PNWD-3202 WTP-RPT-032

# Effect of Eluant Flow Direction on the Elution Characteristics of SuperLig<sup>®</sup>-644 Ion Exchange Resin

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Test specification: TSP-W375-00-00034 Test plan: TP-PNNL-WTP-044 Test exceptions: None R&T focus area: Pretreatment, ion exchange Test Scoping Statement(s): None

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#### **Completeness of Testing**

This report describes the results of work and testing specified by TSP-W375-01-00002 Rev. 0 and TP-RPP-WTP-066 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 Date

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## Summary

## Objectives

Battelle –Pacific Northwest Division (PNWD) is contracted to Bechtel National Inc. (BNI) on the River Protection Project – Waste Treatment Plant (RPP-WTP) project to perform research and development activities. Unit operations of the WTP process include the separation of <sup>137</sup>Cs and <sup>99</sup>Tc from the liquid portion of the waste by ion exchange. SuperLig<sup>®</sup>644 (SL-644) and SuperLig<sup>®</sup>639 (SL-639) ion exchange resins were selected by the project to perform <sup>137</sup>Cs and <sup>99</sup>Tc separations, respectively.

Ion exchange testing needs were delineated by Barnes et al (2002) in the Research and Technology Plan. As described by Johnson (2000), the objective of the task described by this report was to investigate the effect of eluant flow direction on the volume of eluant required to elute the SL-644 ion exchange resin and the elution characteristics. All of the test objectives were achieved.

## **Conduct of Testing**

The investigation was performed using an apparatus consisting of a column containing a ~10 mL bed of SL-644 resin from batch 010319SMC-IV-73 that was plumbed to enable flow either up or down through the bed. The as-received resin was received dry and probably in the potassium form. The resin was immediately sieved after receipt and then ~10 mL from the size fraction 212  $\mu$ m to 425  $\mu$ m (note that the tests described in section 3 indicate that the resin volume is ~50% larger when submerged in simulated AW-101 LAW and in the Na form than when in the dry, as-received form) was loaded into the column approximately four months later. The resin was pre-conditioned with 0.5 M nitric acid and then rinsed with deionized (DI) water before commencing the simulated LAW processing operations. The test consisted of cycling the resin through a number of operations. A cycle commenced by contacting the resin with 0.25M NaOH to convert the resin to the Na form. Simulated low activity waste (LAW) from tank AW-101 containing sufficient Cs projected to provide 50% breakthrough after processing 150 bed volumes was then processed to load the resin with Cs. The column was rinsed with 0.1M NaOH and then DI water before elution with 0.5M nitric acid. Elution continued until the concentration of Cs in the eluant was less than 1% of that in the simulated LAW feed. The final operation was a column rinse with DI water.

## **Results and Performance Against Objectives**

The resin performance was poorer than desired in the first simulated LAW processing operation yielding a breakthrough (the effluent Cs concentration expressed as a percentage of that in the feed) of 50% after processing only 8 bed volumes (BVs). The poor performance was considered to be due to degradation from air oxidation while stored and / or inadequate acid pre-conditioning to remove potassium salts leftover from the resin manufacture. Therefore, the first cycle was repeated to investigate if the elution of cycle 1 would have improved performance. Breakthrough performance significantly improved in the second cycle, providing 2% breakthrough after processing ~10 BVs of simulated LAW. Clumping was observed during regeneration for the second cycle, raising the possibility that clumping during the first cycle, though not observed, contributed to the poor loading performance.

Down-flow and up-flow elution operations were then performed in two consecutive cycles. Final Cs breakthroughs after processing  $\sim$ 70 BVs of simulated LAWin the two cycles were approximately the same at  $\sim$ 22% such that comparison of the up-flow and down-flow elution profiles is appropriate. The elution profile is characterized by two phases. Typically  $\sim$ 95% of the Cs is eluted in the first phase and is

identified by the peak in the eluant Cs concentration when it is considered a function of the eluant volume. The second phase is identified by a relatively slow decline in the eluant Cs concentration when the remainder of the Cs is eluted. The target eluant Cs concentration of 1% of that in the LAW feed is achieved at some point in phase 2.

Elution of Cs from the ion exchange resin SL-644 when eluant is pumped in the down-flow direction is characterized by almost complete removal (~95%) of the Cs from the resin in approximately 1 BV of eluant. Nearly complete elution (~95%) was achieved in 5 BVs of eluant when it was pumped up-flow through the column. From this viewpoint, down-flow elution appears better since the Cs is eluted into a smaller volume. The difference in behavior between up-flow and down-flow elution is associated with mixing of the eluate in the head of liquid above the bed and consequent dilution of the eluted Cs. This hypothesis is supported by the good fit obtained by applying the continuously stirred tank mixing model to the data, albeit with qualifications associated with imperfect mixing and mass transfer and chemical equilibrium effects.

