to RPG-CMC-408. The sums of the individual alpha emitters, as discussed below, agree well with the total alpha data indicating minimal losses due to alpha self-absorption. The hot cell process blank did not show any alpha contamination. It should be noted that the sum of the alpha emitters is well below the MRQ value of 0.23 µCi/ml for this sample. The LCS and matrix spike recoveries with ^{239}Pu were 112% and 79%, respectively. No alpha contamination was detected in the laboratory blank. RPD value was 13%.

Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed 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. Plutonium recovery was traced with ^{242}Pu. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. Both the plutonium and americium radiochemical yields were excellent, averaging about 95%. Neither Pu, Am nor Cm were detected in the laboratory preparation blank. The hot cell process blanks and laboratory blanks did not show any significant alpha contamination for Pu, Am, or Cm.

The LCS and matrix spike recoveries were 98 to 113% for both Pu and Am. RPD and RSD values were generally quite good except for ^{243,244}Cm in sample AP-101-DF, where the counting uncertainties are quite high.

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). Two of the separated fractions contained a small amount of ^{137}Cs and a correction to the beta count rate was applied for these samples. However, this correction was negligible with respect to the activity in the samples. No ^{90}Sr was found in the laboratory preparation blank. All of the hot cell process blanks showed ^{90}Sr activities. However, the levels are not significant except for sample AP-101-DF where the initial hot cell contamination was about 15% of the sample activity. Due to the hot cell contamination, this sample was reanalyzed. However, the reanalysis showed about the same level of contamination as the original analysis, although the activities in the samples were lower for the reanalyses. It should be noted that the level of ^{90}Sr in the AP-101-DF sample is about half the requested MRQ value of 0.15 uCi/ml. The RPD value was 10%, indicating good sample reproducibility. The LCS and matrix spike recoveries were 105% and 95%, respectively, for the reanalysis of sample AP-101-DF.

Total Uranium

Total uranium was measured according to procedure PNNL-ALO-4014 using Kinetic Phosphoresence Analysis (KPA). Sample solutions from the hot cell were evaporated dry with nitric acid, then re-dissolved in dilute nitric acid for uranium measurement. No uranium separation was done. The repeatability of duplicate samples is excellent with RPD values of 2%. Uranium was found in the hot cell blanks, but at a negligible concentration compared to the samples.

No instrument blanks had detectable uranium. All the instrument standards gave results within expected uncertainty. The instrument standards at 10 or more times the detection limit were all within 3% of the known concentration. The lowest standard, at 2.5 times the detection limit, has an inherently higher uncertainty.

Pertechnetate ^{99}Tc

The radiochemical ^{99}Tc determination was requested to measure only Tc in the +7 oxidation state (pertechnetate). To this end, all sample manipulations had to be non-oxidizing so as not to alter the original Tc oxidation state. Small aliquots from the as-received material (no digestion) were taken for analysis according to procedure PNL-ALO-432. This procedure normally requires the use of sodium dichromate addition to oxidize the Tc to the +7 oxidation state. The sodium dichromate addition was omitted and the procedure otherwise was performed as written. The separated fractions were then counted according to procedure RPG-CMC-408. The samples were also counted by liquid scintillation counting according to procedure PNL-ALO-474 to confirm that the beta energy spectra matched that of ^{99}Tc and that no other beta emitters were present. The LCS blank spike recovery of a 99Tc standard was 92%. However, a matrix spike gave a standard recovery of 248%. The reason for this high recovery is not known. Sample duplicates and lab replicates gave good agreement with RPD values less than 12%. The ^{99}Tc activities in the hot cell and laboratory blanks were negligible or non-detectable and well below the requested MRQ values.

Review.
C. Soderquist 6-29-01

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

01-0520

08/20/01

Client : Mike Urie

Cognizant Scientist: LRGreenwood Date : 8/20/01

Concur : T Trang-le Date : 8/23/01

PNL-ALO-476 (Sr-90)
PNL-ALO-450 (GEA)

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

ALO ID Client ID		Sr-90 Error %	Cs-134 Error %	Cs-137 Error %	Co-60 Error %	Sb-125 Error %	SnSb-126 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-0520PB1		1.05E-2		8.49E-3							
Process Blank		5%		4%							
	MDA	<4.E-4	<3.E-4	<3.E-4	<6.E-4	<6.E-4	<2.E-4	<3.E-4	<1.E-3	<8.E-4	<1.E-3
01-0520PB2				1.00E-2							
Process Blank				3%							
	MDA		<3.E-4	<3.E-4	<6.E-4	<6.E-4	<2.E-4	<3.E-4	<9.E-4	<8.E-4	<1.E-3
01-0520		6.79E-2	2.96E-2	1.27E+2	2.50E-3						
AP-101-DF A		4%	2%	2%	5%						
	MDA	<3.E-3	<1.E-3	<6.E-3	<2.E-4	<3.E-2	<1.E-2	<7.E-4	<1.E-3	<1.E-2	<1.E-2
01-0520 DUP		7.49E-2	2.77E-2	1.25E+2	2.53E-3						
AP-101-DF B		4%	2%	2%	5%						
	MDA	<4.E-3	<1.E-3	<6.E-3	<2.E-4	<2.E-2	<1.E-2	<7.E-4	<1.E-3	<1.E-2	<1.E-2
	RPD	10%	7%	2%	1%						
01-0521PB1				1.63E-3							
Process Blank				6%							
	MDA		<3.E-4	<2.E-4	<4.E-4	<4.E-4	<1.E-4	<1.E-4	<7.E-4	<5.E-4	<7.E-4
01-0521PB2				1.53E-3							
Process Blank				8%							
	MDA		<3.E-4	<2.E-4	<4.E-4	<4.E-4	<1.E-4	<1.E-4	<7.E-4	<5.E-4	<8.E-4
01-0521			3.52E-2	1.51E+2	3.01E-3						
AP-101-AR-1R			3%	2%	7%						
	MDA		<2.E-3	<1.E-2	<5.E-4	<4.E-2	<2.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
01-0521 DUP			3.40E-2	1.54E+2	3.17E-3						
AP-101-AR-2R			3%	2%	7%						
	MDA		<2.E-3	<1.E-2	<4.E-4	<4.E-2	<2.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
01-0521 TRIP			3.57E-2	1.59E+2	3.34E-3						
AP-101-AR-3R			3%	2%	7%						
	MDA		<2.E-3	<1.E-2	<4.E-4	<4.E-2	<2.E-2	<1.E-3	<2.E-3	<2.E-2	<2.E-2
	RSD		2%	3%	5%						
Sample spike		105%									
Blank spike		95%									
Blank		<3.E-4									

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

01-0520

08/20/01

Client : Mike Urie

Cognizant Scientist: LRGreenwood Date : 8/20/01

Concur : T Trang-le Date : 8/23/01

RPG-CMC-408 (Total Alpha)
PNL-ALO-417,496 (Pu, Am/Cm)

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

ALO ID Client ID		Alpha Error %	Pu-238 Error %	Pu-239/240 Error %	Am-241 Error %	Cm-243/244 Error %	Cm-242 Error %	Sum of Alpha Error %
01-0520PB Process Blank					9.91E-7 28%			9.91E-7 4%
	MDA	<2.E-4	<6E-7	<6E-7	<7E-7	<6E-7	<4E-7	<3E-6
01-0520 AP-101-DF A		2.03E-04 17%	1.51E-5 5%	1.10E-4 2%	1.37E-4 2%	2.89E-6 13%		2.65E-4 4%
	MDA	<9E-5	<4E-7	<5E-7	<6E-7	<4E-7	<4E-7	<2E-6
01-0520 DUP AP-101-DF B		2.31E-04 16%	1.29E-5 6%	1.10E-4 3%	1.39E-4 3%	1.34E-6 22%		2.63E-4 4%
	MDA	<9E-5	<8E-7	<7E-7	<5E-7	<5E-7	<4E-7	<3E-6
	RPD	13%	16%	0%	1%	73%		1%
Sample spike		79%		113%	99%			
Blank spike		112%		112%	98%			
Blank		<2.E-4	<9.E-7	<1.E-6	<2.E-6	<2.E-6	<1.E-6	

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

01-0520

08/20/01

Client : Mike Urie

Cognizant Scientist: LRGreenwood Date : 8/20/01

Concur : T Trang-le Date : 8/23/01

PNL-ALO-432 (Tc-99)

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

ALO ID Client ID		Pertechnetate Tc-99 Error %	MDA
01-0520 CB Process Blank		4.46E-6 15%	<2.E-6
01-0520 AP-101-DF A		3.24E-2 4%	<4.E-6
01-0520 DUP AP-101-DF B		3.67E-2 4%	<5.E-6
	RPD	12%	
Sample spike		248%	
Blank spike		92%	
Blank		7.89E-06 23%	

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

01-0520

08/27/01

Client : Mike Urie

Cognizant Scientist: LRGreenwood Date : 8/27/01

Concur : C. Soderquist Date : 8.27-01

RPG-CMC-4014 (Uranium KPA)

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

ALO ID Client ID	Uranium Error %	MDL
01-0520PB1 Process Blank	3.41E-1 2%	6.E-3
01-0520PB2 Process Blank	1.92E-1 2%	6.E-3
01-0520 AP-101-DF A	4.11E+1 4%	6.E-3
01-0520 DUP AP-101-DF B	4.02E+1 4%	6.E-3
RPD	2%	
01-0521PB1 Process Blank	3.30E-2 2%	6.E-3
01-0521PB2 Process Blank	2.28E-2 2%	6.E-3
01-0521 AP-101-AR-1R	4.87E+1 4%	6.E-3
01-0521Rep AP-101-AR-1R	4.95E+1 4%	6.E-3
01-0521 DUP AP-101-AR-2R	4.78E+1 4%	6.E-3
01-0521 TRIP AP-101-AR-3R	4.95E+01 4%	6.E-3
RSD	2%	
Blank	<6.E-3	

Instrument Standards, μg/mL	Observed	Expected	Spike Yield	
Blank 1	0.00E+0			
Blank 2	4.60E-7			
Blank 3	7.60E-7			
IV 5E-1	5.07E-1	5.02E-1	101%	
IV 5E-5	6.25E-5	5.07E-5	123%	(This standard is near the detection limit)
IV 5E-1	5.07E-1	5.02E-1	101%	
AA 5E-1	5.01E-1	5.05E-1	99%	
AA 5E-3	4.93E-3	5.03E-3	98%	

Sample solutions ranged from 8E-5 to 1.9E-1 μg/mL

Battelle

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Date: 03/21/01

Subject: Hydroxide Analyses for: AP-101 Diluted Feed

ASR: 6031

To: Mike Urie

From: Bob Swoboda RG Swoboda 3/22/01

Two sample replicates of the composite from tank **AP-101 (0100520)** were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. A 0.1186 N NaOH solution was prepared for use as a standard and sample spike and the titrant was a 0.2040 M HCl prepared solution. The sample and duplicate results and their replicate runs gave an average OH molarity of 1.94 with a standard deviation of <1%. This result is equivalent to 3.29E+4 ug/ml, well below the required MRQ value of 7.5E+4 ug/ml. The hydroxide standard recovery averaged 93% and a sample spike recovered at 97%. No hydroxide was detected in the cell blanks or a reagent blank. The second and third inflection points frequently associated with carbonate and bicarbonate, were detected in the samples at an average of 0.79 moles, with a standard deviation of 4%, and at 0.56 moles, with a standard deviation of 7%, respectively, for the quadruplicate measurements. 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.