The objective of the testing was met: up-flow elution of the SL-644 resin is not more efficient than down-flow elution.

## **Quality Requirements**

PNWD implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP QA organization. This work was conducted to the quality requirements in NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, subpart 2.7 as instituted through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP).

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

### Issues

Eluant flow direction appeared not to have a significant impact on the tail of the elution profile and the total volume to achieve an eluant Cs concentration 1% of that in the simulated LAW feed. However, greater volumes of eluant were required to achieve eluant Cs concentrations 1% of those in the simulated LAW feeds in both cycles than previously required for this resin. Approximately 45 BVs were required for the down-flow elution cycle compared to less than 15 BVs required in previous tests. The poorer performance is hypothesized to be due to some characteristic of the bed induced by air oxidation during storage or insufficient pre-conditioning that is manifested in the long elution tail and that has persisted through subsequent cycles. Proper storage and rigorous pre-conditioning of the resin with nitric acid appear, therefore, essential for its optimal performance throughout its operational life.

# Acronyms and Abbreviations

AV	Apparatus volume
BNI	Bechtel National, Inc.
BV	Bed volume
DI	De-ionized
GEA	Gamma energy analysis
HLW	High level waste
IC	Ion chromatography
ICP-AES	Inductively coupled plasma – atomic emission spectrometry
ICP-MS	Inductively coupled plasma – mass spectrometry
λ	Number of bed volumes processed at 50% breakthrough
LAW	Low activity waste
RPP-WTP	River Protection Project – Waste Treatment Plant
SL	SuperLig <sup>®</sup>
SRTC	Savannah River Technology Center

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## 1.0 Introduction

## 1.1 Background

Battelle – Pacific Northwest Division (PNWD) is contracted to Bechtel National Inc. (BNI) on the River Protection Project – Waste Treatment Plant (RPP-WTP) project to perform research and development activities. The purpose of the RPP-WTP project is to design, construct, and commission a plant to treat and immobilize high-level waste (HLW) and low-activity waste (LAW) stored in underground storage tanks at the Hanford Site. Unit operations of the LAW treatment process include the separation of <sup>137</sup>Cs and <sup>99</sup>Tc from the liquid portion of the waste by ion exchange. SuperLig<sup>®</sup>644 (SL-644) and SuperLig<sup>®</sup>639 (SL-639) ion exchange resins were selected by the project to perform <sup>137</sup>Cs and <sup>99</sup>Tc separations, respectively, and are available from IBC Advanced Technologies, Inc. (American Fork, UT). PNWD and the Savannah River Technology Center (SRTC) have tested these resins with actual waste and shown that they satisfy the performance criteria delineated by the RPP-WTP project. The task described by this report is concerned with only the SL-644 resin.

The ion exchange process consists of a cycle of operations:

- Resin regeneration The cycle begins with the ion exchange sites of the resin occupied by H<sup>+</sup> ions, and these need to be replaced by Na<sup>+</sup> ions for Cs separation. This is achieved by contacting the resin with 0.25M NaOH.
- LAW processing The LAW is contacted with the resin to remove Cs.
- Column rinse The resin is rinsed with 0.1M NaOH and then water to prepare it for subsequent elution.
- Elution The Cs is recovered from the resin by eluting it with 0.5M HNO<sub>3</sub>. The Cs and Na ions are replaced by  $H^+$  ions at the ion exchange sites. The eluant containing the recovered Cs is forwarded for vitrification as HLW.
- Column rinse The resin is rinsed with water to prepare it for the regeneration operation of the subsequent cycle.

One parameter important to optimize is the volume of eluant required to recover the Cs from the resin because this is directly related to HLW process facility and equipment costs. The direction in which the eluant is pumped through the resin bed was considered by the RPP-WTP project as a parameter that could affect the required volume of eluant. In addition, one potential advantage of up-flow elution is that the eluant would more easily displace any gas bubbles from the resin bed.

## 1.2 Objectives

The objective of this task was to determine the effect of eluant flow direction on the volume of eluant required to elute the SL-644 ion exchange resin and the elution characteristics, as directed by the client (Johnson, 2000). The effect of eluant flow direction on the elution of other cations (e.g., Na and K) that may have been loaded onto the resin was not part of the scope although it could be accomplished by chemical analysis of samples taken during the tests.

Ion exchange testing needs were delineated by Barnes et al (2001) in the Research and Technology Plan. This investigation was conducted according to the test plan prepared by Fiskum (February, 2001) in response to the test specification provided by the client (Johnson, 2000).

### 1.3 Purpose

This report documents testing, results, and analysis associated with the eluant flow direction investigation. The purpose of the investigation was to provide information for an assessment of the eluant flow direction through the bed. The report is intended to aid the RPP-WTP project in decisions regarding the design and operation of the Cs ion exchange system in the WTP.

## 1.4 Quality Assurance

PNWD implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP QA organization. This work was conducted to the quality requirements in NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, subpart 2.7 as instituted through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP).

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

## 2.0 Test Design and Preparation

### 2.1 Simulated LAW Preparation

Tests were performed using a simulated LAW since using actual waste would have proved unacceptably expensive and impractical from a supply standpoint for the scale of the test. The LAW currently stored in tank 241-AW-101 was selected as that to simulate and test since processing of the LAW in this tank is scheduled for the WTP and the performance of the SL-644 in this matrix has been extensively tested. The recipe for preparing this simulated LAW was taken from Golcar et al. (2000). The recipe was used with no modifications, except for the concentrations of Mn and Cs, and is presented in Table 2.1. The published Mn concentration is erroneous and the recipe was modified to include the concentration intended by the authors. The Cs concentration was modified from the recipe value so that the resin would become fully loaded within the test duration and this is discussed in more detail below. The solution was filtered immediately prior to processing and then spiked with <sup>137</sup>Cs so that the Cs behavior could be monitored by gamma spectroscopy.

	Final target
Species	concentration (M)
EDTA	3.70E-03
Citric acid	3.70E-03
Na <sub>3</sub> HEDTA·2H <sub>2</sub> O	3.70E-03
Na <sub>3</sub> NTA	3.70E-03
NaGluconate	3.70E-03
Na <sub>2</sub> Iminodiacetate	3.70E-03
$Fe(NO_3)_3 \cdot 9H_2O$	5.00E-05
$Mg(NO_3)_2 \cdot 6H_2O$	1.50E-03
Mn(NO <sub>3</sub> ) <sub>2</sub> , 50%	6.63E-05
MoO <sub>3</sub>	2.86E-04
$Ni(NO_3)_2 \cdot 6H_2O$	1.33E-04
SiO <sub>2</sub>	2.93E-03
BaNO <sub>3</sub>	1.33E-04
CsNO <sub>3</sub> See below	
Ca(NO <sub>3</sub> ) <sub>2</sub> 4.13E-04	
Sr(NO <sub>3</sub> ) <sub>2</sub> 1.30E-05	
RbNO <sub>3</sub> 1.00E-05	
LiNO <sub>3</sub>	5.51E-04
КОН	4.30E-01
NaOH	3.89E+00
$Al(NO_3)_3 \cdot 9H_2O$	5.06E-01
Na <sub>2</sub> CO <sub>3</sub>	1.00E-01
Na <sub>2</sub> SO <sub>4</sub> 2.36E-03	
NaHPO <sub>4</sub> ·7H <sub>2</sub> O	1.73E-03
NaCl	6.93E-02
NaF	1.10E-02
NaNO <sub>2</sub>	7.90E-01

Table 2.1. Simulated AW-101 LAW Component List

The Cs concentration of the simulated LAW was to be selected to provide 50% breakthrough when the equivalent of 150 bed volumes (BVs) of waste had been processed through the column. For ideal ion exchange performance, the number of bed volumes of waste processed at 50% breakthrough is the column distribution coefficient, designated by  $\lambda$ , given by the expression,

$$\lambda = K_{d} \frac{M}{V_{b}}$$
(1)

Here, M is the mass of dry resin in the Na form and  $V_b$  is the volume of resin in 0.25M NaOH. This ratio was determined to be 0.22 g/mL in a column test by Fiskum (2002) although at the time of this test only a preliminary value of 0.269 g/mL was available.  $K_d$  is the batch equilibrium coefficient. Batch contact data for simulated AW-101 LAW was obtained by Fiskum (2002). However, only preliminary data were available at the time of this test and the batch equilibrium coefficient,  $K_d$ , was fitted to the correlation,

$$K_d = 589 Log_{10} \left( \frac{[Na]}{[Cs]} \right) - 1770$$
 (2)

A first approximation to the Cs concentration providing 50% breakthrough after processing 150 bed volumes of waste was calculated by considering equations 1 and 2. For the  $M/V_b$  value of 0.269, a K<sub>d</sub> of 560 is required for a  $\lambda$  value of 150 and from equation 2, the required Cs concentration is 5.54E-04 M for a solution containing 5M Na. However, resin performance proved very poor, as described later, when this Cs concentration was used and RPP-WTP and PNWD staff decided to repeat the test with a second batch of simulated LAW at a lower Cs concentration of 2.36E-4M.

The simulated AW-101 LAW feeds were analyzed by ion chromatography (IC), inductively coupled plasma – atomic emission spectrometry (ICP-AES), inductively coupled plasma – mass spectrometry (ICP-MS) and carbon oxidation using hot persulfate methods. Appendix A contains the analytical reports and Table 2.2 and Table 2.3 compare the target constituent concentrations with those determined by the appropriate analysis methods for the simulated LAW feeds for the down-flow and up-flow elution tests, respectively. The overall error for analytical results is estimated to be within 15% except those ICP-AES results in italics that are within ten times their detection limits with errors likely exceeding 15%.