Review: LRGreenwood 3/22/01

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

ASR # **6031**

WP# **k88426**

Hydroxide and Alkalinity Determination

Procedure: **PNL-ALO-228** Equip # **WB76843**

Analyst: Lori P. Darnell 3-22-01

Reviewer: 3/22/01

			Concentration, moles		
RPG #	**Client ID**		**First Point**	**Second Point**	**Third Point**
01-0520-CB	**Cell Blank**		**0**	**0**	**0**
01-0520-CB	**Cell Blank**	**Rep**	**0**	**0**	**0**
		RPD			
01-0520	**AP-101-DF A**		**1.90**	**0.78**	**0.55**
01-0520	**AP-101-DF A**	**Rep**	**1.91**	**0.83**	**0.54**
		RPD	**1%**	**7%**	**2%**
01-0520-DUP	**AP-101-DF B**		**1.97**	**0.77**	**0.53**
01-0520-DUP	**AP-101-DF B**	**Rep**	**1.96**	**0.78**	**0.60**
		RPD	**0.2%**	**1%**	**13%**
		Average (4)	**1.94**	**0.79**	**0.56**
Hydroxide concentration in ug/ml			**3.29E+4**		
Reag. Blk.3			**0**		
Standard 1			**93%**		
Standard 2			**93%**		
MS 01-0520	**Matrix spike**		**97%**		

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 ... ICPAES Data Report

Project / WP#:	42365 / W58168
ASR#:	6097
Client:	S. Fiskum
Total Samples:	1

	From	To
RPL#:	01-00810	
Client ID:	"AP101L-RGN-A"	
Sample Preparation: PNL-ALO-106/SRPL/ms (1 mL/20mL) **[0.25 M NaOH, 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): 07-16-01 (A0698)

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)

Jerry Wagner 8-22-01
Reviewed by

8/30/01
Concur

8/22/2001

One AP101 sample, **AP101L-RGN-A** (RPL# 01-00810), was processed by SRPL using PNL- ALO-106 acid digestion procedure. Sample was prepared using 1 mL of liquid material. After digestion the sample was diluted to a final volume of approximately 20mL. Sample density estimated by weighing two 1 mL aliquots was about 1.008 g/mL (See PNL-ALO-106 worksheet in supporting documents for additional sample preparation information).

Sodium was the specific analyte requested for analysis by ICPAES.

Please note: the expected Na concentration in the sample, according to the ASR for 0.25 M NaOH, should be about 5,750 µg-Na/mL. The concentration of Na found was only about 1,000 µg/mL.

The process blank appears to have a small amount of Al, B, Ba, Fe, Na, Ni, and Si present. Boron, Na, and Si may come from glassware used to digest the samples or from the digestion reagent (nitric acid). The Na concentration in the process blank is about 15% of that found in the samples.

See attached ICPAES Data Report for final results. ICPAES measurement results are reported in µg/mL and have been corrected for dilution resulting from sample processing. Analyte concentrations reported other than specifically requested are for information only.

Quality control check-standard results met tolerance requirements for the specific analyte of interest (Na) with exceptions noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan. LCS/BS, Matrix-spikes and duplicate were prepared with the samples and analyzed. Matrix-spike was prepared using 1mL respectively of a custom multi-element solution "INT-QC-MCVA-1B" (CMS# 186946) per 20 mL sample digestate volume. LCS/blank-spiked sample was prepared using 1mL respectively of a custom multi-element solution "INT-QC-MCVA-1B" (CMS# 186946) per 25 mL sample digestate volume.

Five fold serial dilution:

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

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 15% RPD for analyte of interest.

Post-Spiked Samples (Group A):

All post-spiked analytes in samples tested were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

All post-spiked analytes in samples tested were recovered within tolerance of 75% to 125%.

Blank Spike:

Blank-Spike recovery for analyte of interest exceeded tolerance of 80% to 120%. Measured recovery for Na was about 128%.

Matrix Spiked Sample:

Matrix-Spike recovery was within tolerance of 80% to 120% except for B, which is not an analyte of interest.

Quality Control Check Standards:

Concentration of all analytes of interest are within tolerance limit of ± 10% accuracy in the check standards: QC_MCVA, QC_MCVB, and QC_SSTMCV.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except K. One of two measurements of K in QC_SST was higher than expected (50 ug/mL) by 8%, The other measurement was higher than expected by 3% that is within tolerance limit of ± 5%.

Process Blanks:

Concentration of Na measured in the process blank (about 15% of Na measured in the sample) exceeded tolerance limit ≤ EQL or less than ≤5% of the sample. Other analytes detected in the process blank include Al, B, Ni, and Si. The source of B, Na and Ni in the process blank may have come from the glassware or reagents used to process the sample.

Laboratory Control Standard (LCS):

See "LCS/blank spike" above.

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.

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

	Multiplier=	20.0	20.0	20.0	
	RPL/LAB #=	01-1203-PB	01-0810	01-0810-DUP	
	Client ID=		*AP101L-RGN-A*	*AP101L-RGN-A*	
Det. Limit	*Run Date=*	7/16/2001	7/16/2001	7/16/2001	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	
0.025	Ag	--	--	--	--
0.060	Al	13.6	[10]	12.8	--
0.250	As	--	--	--	--
0.050	B	116	69.3	89.7	--
0.010	Ba	[0.33]	--	[0.43]	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.250	Ca	--	--	--	--
0.015	Cd	--	--	--	--
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.020	Cr	--	--	--	--
0.025	Cu	--	--	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	[0.8]	[1.1]	[0.98]	--
2.000	K	--	--	--	--
0.050	La	--	--	--	--
0.030	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.050	Mo	--	--	--	--
0.150	Na	152	1,030	1,000	--
0.100	Nd	--	--	--	--
0.030	Ni	[1.5]	--	--	--
0.100	P	--	--	--	--
0.100	Pb	--	--	--	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	113	[91]	109	--
1.500	Sn	--	--	--	--
0.015	Sr	--	--	--	--
1.500	Te	--	--	--	--
1.000	Th	--	--	--	--
0.025	Ti	--	--	--	--
0.500	Tl	--	--	--	--
2.000	U	--	--	--	--
0.050	V	--	--	--	--
2.000	W	--	--	--	--
0.050	Y	--	--	--	--
0.050	Zn	--	--	--	--
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).

Data (1) from ~A0698 I.BURGESON ASR-6121 S.FISKUM ASR-6097 ICP98 hi.XLS

8/22/2001 @ 1:21 PM

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

01-0780

05/18/01

Client : Fiskum

Cognizant Scientist: LRGreenwood   Date : 5/18/01

Concur : T Trang-le   Date : 5/18/01

Procedure: PNL-ALO-450 Gamma Energy Analysis

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

ALO ID Client ID	Co-60 Error %	Sr-85 Error %	RuRh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-0780 AP101-F0-A	<7E-3		<5E-1	<3E-1	<1E-1	<2E-2	1.02E+2 2%	<1E-2	<2E-1	<2E-1
01-0781 AP101L-F3	2.57E-3 2%		8.52E-4 5%	1.61E-3 3%	2.52E-4 2%	<1E-5	2.18E-4 4%	<2E-5	<4E-5	1.37E-4 21%
01-0782 AP101L-F6	2.60E-3 2%		8.94E-4 4%	1.58E-3 3%	2.46E-4 2%	<7E-6	1.28E-4 4%	6.66E-5 6%	<3E-5	1.45E-4 14%
01-0783 AP101L-F9	2.49E-3 2%	1.82E-4 8%	8.27E-4 9%	1.45E-3 3%	2.28E-4 3%	<1E-5	4.69E-3 3%	<2E-5	<6E-5	<9E-5
01-0784 AP101L-F11	2.69E-3 3%		1.48E-3 16%	1.71E-3 8%	2.77E-4 9%	<5E-5	6.49E-2 3%	<1E-4	<3E-4	<6E-4
01-0785 AP101L-F13	2.56E-3 3%		<2E-3	1.45E-3 30%	<5E-4	<9E-5	2.76E-1 2%	<2E-4	<7E-4	<7E-4
01-0786 AP101P-F4	2.32E-3 2%		8.34E-4 5%	1.52E-3 3%	2.36E-4 2%	<1E-5	6.17E-5 7%	<2E-5	5.44E-5 21%	1.28E-4 34%
01-0787 AP101P-F8	2.54E-3 2%		8.28E-4 6%	1.56E-3 3%	2.33E-4 3%	<1E-5	9.00E-5 8%	<3E-5	<6E-5	1.13E-4 44%

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

ALO ID Client ID	Co-60 Error %	Sr-85 Error %	RuRh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-0788 AP101P-F13	2.51E-3 2%		7.65E-4 7%	1.53E-3 3%	2.33E-4 3%	<2E-5	7.40E-5 10%	<4E-5	<7E-5	<2E-4
01-0789 AP101-FComp2-A	2.03E-3 2%		8.51E-4 5%	1.52E-3 3%	2.42E-4 2%	<9E-6	9.79E-5 5%	<2E-5	<3E-5	1.46E-4 18%
01-0790 AP101-FComp3-A	2.39E-3 2%		7.77E-4 6%	1.47E-3 3%	2.37E-4 3%	<2E-5	1.09E-4 8%	<4E-5	<5E-5	1.27E-4 20%
01-0791 AP101-FComp4-A	2.36E-3 2%		7.88E-4 5%	1.42E-3 2%	2.27E-4 2%	<1E-5	2.31E-4 3%	<3E-5	6.16E-5 22%	1.20E-4 12%
01-0792 AP101-FD2-A	1.15E-3 2%		<3E-4	4.57E-4 9%	<4E-5	<3E-5	5.93E-3 2%	<7E-5	<9E-5	<9E-5
01-0793 AP101-FD3-A	1.20E-4 3%		<3E-5	2.81E-5 16%	<3E-6	1.43E-5 9%	2.08E-3 3%	<8E-6	<2E-5	<4E-5
01-0794 AP101-FD6-A	4.11E-5 5%		<5E-5	<2E-5	<4E-6	<4E-6	1.61E-3 3%	<1E-5	<2E-5	<6E-5
01-0795 AP101-DI2-A	7.01E-5 5%		<9E-5	<3E-5	<1E-5	<7E-6	2.05E-3 2%	<2E-5	<3E-5	<2E-5
01-0796 AP101-DI5-A	3.16E-5 5%		<3E-5	<1E-5	<2E-6	<3E-6	1.82E-3 3%	<7E-6	<1E-5	<4E-5
01-0797 AP101L-E1-DA	<2E-4		<1E-3	<5E-4	<1E-4	<2E-4	1.21E-2 3%	<4E-4	<5E-4	<1E-3
01-0798 AP101L-E2-DA	<2E-4		<1E-3	<5E-4	<1E-4	<2E-4	1.08E-2 3%	<4E-4	<8E-4	<1E-3
01-0799 AP101L-E3-DA	<3E-2		<3E+0	<2E+0	<1E-1	6.55E-1 6%	2.66E+3 2%	<7E-2	<2E+0	<4E+0
01-0800 AP101L-E4-DA	<1E-1		<2E+1	<2E+1	<1E+0	<7E-1	8.11E+3 2%	<3E-1	<1E+1	<3E+1

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

ALO ID Client ID	Co-60 Error %	Sr-85 Error %	RuRh-106 Error %	Sb-125 Error %	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-0801 AP101L-E5-DA	<2E-1		<2E+1	<1E+1	<8E-1	<5E-1	4.75E+3 2%	<2E-1	<9E+0	<2E+1
01-0802 AP101L-E6-DA	<4E-2		<2E+0	<1E+0	<4E-2	<5E-2	3.61E+2 2%	<7E-2	<9E-1	<2E+0
01-0803 AP101L-E8-DA	<1E-3		<8E-2	<5E-2	<2E-2	<2E-3	7.83E+0 2%	<2E-3	<2E-2	<2E-2
01-0804 AP101L-E9-DA	<6E-4		<3E-2	<1E-2	<4E-3	<1E-3	3.66E+0 3%	<2E-3	<1E-2	<2E-2
01-0805 AP101L-E10-DA	<6E-4		<2E-2	<1E-2	<2E-3	<1E-3	2.01E+0 3%	<1E-3	<9E-3	<2E-2
01-0806 AP101L-E11-DA	<8E-4		<3E-2	<2E-2	<2E-2	<1E-3	1.32E+0 3%	<2E-3	<1E-2	<2E-2
01-0807 AP101L-E15-DA	<2E-4		<3E-3	<1E-3	<2E-4	<2E-4	5.50E-1 3%	<4E-4	<1E-3	<2E-3
01-0808 AP101L-E18-DA	<5E-4		<9E-3	<4E-3	<5E-4	<6E-4	3.76E-1 3%	<2E-3	<4E-3	<7E-3
01-0809 AP101L-E21-DA	<2E-4		<4E-3	<2E-3	<3E-3	<2E-4	3.59E-1 3%	<5E-4	<2E-3	<3E-3
01-0810 AP101L-RGN-A	<1E-5		<9E-4	<5E-4	<1E-4	<4E-5	5.85E-2 3%	<5E-5	<3E-4	<7E-4
01-0879 AP101L-E7-DA	<8E-3		<5E-1	<3E-1	<2E-1	<1E-2	4.94E+1 2%	<1E-2	<2E-1	<2E-1

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Date: 6/20/01

Subject: Hydroxide Analyses for: S. Fiskum
ASR: 6097

To: S. Fiskum

From: L. Greenwood

A sample from tank AP-101L-RGN-A was 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 # **6097**

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

Analyst: Lori P. Darnell 6-21-01

Reviewer: LR Greenwood 6-20-01

Summary Report

RPG #	Client ID		Concentration, moles First Point	Second Point	Third Point
01-0810	**AP101L-RGN-A**		**0.032**	**0.016**	
01-0810	**AP101L-RGN-A**	**Rep**	**0.027**	**0.016**	
		RPD	**18%**	**3%**	
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.