In general, the ICP-AES results are consistent with the target concentrations given the measurement errors. However, analytical results for Al and K were consistently below their target concentrations even taking account of the analytical error. This is considered likely due to water vapor in the air becoming absorbed into the reagents and giving rise to artificially high formula weights. Analytical results for Si are ~5 times higher than the target concentrations likely due to contamination from the glassware used to perform the acid digests preceding analysis.

IC analysis was only performed on the down-flow elution test feed. IC results are generally consistent with the target concentrations except for F which was measured to be  $\sim$ 4 times higher than the target. The high F value might have been due to interference from acetate in the IC measurement.

Constituent	Analysis mothed	Target concentration	Analyzed
Constituent	Analysis method	(M)	concentration (M)
Al	ICP-AES	5.06E-1	3.67E-1
Ba	ICP-AES	1.33E-4	1.20E-4
Ca	ICP-AES	4.13E-4	9.25E-4
Cl	IC	6.93E-2	7.85E-2
Cs	ICP-MS	2.36E-4	2.18E-4
F	IC	1.10E-2	4.26E-2
Fe	ICP-AES	5.00E-5	2.29E-4
K	ICP-AES	4.30E-1	3.87E-1
Na	ICP-AES	5.00E-0	5.17E-0
Li	ICP-AES	5.51E-4	6.00E-4
Mn	ICP-AES	6.63E-5	1.18E-4
Мо	ICP-AES	2.86E-4	2.98E-4
NO <sub>2</sub>	IC	7.90E-1	9.71E-1
NO <sub>3</sub>	IC	1.52E-0	1.55E-0
Ni	ICP-AES	1.33E-4	1.38E-4
Р	ICP-AES	1.73E-3	2.11E-3
PO <sub>4</sub>	IC	1.73E-3	<5.26E-3
Rb	ICP-MS	1.00E-5	2.66E-5
$SO_4$	IC	2.36E-3	<5.21E-3
Si	ICP-AES	2.93E-3	9.43E-3
Sr	ICP-AES	1.30E-5	2.27E-5
Total inorganic	Hot persulfate	1.00E-1 (as CO <sub>3</sub> <sup>-</sup> )	1.14E-1
carbon			
Total organic carbon	Hot persulfate	1.55E-1	1.26E-1
Note: Results in italic	es are within ten times the	ir detection limit with erro	rs likely exceeding 15%.

 Table 2.2.
 Comparison of Analyzed and Target Simulated LAW Composition (down-flow elution test)

Constituent	Analysis method	Target concentration (M)	Analyzed concentration (M)
Al	ICP-AES	5.06E-1	4.85E-1
Ba	ICP-AES	1.33E-4	1.23E-4
Ca	ICP-AES	4.13E-4	7.50E-4
Cs	ICP-MS	2.36E-4	2.27E-4
Fe	ICP-AES	5.00E-5	2.39E-4
Κ	ICP-AES	4.30E-1	3.74E-1
Na	ICP-AES	5.00E-0	5.09E-0
Li	ICP-AES	5.51E-4	6.14E-4
Mn	ICP-AES	6.63E-5	5.82E-5
Мо	ICP-AES	2.86E-4	2.82E-4
Ni	ICP-AES	1.33E-4	1.30E-4
Р	ICP-AES	1.73E-3	2.08E-3
Rb	ICP-MS	1.00E-5	2.66E-5
Si	ICP-AES	2.93E-3	9.43E-3
Sr	ICP-AES	1.30E-5	2.16E-5
Note: Results in itali	cs are within ten times the	ir detection limit with erro	rs likely exceeding 15%.

Table 2.3. Comparison of Analyzed and Target Simulated LAW Composition (up-flow elution test)

### 2.2 Reagent Preparation

All reagents were 'reagent grade'. Sodium hydroxide solutions were prepared by dissolving the required mass of sodium hydroxide pellets in DI water. The solution of 0.5M HNO<sub>3</sub> was prepared by diluting the 68 - 70 wt% HNO<sub>3</sub> commercial stock with DI water.

### 2.3 Ion Exchange Resin Preparation and Storage

SL644 Cs ion exchange resin from batch 010319SMC-IV-73 was received in late March of 2001 in a dry, probably K form with  $K_2CO_3$  salt residual from its manufacture. The resin was immediately sieved to produce fractions with defined size ranges that were stored in plastic bottles. Ion exchange resin characteristics were determined by Fiskum (2002) and are reported here for completeness. Table 2.4 presents the weight distribution determined from the sieving operation.