Project / WP#: 42365 / W58168
ASR#: 6121
Client: I. Burgeson
Total Samples: 1

	From	To
RPL#:	01-01203	
Client ID:	"AP1-Tc-O-C"	

Sample Preparation: PNL-ALO-106/SRPL/ms (1 mL/25mL)
[AP101 tank waste with ^{137}Cs removed] AOI: Al, K, and Na

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

Analyst: D.R. Sanders

Analysis Date (File): 07-16-01 (A0698)

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)

Jerry Wagner 8-22-01
Reviewed by

8/30/01
Concur

One sample of AP101 tank waste with ^{137}Cs removed, **AP1-Tc-O-C** (RPL# 01-01203), was processed by SRPL using PNL- ALO-106 acid digestion procedure. Sample and duplicate was prepared using 1 mL (weighed) aliquots of liquid material. After digestion the sample was diluted to a final volume of 25mL (weighed). Sample density of liquid before processing was estimated to be about 1.03 g/mL by weighing 1 mL aliquots. See PNL-ALO-106 worksheet in supporting documents for sample preparation information.

Analytes of interest requested for analysis by ICPAES include Al, K and Na. The process blank appears to have a small amount of Al, B, Ba, Fe, Na, Ni, and Si present. Boron, Na, and Si may come from glassware used to digest the samples or from the digestion reagent (nitric acid). The Na and Al concentration in the process blank are less than 0.3% of that found in the samples.

See attached ICPAES Data Report for final results. ICPAES measurement results are reported in **µg/mL** and have been corrected for dilution resulting from sample processing. Analyte concentrations reported other than specifically requested are for information only.

Quality control check-standard results met tolerance requirements for the specific analytes of interest except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan. LCS/BS, Matrix-spikes and duplicate were prepared with the samples and analyzed. LCS/blank-spike and matrix-spike was prepared using 1mL each of a custom multi-element solution "INT-QC-MCVA-1B" (CMS# 186946) per 25 mL sample digestate volume.

Five fold serial dilution:

All analytes above EQL in samples tested were within tolerance limit of 10% after correcting for dilution except K (14%) and Na (16%). The more dilute aliquot measured may have been measure be for the instrument was sufficiently clean from previous samples high in Na and K. A 10-fold vs 5-fold dilution of the duplicate sample after correction for dilution was well within tolerance limit for both K and Na.

Duplicate RPD (Relative Percent Difference):

The original and duplicate sample was within tolerance limit of ≤ 15% RPD for analytes of interest.

Post-Spiked Samples (Group A):

All post-spiked analytes of interest in samples tested were recovered within tolerance of 75% to 125%.

Post-Spiked Samples (Group B):

All post-spiked analytes in samples tested were recovered within tolerance of 75% to 125%.

Blank Spike:

Blank-Spike recovery for analytes of interest was within tolerance of 80% to 120% except Na. Measured recovery for Na was about 128%.

Matrix Spiked Sample:

Matrix-Spike recovery for analytes of interest could not be verified because Al, K and Na concentration in the sample was greater than 10 times the concentration of the spike.

Quality Control Check Standards:

Concentration of all analytes of interests are within tolerance limit of ± 10% accuracy in the check standards: QC_MCVA, QC_MCVB, and QC_SSTMCV.

High Calibration Standard Check:

Verification of the high-end calibration concentration in QC_SST for all analytes contained in the standard is within tolerance of ± 5% accuracy except K. One of two measurements of K in QC_SST was higher than expected (50 ug/mL) by 8%, The other measurement was higher than expected by 3% that is within tolerance limit of ± 5%.

Process Blanks:

Concentration of analytes of interest (Al, Na, K) measured in the process blank were all within tolerance limit of ≤ EQL or less than ≤5% of that in the sample. Other analytes detected in the process blank include B, Ni, and Si. The source of analytes detected in the process blank may have come from the glassware or reagents used to process the sample.

Laboratory Control Standard (LCS):

See “LCS/blank spike” above.

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.

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

	Multiplier=	25.0	125.0	125.0	
	RPL/LAB #=	01-1203-PB	01-1203 @5	01-1203-DUP @5	
	Client ID=		*API-Tc-0-C*	*API-Tc-0-C*	
Det. Limit	*Run Date=*	7/16/2001	7/16/2001	7/16/2001	
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	
0.025	Ag	--	--	--	--
0.060	Al	17.0	6,480	6,760	--
0.250	As	--	--	--	--
0.050	B	145	211	208	--
0.010	Ba	[0.41]	--	--	--
0.010	Be	--	--	--	--
0.100	Bi	--	--	--	--
0.250	Ca	--	--	--	--
0.015	Cd	--	--	--	--
0.200	Ce	--	--	--	--
0.050	Co	--	--	--	--
0.020	Cr	--	138	143	--
0.025	Cu	--	--	--	--
0.050	Dy	--	--	--	--
0.100	Eu	--	--	--	--
0.025	Fe	[1.0]	--	--	--
2.000	K	--	28,400	29,800	--
0.050	La	--	--	--	--
0.030	Li	--	--	--	--
0.100	Mg	--	--	--	--
0.050	Mn	--	--	--	--
0.050	Mo	--	[12]	[13]	--
0.150	Na	191	108,000	113,000	--
0.100	Nd	--	--	--	--
0.030	Ni	[1.9]	[5.8]	--	--
0.100	P	--	326	332	--
0.100	Pb	--	--	--	--
0.750	Pd	--	--	--	--
0.300	Rh	--	--	--	--
1.100	Ru	--	--	--	--
0.500	Sb	--	--	--	--
0.250	Se	--	--	--	--
0.500	Si	142	[320]	[310]	--
1.500	Sn	--	--	--	--
0.015	Sr	--	--	--	--
1.500	Te	--	--	--	--
1.000	Th	--	--	--	--
0.025	Ti	--	--	--	--
0.500	Tl	--	--	--	--
2.000	U	--	--	--	--
0.050	V	--	--	--	--
2.000	W	--	--	--	--
0.050	Y	--	--	--	--
0.050	Zn	--	--	--	--
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).

Data (1) from ~A0698 I.BURGESON ASR-6121 S.FISKUM ASR-6097 ICP98 hi.XLS

8/22/2001 @ 11:58 AM

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

Client:	I. Burgeson	Charge Code/Project:	W58168/42365
ASR Number:	6121	Sample Receipt Date:	06/05/2001
Sample Prep Date:	N/A	Sample Analysis Date:	06/12-13/2001
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.

Sample (01-01203) Final Results:

RPL Number	Sample ID	F (1) 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	53	53	525	53	1,250	105	105	105
01-01203	AP1-Tc-0-C	1,830	1,410	36,200	< 50	117,000	690	3,090	940
01-01203 Dup	AP1-Tc-0-C Dup	1,870	1,490	35,400	< 50	116,000	690	3,150	950
	RPD	2%	6%	2%	n/a	1%	1%	2%	1%
01-01203 MS	AP1-Tc-0-C MS	101%	93%	96%	103%	84%	92%	94%	102%
LLS % recovery		106%	109%	102%	100%	98%	98%	98%	108%
LCS % recovery		107%	108%	103%	105%	101%	101%	100%	110%

(1) The fluoride results should be considered the upper bound concentration for the fluoride. Significant peak distortion of the fluoride peak suggests the presence of co-eluting anion(s), possibly formate or acetate.

The sample was prepared for ion chromatography anion analysis by dilution at 400-fold (F, Cl, Br, PO_4, SO_4, and C_2O_4), 2000-fold (NO_2) and 5000-fold (NO_3) in order to ensure that the anions were measured within the calibration range. Column overloading prohibited analysis of the sample as dilutions less than 400-fold. The estimated quantitation limits which are based on the lowest calibration standard and the dilutions used for reporting the results are provided in the table.

Q.C. Comments:

Duplicates: No duplicate was provided. However, the 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 and all anion recoveries were within the 75% to 125% recovery acceptance criteria.

Laboratory Control Sample -LSC (Blank Spike): 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): A LLS was prepared and analyzed with recoveries meeting the recommended 75% to 125% recovery acceptance criteria.

System Blank/Processing Blanks: Twelve 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: Seven mid-range verification standards were analyzed throughout the analysis runs. Except for two oxalate measurements, all anions recoveries were within the acceptance criteria from 90% to 110% for the verification standard. The two oxalate measurement that failed produced recoveries of 111%.

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.

Analyst: ____________ **Date** 7-11-01

Approval: MJ Steele **Date** 7-17-01

Archive Information:

Files: ASR 6121 Burgeson.doc ASR 6105, 6121 Burgeson.xls

Battelle Pacific Northwest Laboratory 01-1203
Radiochemical Processing Group-325 Building
Chemical Measurement Center 07/23/01

Client : I. Burgeson

Cognizant Scientist: LRGreenwood Date : 7/23/01

Concur : T Trang-le Date : 9/12/01

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

ALO ID Client ID		Tc-99* Error %
01-1203 AP1-Tc-O-C		3.71E-2 4%
01-1203 Lab Dup AP1-Tc-O-C		4.19E-2 4%
	RPD	12%
Matrix Spike		83%
Blank Spike		95%
Blank		<6.E-7

*Note: Tc-99 was chemically separated without adding sodium dichromate such that the above result only represents the Tc-99 in the sample as a pertechnetate.

Battelle Pacific Northwest Laboratory 01-1203
Radiochemical Processing Group-325 Building
Chemical Measurement Center 06/30/01

Client : I. Burgeson

Cognizant Scientist: L. R. Greenwood Date : 6/30/01
Concur : Kathie Thomas Date : 7/2/01

PNL-ALO-450 (GEA)
Reference Date: 6/28/01

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

ALO ID Client ID	Co-60 Error %	Ru/Rh-106 Error %	Sb-125 Error %	Sn/Sb-126 Error %	Cs-137 Error %	Eu-154 Error %	Eu-155 Error %	Am-241 Error %
01-1203 AP1-Tc-O-C	2.23E-3 2%	7.64E-4 5%	1.45E-3 2%	2.33E-4 2%	1.08E-4 6%	<3.E-5	7.35E-5 18%	1.49E-4 12%

Battelle PNWD
PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 1

Project / WP#: 42365 / W58168
ASR#: 6121
Client: Ingrid Burgeson
Total Samples: 1

RPL #	Client ID
01-1203	AP1-Tc-O-C

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: 9/18/01

Analysis Files:
Experiment – 12SEP01
Procedure - 010912a
Element Menu - CsTcRb

Revised Report Date: 15 January 2004

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

tmP Thomas — 15Jan04
Revised By — Date

Orville Thomas Farmer — 15Jan04
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).

One sample, AP1-Tc-O-C, submitted for analysis was analyzed on a radioactive-material-contained ICP/MS for the requested analyte, Tc^{99}.

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, a replicate analysis was also performed. The RPD for both duplicate and replicate analyses met the duplicate RSD criteria of < 15%.

Matrix Spike (MS). In addition to the matrix spike sample submitted, a post spike was also performed. The spike recovery for both the matrix spike and post spike met the QC criteria of 70% – 130%.

Process Blank (PB), Blank Spike (BS). The PB concentration was below detection limit and MRQ. The BS and a post spike of the PB met the spike recovery QC criteria of 70% – 130%.

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), was met.

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

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

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%.