ASTM Sieve size	Particle size (µm)	Weight fraction (%)
18	>1000	0.06
30	600 - 1000	37.27
40	425 - 600	38.23
50	300 - 425	18.01
70	212 - 300	6.08
100	150 - 212	0.26
140	106 - 150	0.06
>140	<106	0.03

Table 2.4. Dry Weight Distribution of SL644 Resin Batch 010319SMC-IV-73

The fraction defined by a particle size range of 212  $\mu$ m to 425  $\mu$ m, or 24.09% of the total weight, was used throughout these tests for consistency with the size range used by Kurath et al (2000). Note that the tests described in section 3 indicate that the resin volume is ~50% larger when submerged in simulated AW-101 LAW and in the Na form than when in the dry, as-received form. Table 2.3 presents various properties of the as-received resin and the resin in the size range 212 – 425  $\mu$ m.

Property	Value (as- received)	Value (212 – 425 μm)
Bulk density	0.84 g/mL	0.70 g/mL
F factor (for water loss)	0.871	0.877
L factor (solids fraction remaining after conversion to $H^+$ form)	0.556	0.538
I factor (mass increase from H <sup>+</sup> form to Na <sup>+</sup> form)	1.22	1.25

Table 2.3. Physical Properties of Batch 010319SMC-IV-73 SL644 Resin

The F factor indicates the loss in mass from drying the as-received resin at 50°C under vacuum to constant mass and is defined by the equation

$$F = \frac{m_d}{m_i} \tag{3}$$

where  $m_d = mass$  of as-received resin dried at 50°C under vacuum  $m_i = initial mass$  of as-received resin

The L factor indicates the loss in mass from acid washing (corrected for water loss) and is determined from the equation

$$L = \frac{(m_H F_H)}{(m_I F)} \tag{4}$$

where  $F_H = F$  factor for the H<sup>+</sup> form resin

 $m_i$  = initial mass of the as-received resin

F = F factor of the as-received resin.

The I factor defines the mass increase upon conversion from the  $H^+$  form to the Na<sup>+</sup> form and is determined from the following equation

$$I = \frac{m_{Na}}{m_H F_H} \tag{5}$$

where  $m_{Na}$  is the dry mass of the Na<sup>+</sup> form resin.

The color of the  $212 - 425 \mu m$  fraction was observed to have changed from red - black when it was received in March 2001 to gray when it was loaded in the column in July 2001. The color change may be indicative of atmospheric oxidation since another fraction stored in a bottle with a smaller headspace had retained its original color. Aging of the resin also appears to have been manifested in the mechanical properties of the resin, specifically its volume change when in different solutions. Fiskum (2002) reported resin in the  $212 - 425 \mu m$  range occupied 20% less volume in 0.5M nitric acid than in 0.25M NaOH shortly after it was received. However, results reported herein indicate that it occupies ~50% less volume approximately 4 months after receipt.

## 2.4 Ion Exchange Column Test Setup

A process schematic of the apparatus is provided in Figure 2.1. The apparatus consists of an ion exchange column of internal diameter 1.5 cm and height 20 cm containing nominally 10 mL of SL-644 resin expanded in simulated LAW, a metering pump, pressure relief valve, pressure gage and six 3-way valves.



Figure 2.1. Eluant Flow Direction Test Schematic

The column is a Spectrum Chromatography Spectra/Chrom<sup>®</sup> column manufactured from glass with plastic plungers on the ends that can be adjusted to control the distance between the top of the resin bed and the column feed. The space between the bed top and column feed was always flooded.

The pump is a Fluid Metering, Inc. (FMI) piston pump with the flow rate controlled using a FMI stroke rate controller. The pump was pre-calibrated using water and could provide pumping rates between approximately 0.5 mL/h and 50 mL/h.

The pressure relief valve was set to open at a pressure of 10 psi, which is below the maximum operating pressure of the column. Valves 1 and 2 placed between the pump outlet and column were used to eliminate air from the system or isolate the column from the pump. Valve 6 was used to prevent the column from draining while the pump was stopped. Valves 2 through 5 were used to direct the flow through the column in either an upward or downward direction. The equipment and fittings were connected using 1/16 inch internal diameter polyfluorinated plastic tubing.

## 3.0 Test Operation

## 3.1 Bed Volume (BV) Definition

Solution volumes and flow rates are reported relative to the volume of resin measured in 0.25M NaOH, typically the regeneration operation at the beginning of each cycle.

## 3.2 Resin Conditioning

Resin conditioning was performed in July, 2001 and testing was completed in early August, 2001. Asreceived SL-644 resin, believed to be in the K form, of mass 4.009g and dry bed volume 7 mL was washed by contacting it with de-ionized water for 16 hours and 50 minutes in a beaker. The resin was then transferred to the column using DI water to form a bed with a volume of 8.85 mL. The ion exchange resin was conditioned in the column with 0.25M NaOH and 0.5M HNO<sub>3</sub> solutions before cycle testing to remove any potassium salts remaining from its manufacture. The conditioning operation is outlined in Table 3.1.