Battelle, PNNL, AIAL
ICP/MS Analysis Data Report

Reviewed by: 11-26-01
Analyst: James Bramson 11/27/01

Client: Ingrid Burgeson
WP/Project: W58168 / 42365
ASR/Log-In: 6121, 01-01203
Report Date: 11/25/01

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

RPL Log-in #	Sample ID	ICP/MS ID	MDL µCi/ml	Tc 99 ± 1SD µCi/ml	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	
	True Value			**1.70E-06**	
	ICV	ICV		1.62E-06 ± 3.43E-08	95.7%
	CCV	CCV1		1.62E-06 ± 4.61E-08	95.4%
	CCV	CCV2		1.66E-06 ± 4.23E-08	97.8%
	CCV	CCV3		1.82E-06 ± 1.56E-07	107%
01-01203-PB	PROCESS BLANK	Sample1	7.22E-06	< 7.22E-06	
01-01203-PB	PROCESS BLANK Post Spike	Sample2	7.26E-06	4.70E-03 ± 1.07E-04	
Post Spike Concentration Expected				4.24E-03	**111%**
01-01203-BS	BLANK SPIKE	Sample3	7.27E-06	1.28E-03 ± 9.28E-05	
Matrix Spike Concentration Expected				1.70E-03	**76%**
01-01203	AP1-Tc-O-C	Sample4	1.52E-05	5.01E-02 ± 1.65E-04	
01-01203	AP1-Tc-O-C replicate	Sample5	1.59E-05	4.96E-02 ± 2.20E-04	**0.90%**
01-01203-DUP	AP1-Tc-O-C DUP	Sample6	1.55E-05	5.15E-02 ± 5.16E-04	**2.91%**
01-01203	AP1-Tc-O-C Post Spike	Sample11	1.26E-05	5.90E-02 ± 1.79E-03	
Post Spike Concentration Expected				8.48E-03	**106%**
01-01203-MS	AP1-Tc-O-C MS	Sample12	1.29E-05	5.13E-02 ± 1.29E-04	
Matrix Spike Concentration Expected				1.70E-03	**75%**

MTE: ICP/MS VG (WB36913), Mettler 512-06-01-014
Procedure: PNL-ALO-280 Rev. 1
Analysis Date: 9/18/01
Instrument Filenames: Experiment (12SEP01), Procedure(010912a), Element Menu (CsTcRb)

Battelle
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Date: 6/27/01

Subject: Hydroxide Analyses for: 01-1203
ASR: 6121

To: Ingrid Burgeson

From: Larry Greenwood

Sample 01-1203 was analyzed in triplicate for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. For this analysis, 0.0103 M NaOH solution was prepared for use as a standard and sample spike and the titrant chosen was 0.0203 M HCl. See attached Chemrec_72 for standardization of these reagents.

The results indicated good agreement between the sample and the replicates. The laboratory control standards showed excellent recovery at 102-103% and a matrix spike recovered at 105%. The measured hydroxide concentrations in the samples were well below the MRQ value of 7.5E+4 ug/ml or about 4.4 moles/liter.

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

ASR # 6121

WP# K88426

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

Analyst: Lori P. Darnell 7-18-01

Reviewer: LR Greenwood 7-18-01

Summary Report

RPG #	Client ID		Concentration, moles First Point	Second Point	Third Point
01-1203	AP1-Tc-O-C		2.44	0.53	0.49
01-1203	AP1-Tc-O-C		2.36	0.48	0.48
01-1203	AP1-Tc-O-C		2.40	0.46	0.55
		RSD	2%	7%	7%
Standard 3			102%		
Standard 4			103%		
Standard 5			102%		
MS-1203	AP1-Tc-O-C	MS	105%		

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, Pacific Northwest Division
Richland, WA
Radiochemical Processing Group

filename 01-1203

1/22/2002

Client : Burgeson
ASR 6121

Cognizant Scientist: L.R. Greenwood Date : 1/23/02

Concur : C. Soderquist Date : 1-22-02

Procedure: PNL-ALO-4014, Rev 1
Chem-Chek model KPA 11R uranium analyzer
Analysis date 1/8/02

Measured Concentration (ug/ml) with 1-sigma error

ALO ID Client ID	Uranium µg/ml	Uranium +/-%	Detection Limit µg/mL
01-1203 AP1-Tc-O-C	9.85E-1	2%	4E-2
01-1203 DUP AP1-Tc-O-C	8.71E-1	2%	4E-2
RPD	12%		
Matrix Spike (01-1613)	95%		
LCS	100%		
Blank	4.06E-3	2%	

The detection limit is corrected for the sample dilutions. The blank did not undergo as many dilutions and consequently has a lower detection limit than the samples.

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Project No. 42365

Internal Distribution
G. Smith
S. Fiskum
File/LB

Date May 3, 2002

To I. Burgeson

From L. R. Greenwood

Subject Radiochemical Analyses for AP-101 Composite and Tc-Ap1-Eluate – ASR 6192

A sample of the composite from tank AP-101 and the Tc-Ap1-Eluate were analyzed for gamma emitters, ^{3}H, ^{90}Sr, total alpha, Pu/AEA, ^{241}Pu, ^{151}Sm, Am/Cm, and ^{99}Tc according to ASR 6192. Analyses were performed on the as received samples. 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. In order to meet the requested detection limits, the samples were counted for up to 14 hours. The Tc-Ap1-Eluate showed significant activity for ^{95m}Tc presumably used in the Tc removal process. ^{54}Mn and ^{95}Nb could not be detected in this sample due to strong interference from ^{95m}Tc. The requested MRQ values were met except for ^{154}Eu and ^{155}Eu for the composite due to the very high ^{137}Cs activity.

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. The composite results have a high counting uncertainty and alpha activity could not be detected in the eluate. Summing the individual alpha emitters, as discussed below, provides a better estimate of the total alpha activity. No alpha activity was seen in the laboratory blank and blank spike and matrix spike recoveries were acceptable. The sum of the alpha emitter activities is well below the requested MRQ value.

Plutonium, Americium, and Curium

The Pu and Am/Cm separations were performed 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. Plutonium recovery was traced with ^{242}Pu. The curium is known to follow the americium and both these isotopes were traced with ^{243}Am. Both the plutonium and americium radiochemical yields were excellent, averaging about 95%. Duplicate results are in good agreement. Neither Am or Cm were detected in the laboratory preparation blank and ^{239}Pu was negligible relative to the sample activity. The LCS and matrix spike recoveries were 85 to 98% for both Pu and Am. Measured values for the Am, Cm, and Pu isotopes are well below the requested MRQ values. The sum of the alpha emitters given in the last column of the report is the best indicator of the total alpha content for these samples.

Plutonium-241

The ^{241}Pu was measured by placing the precipitation mounts from the Pu/AEA method described above into liquid scintillation cocktail and counting according to procedure PNL-ALO-474. Blank and matrix spikes were prepared and handled along with the Pu/AEA samples and a ^{241}Pu standard was used to determine the liquid scintillation counting efficiency. The Pu/AEA radiochemical yields determined with a ^{242}Pu tracer were used for the ^{241}Pu recovery for each sample. The LCS and matrix spike recoveries were 100% and 102%, respectively. No contamination was seen in the laboratory blank. No MRQ was specified for this isotope.

Samarium-151

Rare earth elements, including samarium, follow the Am/Cm fraction in the procedure PNL-ALO-417 described above. The Am/Cm-AEA mounts were placed into scintillation cocktail and counted according to procedure PNL-ALO-474. A ^{151}Sm spike was added prior to the chemical separations and the ^{243}Am tracer was used to determine the radiochemical yields. The LCS recovery was 86%; however, the matrix spike recovery was only 63% possibly indicating a low bias in the measurement. Since Eu will also be separated along with the Sm, the ^{154}Eu and ^{155}Eu activities (about 7% of the total activity) determined by gamma energy analysis were subtracted from the measured ^{151}Sm activity. All of the samples had counts above background in the energy region expected for ^{151}Sm. However, the beta energy spectra did not show any peak corresponding to the energy region expected for ^{151}Sm. Hence, the presence of ^{151}Sm in these samples is doubtful. No MRQ was specified for this isotope.

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). No ^{90}Sr was found in the laboratory preparation blank. The samples were analyzed in two batches since the first attempt with the composite failed to detect 90Sr and did not meet the requested MRQ value. The LCS and matrix spike recoveries were 99% and 95%, respectively, for the batch with the eluate and 127% and 110%, respectively, for the batch with the composite. No 137Cs contamination was seen in the beta spectra for the eluate; however, some contamination was detected with the composite and the data were corrected accordingly. The high LCS recovery has a very high uncertainty since the spike activity was too low relative to the sample activity. The measured activities were well below the requested MRQ value.

Tritium

Tritium was distilled from the samples according to procedure PNL-ALO-418 and measured by liquid scintillation counting according to procedure PNL-ALO-474. The samples were analyzed in two batches since the first batch showed beta contamination (probably ^{90}Sr) in some of the beta energy spectra. The LCS recovery was 93% for the first batch; however, the matrix spike was highly contaminated with a higher energy beta emitter such that the tritium yield could not be determined. The second batch had an LCS recovery of 96% and a matrix spike recovery of 79%. The laboratory blanks showed weak tritium contamination for one batch and no tritium for the second batch. No MRQ value was specified for this isotope.

Uranium

Uranium was measured by kinetic phosphorescence analysis (KPA) according to procedure PNL-ALO-4014. The samples were acid digested, taken to dryness, and dissolved in 0.5 M HNO_3 prior to analysis to eliminate potential interferences. Both samples were analyzed in duplicate with good reproducibility. The eluate contained very little uranium, only slightly above the level seen in the laboratory blank. However, the level of uranium in the eluate is a factor of about 1.E+5 below the requested MRQ value of 600 mg/L (or ug/ml). The laboratory control standard recovery was 100%. The matrix spike (made from sample 01-1613, ASR 6162) had a 95% recovery.

Nickel-63

Direct sample aliquots were analyzed for ^{63}Ni according to procedure PNL-ALO-495. The separated nickel fractions were counted by liquid scintillation counting according to procedure PNL-ALO-474. The beta energy spectra did not indicate any significant contamination with other beta emitters. The AP101L-E-composite sample showed a low level of ^{63}Ni; however, no activity could be detected in the Tc-Ap1-Eluate. Sample duplicates for the composite showed good agreement with an RPD value of 2%. The laboratory control standard recovery for a ^{63}Ni standard was 86% and the matrix spike recovery was 89%. A laboratory blank did not show any activity above background. No MRQ value was specified for ^{63}Ni.

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

01-1842

10/01/01

Client : Burgeson

Cognizant Scientist: L. R. Greenwood Date : 10/1/01

Concur : T Trang-Le Date : 10/1/01

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

ALO ID Client ID	Cr-51 Error %	Mn-54 Error %	Fe-59 Error %	Co-60 Error %	Y-88 Error %	Nb-95 Error %	Ru-103 Error %	Ru-106 Error %	Sn-113 Error %	Sb-125 Error %
01-1842 AP101L-E-Comp	<0.9	<2.E-2	<3.E-2	<5.E-3	<2.E-2	<2.E-2	<2.E-1	<7.E-1	<2.E-1	<4.E-1
01-1843 Tc-Ap1-Eluate	<2.E-3	*	<5.E-5	<3.E-5	<6.E-5	*	<3.E-4	<3.E-3	<3.E-4	<7.E-4

*Not detectable due to strong interference from Tc-95 and Tc-95m

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

ALO ID Client ID	SnSb-126 Error %	Cs-134 Error %	Cs-137 Error %	Ce-144 Error %	Eu-152 Error %	Eu-154 Error %	Eu-155 Error %	Th-232 Error %	Tc-95 Error %	Tc-95m Error %
01-1842 AP101L-E-Comp	2.69E-1 19%	1.56E-1 6%	7.65E+2 2%	<0.6	<1.E-2	<3.E-2	<3.E-1	<2.E-1	<2.E-2	<2.E-1
01-1843 Tc-Ap1-Eluate	4.15E-3 10%	<3.E-4	<2.E-4	<2.E-3	<2.E-4	<7.E-5	<9.E-4	<4.E-4	2.95E-2 2%	6.25E-1 2%

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

01-1842

4/26/2002

Client : Burgeson

Cognizant Scientist: LRGreenwood Date : 5-2-02

Concur : C. Soderquist Date : 4-26-02

Reference Date: Sept. 26,2001 @11:24 a.m. for Beta
Reference Date: Sept. 20 and 26, 2001 for H-3
Reference Dates: Oct. 30,2001 for 01-1843; Sept. 27 for 01-1842 for Sr-90
Reference Date: Feb 12, 2002 for Ni-63

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

ALO ID Client ID	Beta Error +/-	H-3 Error +/-	Sr-90 Error +/-	Rerun Sr-90 Error +/-	Ni-63 Error +/-
01-1842 AP101L-E-Comp	8.07E+2 4%	1.73E-4 10%	2.91E-2 3%	2.84E-2 5%	8.92E-4 1%
01-1842 DUP AP101L-E-Comp	7.87E+2 4%	1.70E-4 22%	2.98E-2 3%	2.79E-2 5%	8.73E-4 1%
RPD	3%	2%	2%	2%	2%
01-1843 Tc-Ap1-Eluate	6.48E-1 3%	<5.E-5	1.26E-4 4%		<3E-5
01-1843 Dup Tc-Ap1-Eluate					<3E-5
Matrix Spike*	74%	79%	95% 127%	**	89%
Blank Spike*	103%	96% 93%	99% 110%	108%	86%
Blank	<5.E-2	1.47E-5 48%	<4.E-1 <2.E-4	<2.E-4	<3E-5

*The H-3 and Sr-90 measurements were performed in two different batches.