	Bed volume	Volume		Flo	w rate
Reagent	(mL)	(mL)	(BV)	(mL/h)	(BV/h)
0.25M NaOH	11.7	108	9.2	40	3.4
DI water	12.4	112	9.6	37	3.2
0.5M HNO <sub>3</sub>	8.1	100	8.5	44	3.8
DI water	8.0	95	8.1	47	4.0

 Table 3.1.
 Column Conditioning Operations

A total apparatus volume of 23 mL was measured on the first step by monitoring the pH of the effluent and the volume of collected effluent.

## 3.3 Column Operation Overview

The test consisted of a number of operational cycles. A cycle commenced with converting the resin to the sodium form by pumping 0.25M NaOH through the bed. The simulated LAW was then processed, followed by column rinses of nominally the equivalent of two apparatus volumes (AVs) each of 0.1M NaOH and DI water before the resin was eluted with 0.5M HNO<sub>3</sub>. Elution was terminated when the <sup>137</sup>Cs concentration in the eluant was less than 1% of that in the simulated LAW feed. The cycle finished with a rinse of nominally 2 AVs of DI water. Appendix B contains the test operation data and calculations associated with breakthrough and elution performance evaluation.

### 3.3.1 Cycle 1 Operation (down-flow elution)

Details regarding the operation of the first cycle are provided in Table 3.2. Volumes were calculated from the mass of effluent collected and assuming the density of the effluent to be the same as that of the feed. This assumption is generally good except for the feed displacement operation where approximately half of the effluent would be simulated LAW. The waste processing operation of this cycle was prematurely terminated due to high initial breakthrough. The results from this cycle were not considered appropriate for forming conclusions regarding the eluant flow direction since the resin loading was not typical of the historical performance of the resin.

Operation	Reagent	Measured resin bed volume	Total volume of reagent		Flow rate of reagent	
		mL	mL	BV	mL/h	BV/h
Regeneration	0.25M NaOH	12.2	89.0	7.3	29.7	2.4
Waste	Simulated AW-	11.5	617	50.6	30.9	2.5
processing	101					
Feed	0.1M NaOH	NM	68.1	5.6	34.1	2.8
displacement						
Rinse	DI water	NM	61.4	5.0	30.7	2.5
Elution	0.5M HNO <sub>3</sub>	NM	143	11.7	8.59	0.7
Rinse	DI water	NM	Pumped at	nominally	/ 30 mL/h	for 2
			hr.			
NM: not measured						

Table 3.2. Cycle 1 Operational Details

Figure 3.1 presents the Cs breakthrough profile on probability – normal axes for cycle 1 and shows breakthrough of 50% was achieved after processing only  $\sim$ 8 BVs of simulated LAW.



**Figure 3.1**. Cycle 1 Cesium Breakthrough Performance (SL644 resin batch SMC-010319SMC-IV-73, nominal 3 BV/h, ~5.54E-04 M Cs, 5M Na, ambient conditions, BV = 12.2 mL in 0.25M NaOH and 11.5 mL in simulated AW-101 LAW)

The elution profile is presented in Figure 3.2 and shows that the target ratio of 0.01 for Cs concentrations in the feed and eluate was achieved after processing  $\sim 10$  BVs of eluant. This is consistent with previous tests by Fiskum et al (2002). The top  $\sim 75\%$  of the resin bed was observed to have pulled away from the column walls during elution. However, the consistency of these results with those of previous workers (e.g. Fiskum et al (2002) indicate that no significant channeling occurred.



**Figure 3.2**. Cycle 1 Down-flow Elution Profile (SL644 resin batch SMC-010319SMC-IV-73, nominal 1 BV/h, ambient conditions, BV = 12.2 mL in 0.25M NaOH)

### 3.3.2 Cycle 2 Operation (Down-flow elution)

Table 3.3 presents the operational details of cycle 2. The elution operation of cycle 1 would have provided additional conditioning of the resin that may minimize the impacts of degradation and/or further remove potassium salts leftover from its manufacture to thereby improve its performance. Therefore, cycle 2 was performed to check if breakthrough performance would improve. Regeneration of the resin bed appeared to be uneven and the bed surface appeared 'clumpy'. Therefore, regeneration was temporarily suspended while the bed was fluidized. This apparent clumping and the observation of resin pulling away from the column walls during the cycle 1 elution raise the possibility that there was clumping in the bed and resultant channeling during cycle 1 loading. Clumping was not apparent at the time, however.

Operation	Reagent	Measured resin bed volume	Total volume of reagent		Flow rate of reagent	
		mL	mL	BV	mL/h	BV/h
Regeneration	0.25M NaOH	12.0	82.6	6.9	13.8	1.2
Waste	Simulated AW-	NM	101	8.4	28.9	2.4
processing	101					
Feed	0.1M NaOH	NM	68.6	5.7	34.3	2.9
displacement						
Rinse	DI water	NM	60.6	5.1	30.3	2.5
Elution	0.5M HNO <sub>3</sub>	NM	159	13.3	10.1	0.8
Rinse	DI water	8.5	61.0 5.1		30.5 2.5	
NM: not measure	d					

Table 3.3. Cycle 2 Operational Details

The breakthrough and elution profiles were not monitored. However, 2% breakthrough was measured at the conclusion of the waste processing operation or after ~8 BVs of simulated LAW had been processed. This result compares with ~50% breakthrough after the same number of bed volumes had been processed in cycle 1 and indicates that the additional acid washing and/or bed fluidization/repacking greatly improved the resin bed performance. The target Cs concentration ratio in the eluate and feed was measured at 0.003, confirming that the target of 0.01 was achieved.