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

01-1842

10/04/01

Client : Burgeson

Cognizant Scientist: LRGreenwood Date : 10/4/01

Concur : T Trang-le Date : 10/5/01

Reference Date: Sept. 18,2001 @12:00 p.m. for Alpha
Reference Date: Sept. 26,2001 @8:00 a.m. for Pu/Am

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

ALO ID Client ID	Alpha Error +/-	Pu-239+ Pu-240 Error +/-	Pu-238 Error +/-	Am-241 Error +/-	Cm-243+ Cm-244 Error +/-	Cm-242 Error +/-	Sum of Alpha Error +/-
01-1842	9.13E-4	4.70E-4	5.89E-5	3.18E-5	6.59E-6	<2.E-7	5.67E-4
AP101L-E-Comp	11%	2%	4%	3%	6%		2%
01-1842 DUP1		4.56E-4	6.11E-5	2.91E-5	8.00E-6	<2.E-7	5.54E-4
AP101L-E-Comp		2%	4%	3%	6%		2%
RPD		3%	4%	9%	19%		2%
01-1842 DUP2		4.78E-4	6.20E-5				
AP101L-E-Comp		2%	2%				
RPD		5%	1%				
01-1843	<2.E-6	1.62E-7	<6.E-8	7.22E-7	<4.E-8	<4.E-8	8.84E-7
Tc-Ap1-Eluate		18%		9%			8%
Matrix Spike	84%	85%		87%			
Blank Spike	105%	98%		93%			
Blank	<2.E-6	3.19E-7	<3.E-7	<5.E-7	<3.E-7		
		32%					

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

01-1842
Rev. 1
01/31/03

Client : Burgeson
ASR: 6192

Cognizant Scientist: Date : 1/31/03

Concur : C. Soderquist Date : 1-31-03

Reference Date: Oct. 5, 2001 @ 9:08 a.m. for Pu-241
Reference Date: Nov. 2, 2001 @12:00 p.m. for Sm-151

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

ALO ID Client ID	Pu-241 Error +/-	Sm-151* Error +/-
01-1842 AP101L-E-Comp	3.28E-3 8%	1.16E-4 7%
01-1842 DUP AP101L-E-Comp	2.83E-3 8%	1.17E-4 7%
01-1842 Rep AP101L-E-Comp	3.20E-3 8%	8.38E-5 9%
RSD	8%	18%
01-1843 Tc-Ap1-Eluate	<1.E-5 3%	7.81E-6 18%
Matrix Spike (01-1842)	102%	62%
Blank Spike	100%	83%
Blank	<5.E-2	<4.E-6

*Part of the activity reported as Sm-151 may be from other beta emitters. The Sm-151 counting mounts had only a little beta activity and no Sm-151 beta peak.

Note: The Sm-151 results were revised due to the discovery of an error on the preparation of the Sm-151 standard that was used to determine the liquid scintillation counting efficiency.

S. Fiskum

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Project No. 42365

Internal Distribution
G. Smith
S. Fiskum
File/LB

Date	March 19, 2002
To	I. Burgeson
From	L. R. Greenwood LRG
Subject	^{14}C Analyses for AP-101 IX Eluents – ASR 6192

Direct samples of the ion exchange eluents from tank AP-101 with Cs and Tc removed were analyzed for ^{14}C according to procedure PNL-ALO-482. Following sample combustion, the collected $^{14}CO_2$ was counted by liquid scintillation counting according to procedure PNL-ALO-474. The attached report lists the measured ^{14}C 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. The ^{14}C spike recovery through the combustion and collection procedure averaged 95% for three standards. A matrix spike showed poor recovery at 70%. Subsequent analyses indicated that the acidity of the sample caused some decomposition of the ^{14}C spike prior to combustion. Duplicate analyses for sample AP101L-E-Comp showed reasonable agreement taking into account the very high uncertainties in the counting statistics. No activity was detected in the laboratory blank. Periodic rinses analyzed with the batch of samples did not show any significant ^{14}C retention by the furnace and gas collection system. No MRQ value was provided on the ASR.

E54-1900-001 (8/98)

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

File: 01-1842.xls

Client : Burgeson
ASR 6192

03/15/02

Cognizant Scientist: LR Greenwood Date : 3/15/02

Concur : MJ Steele Date : 3/19/02

C-14 Procedure PNL-ALO-482; Reference Date: 3/8/02

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

ALO ID Client ID	C-14	Error%	MDA
01-1842 AP101L-E-Comp	2.88E-5	26%	2.E-5
01-1842 DUP AP101L-E-Comp	1.86E-5	39%	2.E-5
RPD	43%		
01-1843 Tc-Ap1-Eluate	2.10E-5	34%	2.E-5
Lab Blank			2.E-5
Lab reagent spike	95%	Average of three LCS spikes	
Matrix Spike 01-1842*	70%		

*Sample matrix possibly decomposed spike prior to combustion

Battelle – PNNL / AIAL
Inorganic Analysis / 320-ICP/MS Data Report

Project / WP#:	42365 / W57922
ASR#:	6192
Client:	Mike Urie
Total Samples:	2

RPL #	Client ID
Blk-1613	Reagents only
01-1842	AP101L-E-COMP

Procedure: PNL-SC-01 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: Orville Thomas Farmer III

Report Written by: Orville Thomas Farmer III

Analysis Date(s): 9/23/01

Analysis Files:
Experiments – 17NOV01
Procedures – blk-1613, blk-1613 dup, blk-1613 trp, 01-1842, 01-1842 dup, 01-1842 trp.
Element Menus – CS-ISO

Laboratory Record Book (LRB):		
	LRB BNW 56465: Sample login:	p.60
	LRB BNW 56465: Cs Isotopics assay:	p.60

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

J.A. Campbell 11/28/01
Reviewed By

Orville Thomas Farmer III
Concur 28NOV01

1. Analysis

Two liquid samples for Cs isotopic analysis were received from the client and were analyzed by HPIC-ICP/MS. All samples were diluted by a factor of 200 as received and screened to determine an estimate of the Cs concentration. The screening results were used to determine optimum Cs response during the on-line separation into the ICP/MS.

A Dionex HPIC system was configured on-line with the ICP/MS for the separation of Ba and Cs isotopes. A CS3 cation column was used as the stationary phase and a gradient eluent of nitric acid was used as the mobile phase for this separation. Data was collected using peak-hopping data acquisition parameters.

ICP/MS results for all solutions are reported in **atomic abundance** and are instrument blank corrected. In Table 1: reported results are in atomic abundance, the precision of the analysis was determined by analyzing three separate aliquots of a single sample. These three solutions were analyzed to measure the separation and analysis procedures accuracy and precision.

Table 1: Cs Atomic Abundance

File Name	Cs-133 +/- 1 sigma	Cs-135 +/- 1 sigma	Cs-137 +/- 1 sigma
01-1613 BLK	1.0	N.D.	N.D.
01-1842	0.6060 +/-0.00012	0.1543 +/-0.00039	0.2397 +/-0.00094
01-1842 DUP	0.6058 +/-0.00073	0.1543 +/-0.00026	0.2400 +/-0.00056
01-1842 TRP	0.6056 +/-0.00064	0.1543 +/-0.00020	0.2401 +/-0.00049
Average Ratio	0.60581	0.15429	0.23989
Standard Dev.	0.00020	0.00001	0.00021
%RSD	0.033	0.006	0.086

N.D. Blanks were analyzed in the same manner as the samples. The response of the blanks was below instrument detection for Cs-135 and Cs-137 and no Cs ratios were calculated. The 01-1613 blank solution was used only to subtract Cs-133 due to the preparation process. Currently there does not exist a known total Cs check sample and therefore a triplicate is used to measure precision of the procedure.

QC Summary:

Duplicate (DUP): A duplicate was not submitted.

Matrix Spike (MS): No post matrix spike was analyzed for this work.

Post Spike (PS): Not a QC requirement.

Preparation Blank (PB) and Laboratory Control Standard (LCS/BS): A preparation blank was submitted and analyzed three times and meet the criteria of being < 5% of the measured concentration in the samples.

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB): Not a QC requirement.

Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV): Not a QC requirement.

Internal Standard (ISTD): Not a QC requirement.

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

01-1842

11/13/01

Client : Burgeson

Cognizant Scientist: C Soderquist 11-6-01

Concur : L.R. Greenwood 11-13-01

Balance # 1113292667 and 1113162654 (both located on lab 420)

ALO ID Client ID	Measured Density ± 1s
01-1842 AP101L-E-Comp	1.017 g/mL ± 0.5%
01-1843 Tc-Ap1-Eluate	0.996 g/mL ± 0.5%

Density was measured by weighing several aliquots dispensed from a 1 mL pipet. The aliquots were weighed on a balance with a resolution of ± 0.0001g.

The pipet was checked by weighing five aliquots of deionized water, and it delivered 1.00 mL to within 0.2% (1s)

Battelle, Pacific Northwest Division
Richland, WA
Radiochemical Processing Group

filename 01-1842

1/22/2002

Client : Burgeson
ASR 6192

Cognizant Scientist: L.R. Greenwood Date : 1/23/02

Concur : C Soderquist Date : 1-22-02

Procedure: PNL-ALO-4014, Rev 1
Chem-Chek model KPA 11R uranium analyzer
Analysis date 1/8/02

Measured Concentration (ug/ml) with 1-sigma error

ALO ID Client ID	Uranium µg/ml	Uranium +/-%	Detection Limit (µg/mL)
01-1842 AP101L-E-Comp	1.99E+2	4%	4E-2
01-1842 DUP AP101L-E-Comp	2.12E+2	4%	4E-2
RPD	6%		
01-1843 Tc-Ap1-Eluate	6.50E-3	2%	4E-4
01-1843 DUP Tc-Ap1-Eluate	7.08E-3	2%	4E-4
RPD	9%		
Matrix Spike (01-1613)	95%		
LCS	100%		
Blank	4.06E-3	2%	

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

Client: I. Burgeson
RPL Numbers: 01-01613, 01842, 01843
Analyst: MJ Steele

Charge Code/Project: W58950 / 42365
ASR Number: 6162 and 6192
Analysis Date: October 17, 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

		TIC			TOC			TC	
RPL Number	Sample ID	MDL µgC/mL	Results µgC/mL	RPD/ RSD	MDL µgC/mL	Results µgC/mL	RPD/ RSD	Results µgC/mL	RPD
01-01613	TC-AP1-EFF-Comp	16	5,250		44	1,630		6,880	
01-01613 Dup	TC-AP1-EFF-Comp	16	5,280	0.6% / 0.4%	44	1,560	4.4% / 3.1%	6,840	1%
01-01613 MS	Recovery		100%			98%		100%	
01-01842	AP101L-E-Comp	11	<11		30	<30		< 41	
01-01843	TC-AP1-Eluate	8	31		22	150		180	
Blank Spike/LCS	Recovery		102%			103%			

RPD = Relative Percent Difference RSD = Relative Standard Deviation

The TOC/TIC analyses of the samples submitted under ASRs 6162 and 6192 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 TIC analysis uses calcium carbonate and the TOC uses α-D-Glucose as the calibration, laboratory control, and matrix spike standards. (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 (i.e., liquids and solids). 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 as well as the additional QC requirement defined in Table 1-5 (ASR 6192). ASR 6192 provided more stringent duplicate precision acceptance criteria than the QA Plan (i.e,. <15% RSD versus <20% RPD); the LCS and matrix spike acceptance criteria are the same.

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

Laboratory Control Sample (LCS)/Blank Spike(BS): A LCS/BS was analyzed with the samples. The LCS/BS recovered at 102% for the TIC and 103% for the TOC, well within the 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. A matrix spike sample was prepared using sample TC-AP-EFF-Comp. The matrix spike recovery for TIC was 100% and for TOC 98%, both 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 within the QP Plan acceptance criteria of <20% RPD. Besides the RPD, the relative standard deviation (RSD) was calculated (based on two measurements). The RSD for the TIC (0.4%) and the TOC (3.1%) are within the TOC and the TIC/CO_3 acceptance criteria defined in Table 1-5 for samples submitted under ASR 6192.

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: MW Urie **Date** 10-19-01

Review/Approval by: MJ Steele **Date** 10-22-01

Excel Archive File: ASR 6162 6192 HP.xls

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

PO Box 999, Richland, WA 99352

Client	I Burgeson	**Charge Code/Project:**	W58950 / 42365
RPL Numbers:	01-1613, 01-1842, 01-1843	**ASR Number:**	6162/6192
Analyst:	MJ Steele	**Analysis Date:**	9/26 & 10/04, 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
01-01613	Tc-AP1-EFF-Comp	90	3,450	350	3,450	90	6,900
01-01842	AP101L-E-Comp	90	40	180	180	90	220
01-01843	Tc-AP1-Eluate	90	80	180	300	90	380
01-01843 Dup	Tc-AP1-Eluate	90	150	180	250	90	400
	RPD				n/a		n/a
	RSD				n/a		n/a
01-01843 MS	Tc-AP1-Eluate				46%		101%
01-00973 MS(c)	AW101 Feed				98%		

n/a = not applicable (RPD not calculated, since results not >5x MDL)

(a) TIC MDL set to TC MDL

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

(c) Additional MS analyzed in TOC analysis batch

The TOC/TIC analyses of the samples submitted under ASRs 6162 and 6192 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 99% for TOC and 96% for TC.

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 25 µgC and the TC determination produced an average blank of 27 µgC for 5 blank determinations.

For each days analysis run, the QC for the analyses include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes. The ASRs indicate 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 as well as the additional QC requirement defined in Table 1-5 (ASR 6192). ASR 6192 provided more stringent duplicate precision acceptance criteria than the QA Plan (i.e,. <15% RSD versus <20% RPD); the LCS and matrix spike acceptance criteria are the same.

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

Duplicates: The precision between the duplicates for the TC, as demonstrated by the RPD and RSD could not be determined, since the sample selected for duplicate analysis had insufficient carbon concentration.

Matrix Spike: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The TC MS prepared from "Tc-AP1-Eluate" demonstrated a recovery of 101%; however, the TOC MS prepared from the same sample had a recovery of 46% (significantly below the acceptance criteria of 75% to 125%.) The reason for the low TOC recovery is unknown. Another TOC MS sample (01-00973) demonstrated a recovery of 98%.

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
01-01613	Tc-AP1-EFF-Comp	5,250	3,450	1,630	3,450	6,880	6,900
01-01842	AP101L-E-Comp	<11	40	<30	180	< 41	220
01-01843	Tc-AP1-Eluate	31	80	150	300	180	380

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 for sample "Tc-AP1-EFF-Comp." 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. The carbon concentrations in the other samples are to near the MDLs to compare; however, the furnace method measures higher concentrations than the hot persulfate method for all TIC, TOC, and TC.

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: MW Urie **Date** 12-19-01

Review/Approval by: MJ Steele **Date** 1-3-02

Raw Data Calculation/Archive Information:
ASR 6014 6104 5105 6106 6107 6155 6162 6192.xls

Test Plan: ASR 6162 & 6192

Wt% Oxides

Date: 10/15/2001

	Crucible Data		Tc-AP1-Eff-Comp (01-01613)			AP101L-E-Comp (01-01842)			Tc-AP1-Eluate (01-01843)		
Step #	Description	units	Comp	Comp-Dup	Comp-Trip	Comp	Comp-Dup	Comp-Trip	Eluate	Eluate-Dup	Eluate-Trip
1,2	Pre-fired Crucible Tare	g	16.6506	15.8066	15.8092	17.5248	16.3380	16.1260	15.6910	17.1335	16.8034
1,2	Crucible + wet material	g	17.8570	17.0363	16.4751	18.5238	17.2706	17.1273	16.6516	18.1011	17.3295
1,2	net wt. wet material	g	1.2064	1.2297	0.6659	0.9990	0.9326	1.0013	0.9606	0.9676	0.5261
8	Stable dry wt. @ 105 deg C.	g	17.0243	16.1838	16.0157	17.5282	16.3413	16.1292	15.6916	17.1339	16.8038
N/A	net dry wt. @ 105 deg C.	g	0.3737	0.3772	0.2065	0.0034	0.0033	0.0032	0.0006	0.0004	0.0004
13	Total Wt. after 1000-1050 deg C.	g	16.8558	16.0148	15.9233	17.5287	16.3400	16.1321	15.6921	17.1360	16.8065
N/A	Net Wt. after 1000-1050 deg C.	g	0.2052	0.2082	0.1141	0.0039	0.0020	0.0061	0.0011	0.0025	0.0031

			Tc-AP1-Eff-Comp (01-01613)			AP101L-E-Comp (01-01842)			Tc-AP1-Eluate (01-01843)		
Step	Description	units	Comp	Comp-Dup	Comp-Trip	Comp	Comp-Dup	Comp-Trip	Eluate	Eluate-Dup	Eluate-Trip
N/A	Wt% Sample after 105 deg C	%	31.0%	30.7%	31.0%	0.340%	0.354%	0.320%	0.062%	0.041%	0.076%
		Ave	30.9%			0.338%			0.060%		
		Std. Dev.	0.2%			0.017%			0.017%		

			Tc-AP1-Eff-Comp (01-01613)			AP101L-E-Comp (01-01842)			Tc-AP1-Eluate (01-01843)		
Step	Description	Units	Comp	Comp-Dup	Comp-Trip	Comp	Comp-Dup	Comp-Trip	Eluate	Eluate-Dup	Eluate-Trip
N/A	Wt% Oxides after 1050 deg C	%	17.0%	16.9%	17.1%	0.39%	0.21%	0.61%	0.11%	0.26%	0.59%
		Ave	17.0%			0.40%			0.32%		
		Std. Dev.	0.1%			0.20%			0.24%		

Analyst: F. Vaughn Hoopes 10-30-01

Review: 10/31/01

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

REVISION 1 (Correct 01-01613D sample ID on Results Page 1 of 2)

Project / WP#:	42365 / W55950
ASR#:	6162 and 6192
Client:	I. Burgeson
Total Samples:	3 (liquids)

	ASR 6162	ASR 6192
RPL#:	01-01613	01-01842, 01-01843
Client ID:	"TC-AP1-EFF-Comp"	"AP101L-E-Comp" "TC-AP1-Eluate"
Sample Preparation: PNL-ALO-128 (1 mL / 25mL)		

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

Analyst: D.R. Sanders

Analysis Date (File): 10-05-2001 (A0725)

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 11/13/01
Reviewed by

Jerry Wagner 11-13-01
Concur

One liquid sample submitted under Analytical Service Request (ASR) 6162 and two liquid samples submitted under ASR 6192 were prepared by acid digestion per PNL-ALO-128 (in the sample preparation laboratory). The samples were prepared by aliquotting 1.0 mL of sample, digesting per the procedure, and diluting to a final volume of 25 mL. These samples were processed in the same digestion processing batch. The preparation batch duplicate and matrix spike were prepared from the sample submitted under ASR 6162 (i.e., TC-AP1-EFF-Comp). Since the samples submitted under ASR 6162 and 6192 were processed in the same digestion batch and are similar matrices, no matrix spike or duplicate was prepared from the samples submitted under ASR 6192.

In Table 1-4 included with the ASR 6162 and Table 1-3 included with ASR 6192, all analytes measured by the ICPAES were identified as analytes of interest for this work, except Pd, Rh, Ru, Tl and W. The quality control (QC) results for each of the analytes of interest have been evaluated and are presented below. Analytes other than those identified as analytes of interest are reported, but have not been fully evaluated for QC performance and are reported for information only.

The attached ICPAES Results (2 pages) presents the final results. Results are from the direct measurement of the digestates at the ICPAES, except for Na which required an additional 5x dilution for sample TC-AP1-EFF-Comp to bring the Na concentration within the ICPAES linear range.

The following describes the QC measurement results relative to ICPAES analysis requirements of the controlling QA plan and the sample-specific QC Acceptance Criteria defined by Table 1-5 for ASR 6192. [Note: No additional sample-specific QC Acceptance Criteria were defined in ASR 6162.] A digestion processing blank, laboratory control sample (blank spike), matrix spike, and duplicate were prepared. The blank spike was prepared by using 3 mL of a custom multi-element solution "AZ-101 Spike" per a nominal 25-mL digestate volume, and the matrix spikes were prepared by using 1 mL of the same multi-element solution.

Process Blank:

Concentration of analytes of interest measured in the process blank were all within acceptance criteria of ≤ EQL (estimated quantitation level) or less than ≤5% of the concentration in the sample, except for Na, B, and Si. The B and Si demonstrated similar concentrations in both the process blank and the sample digestates, suggesting leaching of the glassware used for digesting the samples. The Na concentration of the samples submitted under ASR 6192 was low (i.e., 300 to 900 µg/mL) and the slight blank contribution (i.e., 180 µg/mL, most likely from leaching of the glassware) exceeded the ≤5% of the sample concentration acceptance criteria.

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, Na, and Si. The failure of the B, Na, and Si is attributed to the differences in the B, Na, and Si leached from the preparative glassware in the process blank and that leached in the BS sample. It should be noted that the BS sample did not contain As, Co, Dy, Eu, Sb, Se, Sn, Te, and Y.

Duplicate:

For those analytes of interest measured above the EQL, the relative percent differences (RPD) were within the acceptance criteria of less than 20%. ASR 6192 defined additional acceptance criteria (i.e.., <15% for all analytes other than Na and <3.5% for Na. The acceptance criteria of <15% was met for all analytes of interest defined in ASR 6192; however, the Na RPD of 5.4% failed the <3.5% criteria.

It should be noted that even for analytes with concentration between the method detection limit (MDL) and the EQL (identified in the Results as italic/bold results), the RPDs meet the acceptance criteria.

Matrix Spiked Sample:

A matrix spike was prepared for TC-AP1-EFF-Comp. The matrix spike recoveries for the analytes of interest fall into four categories. 1) those analytes for which a matrix spike element was not included in the matrix spiking solution (i.e., As, Co, Dy, Eu, Sb, Se, Sn, Te, and Y, 2) those analytes that have spike concentrations less than 20% of the sample concentration (i.e., Al, , Cr, K, and Na) making the spike recovery calculation meaningless, 3) those analytes that failed to meet the acceptance criteria of 75% to 125% (i.e., Cu and P), and 4) those analytes that demonstrated matrix spike recoveries within the acceptance criteria (i.e., all other analytes of interest). For those analytes falling into categories 1), 2), and 3), post spiking of the digestate (or in some cases serial dilution) is used to evaluate matrix interference issues.

Post-Spiked Samples (Spike A Elements; all analytes except Ce, Dy, Eu, La, Nd, Te, Th, U, and Sn):

A post spike was prepared for TC-AP1-EFF-Comp. All post-spiked analytes of interest (i.e., Spike A analytes of interest) were recovered within tolerance of 75% to 125%, except 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 Na was less than 20% of the sample concentration and the recovery results are considered meaningless. For Na, the use of serial dilution results is required to evaluate potential matrix interferences.

Post-Spiked Samples (Spike B Elements; Ce, Dy, Eu, La, Nd, Te, Th, and U):

Post spiking was performed on TC-AP1-EFF-Comp. The post spike recovery for all analytes of interest (i.e., Spike B analyte of interest) were within tolerance of 75% to 125%. It should be noted that Sn was not contained in either the Spike A or Spike B post spike solutions.

Serial dilution:

Serial dilution was required for Na, since the Na in the matrix spike and post spike were less than 20% of the sample concentration (i.e., recoveries could not be evaluated). Sodium for both TC-AP1-EFF-Comp and TC-AP1-Eluate demonstrated a percent difference (%D) within the acceptance criteria of ±10% after correcting for dilution for all the serial dilutions measured.