### 3.3.3 Cycle 3 Operation (down-flow elution)

RPP-WTP and PNWD staff decided to reduce the Cs concentration from nominally 5.54E-04 M to 2.36E-04 M in the simulated LAW feed for this cycle in an effort to reduce the breakthrough. The operational details of cycle 3 are presented in Table 3.4. A full cycle was performed following the apparent confirmation in cycle 2 that the resin bed was better conditioned. The top  $\sim 10\%$  of the resin bed was observed to have pulled away from the column walls during waste processing but no further detachment was observed during elution.

The <sup>137</sup>Cs activity balance presented in Table 3.5 shows that approximately 95% of the Cs fed to the system loaded onto the resin and was subsequently eluted. The apparent ~6% over-recovery of <sup>137</sup>Cs in the effluents is probably due to experimental uncertainty.

Operation	Reagent	Measured resin bed volume	Total vol reage	ume of ent	Flow rea	rate of gent
		mL	mL	BV	mL/h	BV/h
Regeneration	0.25M NaOH	11.5	56.6	4.9	8.71	0.8
Waste	Simulated AW-	10.1	840	73	30.0	2.6
Feed displacement	0.1M NaOH	11.2	68.7	6.0	34.4	3.0
Rinse	DI water	11.5	62.1	5.4	31.1	2.7
Elution	0.5M HNO <sub>3</sub>	7.8	496	43	9.82	0.9
Rinse	DI water	7.8	237	21	12.0	1.0

Table 3.4. Cycle 3 Operational Details

 Table 3.5.
 <sup>137</sup>Cs Activity Balance for Cycle 3

Process Stream	Total Count Rate (CPM)	Fraction of feed (%)
Simulated LAW Feed	4.94E5	100
Simulated LAW effluent (bottle)	2.24E4	4.5
Feed displacement effluent	4.06E3	0.8
Rinse effluent	1.55E2	0.0
Elution effluent	4.95E5	100.3
Rinse effluent	2.39E2	0.0
Total recovery of feed <sup>137</sup> Cs in effluents	5.22E5	105.8

Figure 3.3 shows that resin performance had significantly improved over that observed in cycle 1 with a final breakthrough of 22% after 70 BVs of simulated LAW had been processed when the operation was terminated.

Elution performance was apparently poorer than that observed in cycle 1. The elution profile presented in Figure 3.4 shows that the Cs concentration in the eluant reduced to 1% of that in the simulated LAW feed after ~45 BVs of eluant had been processed in contrast to ~11 BVs observed in cycle 1. Previous tests by Fiskum et al (2002) also achieved eluant Cs concentrations 1% of that in the LAW feed in less than 15 BVs. Counting and weighing uncertainties introduced an error of less than ~5% to the activity concentrations so that the undulations in the profiles appear to be significant. The two maxima observed in the tail of the elution profile may be due to the spontaneous breakage of resin clumps with Cs elution then occurring from the freshly exposed sites. However, breakage of resin clumps were not visually observed. The reason for resin clumping is not apparent. However, the fact that the bed was fluidized prior to processing simulated LAW in cycle 2 may be an important factor why poor elution performance was not observed during that cycle. Some characteristic of the degraded resin that is manifested in the long elution tail is hypothesized to have persisted through subsequent cycles.



**Figure 3.3**. Cycle 3 Cesium Breakthrough Performance (SL644 resin batch SMC-010319SMC-IV-73, nominal 3 BV/h, ~25 ppm Cs, 5M Na, ambient conditions, BV = 11.5 mL in 0.25M NaOH and 10.1 mL in simulated AW-101 LAW)



**Figure 3.4**. Cycle 3 Down-flow Elution Profile (SL644 resin batch SMC-010319SMC-IV-73, nominal 1 BV/h, ambient conditions, BV = 11.5 mL in 0.25M NaOH and 7.8 mL in 0.5M nitric acid)

### 3.3.4 Cycle 4 Operation (up-flow elution)

Table 3.5 presents the operational details for cycle 4. Elution was performed in the up-flow direction. The space between the bed top and plunger is a mechanical design characteristic that makes the column axially asymmetric and thereby may have prejudiced comparison of up and down flow elution. During down-flow elution, the effect of this space would be to initially dilute the acid entering the bed and thereby presumably to delay elution until a critical concentration had been attained. Adjustment of the plunger to reduce the headspace was not considered necessary in down-flow elution since the character of elution once begun would not have been affected. However, the top plunger was re-positioned after  $\sim$ 3 BVs of eluant had been fed to the column to reduce the volume between it and the bed top to  $\sim$ 2 mL during up-flow elution to minimize the head of liquid above the bed. The resin was not observed to have pulled away from the walls to any significant extent.