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=	25	25.0 (Na125.0)	25.0 (Na125.0)	25	25
	RPL#=	01-1613 PB	01-1613	01-1613 D	01-1842	01-1843
Det. Limit ug/mL	Client ID=	Proc.Blank ug/mL	TC-AP1-EFF-COMP ug/mL	TC-AP1-EFF COMP Dup ug/mL	AP101L-E-Comp ug/mL	Tc-AP1-Eluate ug/mL
	Analytes (Pretreatment Specification and Vitrification Request					
0.025	Ag	--	--	--	--	--
0.060	Al	[11]	4,860	5,200	18.6	[14]
0.250	As	--	--	--	--	--
0.050	B	164	169	170	194	177
0.010	Ba	--	[0.27]	[0.28]	[0.29]	--
0.010	Be	--	[0.72]	[0.76]	--	--
0.100	Bi	--	--	--	--	--
0.250	Ca	--	--	[6.3]	[25]	--
0.015	Cd	--	[1.2]	[1.3]	[1.2]	--
0.200	Ce	--	--	--	--	--
0.050	Co	--	--	--	--	--
0.020	Cr	--	102	109	12.0	--
0.025	Cu	--	--	--	[1.2]	--
0.050	Dy	--	--	--	--	--
0.100	Eu	--	--	--	--	--
0.025	Fe	--	[1.7]	[1.7]	[6.1]	[0.74]
2.000	K	--	21,900	22,700	[57]	--
0.050	La	--	--	--	--	--
0.030	Li	--	--	--	--	--
0.100	Mg	--	--	--	--	--
0.050	Mn	--	--	--	--	--
0.050	Mo	--	[9.8]	[10]	--	--
0.150	Na	180	89,400	94,400	912	353
0.100	Nd	--	--	--	--	--
0.030	Ni	[2.8]	[2.7]	[2.5]	[1.6]	[1.0]
0.100	P	--	272	285	--	--
0.100	Pb	--	[5.7]	[5.8]	[4.7]	--
0.500	Sb	--	--	--	--	--
0.250	Se	--	--	--	--	--
0.500	Si	140	288	331	145	133
1.500	Sn	--	[54]	[56]	--	--
0.015	Sr	--	--	--	--	--
1.500	Te	--	--	--	--	--
1.000	Th	--	--	--	--	--
0.025	Ti	--	--	--	--	--
2.000	U	--	--	--	[160]	--
0.050	V	--	--	--	--	--
0.050	Y	--	--	--	--	--
0.050	Zn	--	[3.6]	[3.7]	--	--
0.050	Zr	--	--	--	--	--
	Other Analytes Measured					
0.750	Pd	--	--	--	--	--
0.300	Rh	--	--	--	--	--
1.100	Ru	--	--	--	--	--
0.500	Tl	--	--	--	--	--
2.000	W	--	--	--	--	--

Note: 1) Overall error greater than 10-times detection limit is estimated to be within +/- 15%.
2) Values in brackets [] are within 10-times detection limit with errors likely to exceed 15%.
3) "--" indicate measurement is below detection. Sample detection limit may be found by multiplying "det. limit" (far left column) by "multiplier" (top of each column).

QC Performance

Criteria>	ASR 6162 <20% ASR 6192 <15% (Na <3.5)	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/-10%
QC ID=	01-1613 & 01-1613 D RPD (%)	01-1613 BS (AZ-101 Spike) %Rec	01-1613 MS (AZ-101 Spike) %Rec	01-1613 + Post Spike A %Rec	01-1613 + Post Spike B %Rec	01-1613 @1/@5 Serial Dil %Diff	01-1843 @1/@5 Serial Dil %Diff
Analytes (Pretreatment Specification and Vitrification Request							
Ag		106.7	107.1	100.0			
Al	6.7	97.3	n.r.	104.0		3.3	
As				103.1			
B	0.6	**40.3**	79.1	102.3		4.5	0.0
Ba	***5.3***	97.9	89.9	99.6			
Be	***5.7***	96.5	103.3	106.3			
Bi		99.8	110.5	99.7			
Ca		98.9	103.7	102.6			
Cd	***8.1***	99.4	98.3	104.4			
Ce		95.6	93.4		100.8		
Co				103.3			
Cr	6.1	99.6	n.r.	100.9		4.4	
Cu		105.5	**65.7**	102.9			
Dy					98.1		
Eu					105.8		
Fe	***5.2***	101.3	93.7	102.7			
K	3.4	95.5	n.r.	100.1		3.9	
La		95.8	93.7		95.7		
Li		101.3	96.2	100.8			
Mg		102.8	99.9	107.1			
Mn		100.4	95.6	104.3			
Mo	***4.4***	98.5	102.3	101.0			
Na	***5.4***	**67.1**	n.r.	n.r		2.5	4.4
Nd		95.8	93.4		96.9		
Ni	***6.1***	98.7	94.4	102.5			
P	4.7	96.8	**162.5**	97.2		2.3	
Pb	***1.7***	102.1	99.2	102.4			
Sb				102.3			
Se				102.6			
Si	14.0	**123.3**	99.7	109.5		4.3	-1.6
Sn	***3.4***						
Sr		113.6	120.9	100.3			
Te					101.0		
Th		97.6	94.2		98.6		
Ti		95.3	89.9	97.8			
U		97.0	91.2		96.6		
V		95.3	91.5	97.9			
Y				98.6			
Zn	***3.3***	100.5	107.2	113.3			
Zr		103.3	108.6	101.5			

Other Analytes Measured

Pd					100.2		
Rh					95.5		
Ru							
Tl				99.9			
W							

Shaded/bold results exceed acceptance criteria.

Bold/Italic RPD results calculated from values <EQL; for information only.

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

Battelle
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Date: 9/21/01

Subject: Hydroxide Analyses for: AP-101 Eluants
ASR: 6192 RPL # 01-1842 01-1843

To: Ingrid Burgeson

From: Bob Swoboda Swoboda 9/21/01

AP-101 tank waste eluants were analyzed in duplicate for the hydroxide content following procedure PNL-ALO-228. Direct sample aliquots were analyzed using a Brinkman 636 Auto-Titrator. Sample 01-1842 tested acidic and therefore could not be analyzed. 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 01-1843 sample and its replicate. The attached Report Summary shows average OH molarity (1st inflection point) of 0.0046M on the sample and replicate results with an RPD of 4%, which is excellent considering the very weak base concentration of the sample being titrated. The hydroxide ug/ml results averaged 79+/- 2.2 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 01-1843 was 100%. No hydroxide was detected in the reagent blank. The second inflection point frequently associated with carbonate, also showed an excellent RPD of 2%. 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.

Review: L.R. Greenwood 9-21-01

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

ASR **6192**

WP# **W58950**

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

Report Summary for ASR # -- 6192

				Concentration, moles					
RPG #	**Client ID**		**OH conc ug/mL**	**First Point**	**RPD**	**Second Point**	**RPD**	**Third Point**	**RPD**
01-1843	**AP-101-TC-AP1-Eluate**		8.1E+01	**0.0047**		**0.0027**		**none**	
01-1843	**AP-101-TC-AP1-Eluate**	**Rep**	7.7E+01	**0.0046**	4%	**0.0027**	2%	**none**	
		Ave	7.9E+01	4.6E-03		2.7E-03			
		std de	2.2E+00	1.3E-04		2.9E-05			

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

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: rg Swoboda 9/21/01

Reviewer: LRGreenwood 9/21/01

Battelle PNWD

PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 1

Project / WP#:	42365 / W58950
ASR#:	6192
Client:	Ingrid Burgeson
Total Samples:	2

Analysis: 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: 19 January 2004

Analysis Files:
Experiment – 01FEB02, 05FEB02
Procedure – 020201a, 020205a
Element Menu – 1FEB02, rbtc

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

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

Revised By 19Jan04
Date

Reviewed By 19Jan04
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).

Samples Submitted for Analysis:

RPL #	Client I.D.
01-1843	TC-AP1-Eluate
01-1842	AP101L-E-Comp

The samples (AP-101) 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 7 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

Tc-99 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
Blk-1613	6.94E-06	9.93E-07	1.03E-06
01-1843	4.13E-01	1.51E-02	2.33E-04
01-1842	1.37E-04	1.43E-05	2.00E-05
BS-1613	4.66E-04	7.10E-06	9.13E-07
MS-1613	9.81E-03	6.69E-05	1.55E-05

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 2% 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 (01-1613) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (01-1613) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD). Sample solution ICSD (01-1613) 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 (01-1613) 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 (01-1613) 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 (Blk-1613) 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%.

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%.

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). No LCS/BS was submitted for analysis. However a PBS was analyzed and all analytes of interest met the success criteria of +20%.

Duplicate (DUP). No Dup was submitted for analysis, however the ICS and ICSD met the success criteria of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD). No MS or MSD was submitted for analysis, however an ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

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

Battelle PNWD
PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 2

Project / WP#:	42365 / W58950
ASR#:	6192
Client:	Ingrid Burgeson
Total Samples:	5

Analysis: amu-241, Np-237, Pu-239 and Pu-240

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: 3 February 2004

Analysis Files: Experiment – 14FEB02
Procedure – 14FEB02
Element Menu – 14FEB02

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

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

Revised By 3 Feb 04
Date

Orville Thomas Farmer III 3Feb04
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	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).

Samples Submitted for Analysis:

RPL #	Client I.D.
01-1842	AP101L-E-COMP
01-1843	Tc-AP1-Eluate

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) amu-241, Np-237, Pu-239, and Pu-240. The MS and BS was not spike with the analytes of interest prior to sample dissolution however, an instrument post blank spike (PBS) and post matrix spike (PMS) was analyzed for each isotopes percent recovery.

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 7 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

AMU-241 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)	Dilution Factor	% Rec.
BLK-1613	<7.14E-06		7.14E-06	100	
01-1842	<1.60E-04		1.60E-04	2492	
01-1843	<1.49E-04		1.49E-04	2539	
BS-1613	<5.77E-06		5.77E-06	100	
PBS-1613	2.79E-02	2.66E-04	5.61E-06	100	112
MS-1613	<1.08E-04		1.08E-04	2000	
PMS-1613	2.64E-04	8.50E-05	6.20E-08	1	106

Np-237 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)	Dilution Factor	% Rec.
BLK-1613	5.38E-09	8.52E-10	5.28E-09	100	
01-1842	6.32E-07	7.89E-08	1.19E-07	2492	
01-1843	1.52E-07	1.49E-07	1.10E-07	2539	
BS-1613	1.97E-08	9.87E-09	4.26E-09	100	
PBS-1613	2.00E-04	1.53E-06	4.14E-09	100	114
MS-1613	4.10E-07	2.17E-08	7.97E-08	2000	
PMS-1613	1.86E-06	4.48E-08	4.58E-11	1	106

Pu-239 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)	Dilution Factor	% Rec.
BLK-1613	7.16E-06	6.97E-07	4.46E-07	100	
01-1842	7.72E-04	2.25E-05	1.00E-05	2492	
01-1843	9.90E-05	1.16E-05	9.33E-06	2539	
BS-1613	1.66E-05	3.04E-06	3.61E-07	100	
PBS-1613	1.70E-02	8.71E-05	3.51E-07	100	112
MS-1613	1.47E-04	3.40E-05	6.74E-06	2000	
PMS-1613	1.60E-04	3.50E-06	3.88E-09	1	105

Pu-240 Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)	Dilution Factor	% Rec.
BLK-1613	<1.08E-06		1.08E-06	100	
01-1842	1.35E-04	1.79E-05	2.43E-05	2492	
01-1843	<2.26E-05		2.26E-05	2539	
BS-1613	<8.74E-07		8.74E-07	100	
PBS-1613	1.31E-03	2.57E-05	8.49E-07	100	112
MS-1613	<1.63E-05		1.63E-05	2000	
PMS-1613	1.23E-05	3.58E-07	9.39E-09	1	105

3. Quality Control

3.1 Sample Preparation Batch QC Results

Preparation Blank (PB). The PB met the success criteria of < 10 X MDL for all isotopes except Pu-239.

Laboratory Control Standard / Blank Spike (LCS/BS). All isotopes of interest were not spiked into the LCS/BS sample at the time of sample preparation. However a Post Blank Spike (PBS) was analyzed and met the success criteria of +20%.

Matrix Spike (MS). All isotopes of interest were not spiked into the MS sample at the time of sample preparation. However, a post matrix spike (PMS) was analyzed met the success criteria of ±25%.

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

3.2. 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 isotopes of interest.

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

Internal Standard (IS). The Internal Standards met the QC criteria 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 AMU-241 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 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
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W58950
ASR#:	6192 / 128
Client:	Ingrid Burgeson
Total Samples:	2

Analysis: Pd, Ru, Rh, and Pr

Procedure: PNL-SCO1 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

Point of Contact: Orville Thomas Farmer III

Report Date: 16 April 2002

Analysis Files:
Experiment – 04FEB02, 21MAR02
Procedure – 020204a
Element Menu – multi

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

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 16 Apr 02
Reviewed By Date

TWilson 4.16.02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
01-01842	AP101L-E-COMP
01-01843	Tc-AP1-Eluate

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Pd, Ru, Rh, Pr.

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 7 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.

Both Ru and Pd were determined to be fission yield production; selected isotopes for each analyte were used after correcting for both atomic interferences and altered isotopic abundances.

2. Results

Pd Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
BLK-01613	<1.21E-04		1.21E-04
01-01842	5.95E-03	1.15E-02	3.22E-03
01-01843	1.30E+01	1.38E-01	4.00E-02
BS-01613	1.60E-01	1.71E-03	1.56E-03
MS-01613	3.17E+00	5.67E-02	3.08E-02

Ru Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
BLK-01613	<7.39E-05		7.39E-05
01-01842	3.23E-03	5.68E-04	1.96E-03
01-01843	<1.60E-03		1.60E-03
BS-01613	1.54E-01	1.35E-03	7.81E-05
MS-01613	5.27E+00	7.82E-02	1.31E-03

Rh Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
BLK-01613	<3.30E-05		3.30E-05
01-01842	5.73E-02	3.10E-03	8.77E-04
01-01843	2.31E-03	9.25E-04	7.14E-04
BS-01613	1.90E-01	1.56E-03	3.48E-05
MS-01613	4.65E+00	2.03E-02	5.84E-04

Pr Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
BLK-01613	<1.41E-05		1.41E-05
01-01842	5.29E-04	2.98E-04	3.74E-04
01-01843	4.23E-04	6.31E-04	3.04E-04
BS-01613	1.61E-01	2.29E-03	1.49E-05
MS-01613	3.46E+00	6.27E-02	2.49E-04

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 2% 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)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (01-01613) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (01-01613) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (01-01613) 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 (01-01613) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (01-01613) 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)

Narrative:

An instrument post spike was performed on sample (Blk-01613) 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)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

All analytes in the LCS/BS met the success criteria of ±20%. A PBS was analyzed and all analytes of interest met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

No sample duplicate was submitted for analysis, however, the ICS and ICSD also met the success criteria of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MSD was submitted for analysis. All elements met the success of ±25%. The ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM)

Narrative:

No LCS/SRM was submitted for analysis.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W58950
ASR#:	6192 / 128
Client:	Ingrid Burgeson
Total Samples:	2

Analysis: U and U-iso

Procedure: PNL-SCO1 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

Point of Contact: Orville Thomas Farmer III

Report Date: 30 March 2002

Analysis Files:
Experiment – 29NOV01
Procedure – 011129a
Element Menu – U

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

Analyst(s): James Bramson

Reviewed By 18Apr02
Date

Concur 4.18.02
Date

Samples Submitted for Analysis:

RPL #	Client I.D.
01-01843	Tc-AP1-Eluate
01-01842	AP101L-E-COMP

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) total U and U-isotopic.

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 the 3 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 3 standard blanks solutions by 6.96. 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

Total U Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
01-01843	1.77E-03	7.96E-05	7.99E-04
01-01842	1.92E+02	1.72E+00	7.82E-02

U-233 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
01-01843	<1.98E-07		1.98E-07
01-01842	1.80E-04	1.74E-05	1.94E-05

U-234 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
01-01843	<5.73E-08		5.73E-08
01-01842	1.00E-04	4.39E-06	5.61E-06

U-235 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
01-01843	<3.78E-10		3.78E-10
01-01842	3.57E-06	3.45E-08	3.69E-08

U-236 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
01-01843	<8.62E-10		8.62E-10
01-01842	7.25E-06	8.97E-08	8.43E-08

U-238 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
01-01843	<2.23E-10		2.23E-10
01-01842	6.38E-05	4.76E-09	2.18E-08

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

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)

Narrative:

The ICV/CCV standards for total U met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

Not analyzed.

Instrument Control Solution (ICS)

Narrative:

Not analyzed.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Not analyzed.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

Not analyzed.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (Blk-01613) 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)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

No LCS/BS was submitted for analysis, however a PBS was analyzed and all analytes of interest met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All elements met the success of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MS or MSD was submitted for analysis.

Laboratory Control Standard (LCS/SRM)

Narrative:

No LCS/SRM was submitted for analysis.

Battelle PNWD
PO Box 999, Richland WA 99352

Analytical Chemistry Group (ACG)

ICP/MS Data Analysis Report

Revision 1

Project / WP#:	42365 / W58950
ASR#:	6192
Client:	Ingrid Burgeson
Total Samples:	2

Analysis: I-129

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: 15 January 2004

Analysis Files:
Experiment 26NOV01
Procedure – 011126a
Element Menu – Iodine

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

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

Revised By — 19Jan04 Date

Reviewed By — 19 Jan 04 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).

Samples Submitted for Analysis:

RPL #	Client I.D.
01-1842	AP101L-E-COMP
01-1843	Tc-AP1-Eluate

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) I-129.

1. Analysis

The final results have been corrected for all laboratory preparation and dilutions performed on the sample during analysis. Batch QC samples were submitted under ASR-6162.

Instrument Detection Limits (IDL) and Method Detection Limit (MDL) were determined using 4 instrument standard blank solutions. The IDL was calculated by multiplying the observed standard deviation of the 4 standard blanks solutions by 4.54. An MDL is determined for each solution analyzed by multiplying the IDL by the internal standard drift ratio and that sample total dilution factor.

All samples solutions were modified with (1% HCl / 5 mM P-Cyanophenol) to reduce memory effects and stabilize the Iodine signal. The major interference for the determination of I-129 by ICP/MS is the atomic ion of Xe-129, which was subtracted using the Xe-131 atomic ion. This correction was applied to all sample solutions in the analytical run.

2. Results

I-129 Final Results

Samples	Sample Conc. µCi/g	+/- 1 σ	MDL µCi/g
01-1842	<5.48E-06		5.48E-06
01-1843	<5.39E-06		5.39E-06

3. Quality Control

3.1 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). No LCS/BS was submitted for analysis, however a PBS was analyzed and all analytes of interest met the success criteria of +20%.

Duplicate (DUP). All elements met the success of ± 20% RPD. The ICS and ICSD also met the success criteria of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD). No MS was submitted for analysis. However an ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

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

3.2 Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). The ICB/CCB standards are 1% HCl / 5 mM P-Cyanophenol 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.

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

Analytical results generated were influenced by an identified bias in the calibration and calibration verification standards. Additionally, traceability of the standard source material to certified reference material is questionable. The effect on the reported results needs to include this additional uncertainty. The bias in the standards caused results for I-129 to be biased high for this report. The effect on the results, based on the observed bias and additional uncertainty due to questionable standard source material, will widen the error band for these results. The error band that should be applied to these results is -100% to +100%.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W58950
ASR#:	6192 / 128
Client:	Ingrid Burgeson
Total Samples:	2

Analysis: Ta and Pt

Procedure: PNL-SCO1 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

Point of Contact: Orville Thomas Farmer III

Report Date: 30 March 2002

Analysis Files:
Experiment – 13Feb02
Procedure – 13Feb02
Element Menu – 13Feb02

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

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 15Apr02
Reviewed By Date

TWilson 4.15.02
Concur Date

Samples Submitted for Analysis:

RPL #	Client I.D.
01-01843	TC-AP1-Eluate
01-01842	AP101L-E-COMP

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Ta and Pt.

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 7 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

Ta Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
BLK-1613	3.55E-04	1.56E-04	4.71E-05
01-1843	3.88E-03	6.26E-04	1.14E-03
01-1842	2.76E-03	1.04E-03	1.23E-03
BS-1613	7.59E-02	9.68E-04	4.66E-05
MS-1613	1.18E+00	7.21E-03	7.21E-04

Pt Final Results

Samples	Sample Conc. (ug/g)	(+/-) 1 sigma	MDL (ug/g)
BLK-1613	1.05E-04	3.90E-05	3.33E-05
01-1843	8.71E-02	1.63E-03	8.06E-04
01-1842	3.03E-02	1.35E-03	8.67E-04
BS-1613	1.95E-01	5.74E-04	3.30E-05
MS-1613	3.92E+00	5.89E-03	5.11E-04

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

The ICB/CCB standards are 2% 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)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes.

Instrument Control Solution (ICS)

Narrative:

Sample solution (01-1613) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (01-1613) to determine the instrument solution preparation process.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (01-1613) 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 (01-1613) met the instrument QC success criteria of ± 20% RPD for all analytes.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (01-1613) 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)

Narrative:

An instrument post spike was performed on sample (Blk-1613) 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)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

The LCS/BS was submitted for analysis and failed the success criteria of ±20% for Ta. However, the PBS was analyzed and all analytes of interest met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

No duplicate was submitted for analysis. However the ICS and ICSD met the success criteria of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

No MSD was submitted for analysis. The MS failed the success of ±25% for Ta. However, an ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM)

Narrative:

No LCS/SRM was submitted for analysis.

Battelle, PNNL
Advance Inorganic Analysis Group (AIAG)

ICP/MS Data Analysis Report

Project / WP#:	42365 / W58950
ASR#:	6192 / 128
Client:	Ingrid Burgeson
Total Samples:	5

Analysis: Pu-242

Procedure: PNL-SCO1 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

Point of Contact: Orville Thomas Farmer III

Report Date: 25 April 2002

Analysis Files:
Experiment – 19 April 02
Procedure – 19 April 02
Element Menu – 19 April 02

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

Analyst(s): James Bramson / Teresa Wilson

Orville Thomas Farmer III 25 Apr 02
Reviewed By Date

Samples Submitted for Analysis:

RPL #	Client I.D.
BLK-1613	BLK
01-1842	AP101L-E-COMP
01-1843	Tc-AP1-Eluate
BS-1613	BS
MS-1613	MS

The samples (AP-101) submitted for analysis were analyzed on a radioactive-material-contained ICP/MS for the requested analyte(s) Pu-242. The analysis for Pu-242 was performed using the calibration count rate response from Pu-239. The MS and BS was not spike with any Pu material however, an instrument post blank spike and post matrix spike was analyzed for Pu-239 recovery.

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 7 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

Pu-242 Final Results

Samples	Sample Conc. (uCi/g)	(+/-) 1 sigma	MDL (uCi/g)
BLK-1613	<8.31E-08		8.31E-08
01-1842	<1.56E-06		1.56E-06
01-1843	<1.66E-06		1.66E-06
BS-1613	<6.10E-08		6.10E-08
MS-1613	<1.02E-06		1.02E-06

3. Quality Control

3.1. Instrument QC Results

Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

Narrative:

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)

Narrative:

The ICV/CCV standards met the QC criteria of ± 10% for all analytes.

Dilution Test (DT)

Narrative:

The success criteria of ± 20% recovery were met for this QC for all analytes. The isotope of Pu-239 was used for the instrument QC success criteria.

Instrument Control Solution (ICS)

Narrative:

Sample solution (01-01845 mod-2) is a replicate analysis of the original sample that was submitted for analysis and is compared with the ICSD solution (01-01845 mod-2) to determine the instrument solution preparation process. The isotope of Pu-239 was used for the instrument QC success criteria. The isotope of Pu-239 was used for the instrument QC success criteria.

Instrument Control Solution Duplicate (ICSD)

Narrative:

Sample solution ICSD (01-01845 mod-2) 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 (01-01845 mod-2) met the instrument QC success criteria of ± 20% RPD for all analytes. The isotope of Pu-239 was used for the instrument QC success criteria.

Instrument Control Solution Duplicate Spike (ICSDS)

Narrative:

The ICSDS is a post matrix spike of sample (01-01845 mod-2) 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. The isotope of Pu-239 was used for the instrument QC success criteria.

Post Blank Spike (PBS)

Narrative:

An instrument post spike was performed on sample (Blk-1613) and met the QC success criteria of ± 20% recovery for all analytes. The isotope of Pu-239 was used for the instrument QC success criteria.

Internal Standard (IS)

The Internal Standards met the QC criteria of 30% to 120%.

3.2. Sample Preparation QC Results

Preparation Blank (PB)

Narrative:

The PB met the success criteria being < 10 X MDL for all analytes.

Laboratory Control Standard / Blank Spike (LCS/BS)

Narrative:

Pu isotopes were not spiked into the LCS/BS sample at the time of sample preparation. However a Post Blank Spike (PBS) was analyzed for Pu-239 and met the success criteria of ±20%.

Duplicate (DUP)

Narrative:

All elements were <10X MDL, however the ICS and ICSD met the success criteria of ± 20% RPD.

Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

Narrative:

Pu isotopes were not spiked into the MS sample at the time of sample preparation
However an ICSDS (matrix post spike) was analyzed and all analytes met the success criteria of ±25%.

Laboratory Control Standard (LCS/SRM)

Narrative:

No LCS/SRM was submitted for analysis.

Appendix G

Cesium Ion Exchange and Batch Contacts Testing Personnel

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

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

Analytical Support
J. P. Bramson
L. P. Darnell
O. T. Farmer
S. K. Fiskum
L. R. Greenwood
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