The <sup>137</sup>Cs activity balance presented in Table 3.6 shows that, similar to the performance observed in cycle 3, approximately 98% of the Cs in the simulated LAW fed to the system was separated and then eluted. Experimental error is again probably responsible for the apparent  $\sim$ 7% under-recovery of Cs in the effluents.

A Cs breakthrough of ~22% was measured when the operation was terminated after processing 70 BVs of simulated LAW as indicated in Figure 3.5. Although the final breakthrough was identical to that observed in cycle 3, performance early in the operation was better. For example, breakthroughs of 1% and less than 0.1% were observed in cycles 3 and 4, respectively after processing 30 BVs of simulated LAW.

The long elution tail was again observed in cycle 4. Again, counting and weighing uncertainties introduced an error of less than  $\sim$ 5% to the activity concentrations so that the undulations in the profiles appear to be significant and possibly due to the breakage of resin clumps. Figure 3.6 shows that the Cs concentration in the eluant was 1.2% of that in the simulated LAW feed after  $\sim$ 38 BVs of eluant had been processed when elution was terminated. At the same point in the cycle 3 elution, the Cs concentration in the eluant was  $\sim$ 2.4% but the apparent improvement in cycle 4 is not considered significant given the unexpectedly long elution tail possibly associated with degraded resin.

Operation	Reagent	Measured resin bed volume	Total vol reage	ume of ent	Flow reas	rate of gent
		mL	mL	BV	mL/h	BV/h
Regeneration	0.25M NaOH	11.3	58.4	5.2	9.7	0.9
Waste processing	Simulated AW- 101	9.9	815	72	31.9	2.8
Feed displacement	0.1M NaOH	NM	68.3	6.0	34.2	3.0
Rinse	DI water	NM	60.4	5.3	30.2	2.7
Elution	0.5M HNO <sub>3</sub>	NM	432	38	10.0	0.9
Rinse	DI water	7.3	62.7	5.5	31.1	2.8
NM: not measure	d	•				

Process Stream	Total Count Rate (CPM)	Fraction of feed (%)
Simulated LAW Feed	4.92E5	100
Simulated LAW effluent	7.35E3	1.5
Feed displacement effluent	4.73E3	1.0
Rinse effluent	1.43E2	0.0
Elution effluent	4.46E5	90.7
Rinse effluent	1.75E3	0.4
Total recovery of feed <sup>137</sup> Cs in effluents	4.60E5	93.5

 Table 3.6.
 <sup>137</sup>Cs Activity Balance for Cycle 4



Bed volumes of modified simulated AW-101 LAW processed (volume of bed measured in 0.25M NaOH solution)

**Figure 3.5**. Cycle 4 Cesium Breakthrough Performance (SL644 resin batch SMC-010319SMC-IV-73, nominal 3 BV/h, ~25 ppm Cs, 5M Na, ambient conditions, BV = 11.3 mL in 0.25M NaOH and 9.9 mL in simulated AW-101 LAW)



**Figure 3.6**. Cycle 4 Elution Profile (SL644 resin batch SMC-010319SMC-IV-73, nominal 1 BV/h, ambient conditions, BV = 11.3 mL in 0.25M NaOH)

## 4.0 Results Analysis

The elution operation has been characterized by two phases in previous tests of SL-644. Typically  $\sim$ 95% of the Cs is eluted in the first phase and is identified by the peak in the eluant Cs concentration when it is considered a function of the eluant volume. The second phase is identified by a relatively slow decline in the eluant Cs concentration when the remainder of the Cs is eluted. The target eluant Cs concentration of 1% of that in the LAW feed is achieved at some point in phase 2. These characteristics were repeated in the suite of tests described by this report.

The similarity between the breakthrough profiles of cycles 3 and 4, compared in Figure 4.1, and that similar quantities of Cs (23.0 mg and 24.3 mg in cycles 3 and 4, respectively) were separated onto the resin implies that comparison of their respective elution profiles is appropriate. Cycle 3 and 4 elutions were performed in the down and up flow directions, respectively.



Figure 4.1. Comparison of breakthrough profiles from cycles 3 and 4 (SL644 resin batch SMC-010319SMC-IV-73, nominal 3 BV/h, ~25 ppm Cs, 5M Na, ambient conditions)

Comparison of the elution profiles from cycles 3 and 4 in Figure 4.2 show that the elution peak was broader and shallower for up-flow elution. This observation is more distinct when the profiles are plotted in terms of the cumulative fraction of eluted Cs as presented in Figure 4.3. The number of eluant bed volumes is normalized by subtracting 2 BV from that collected to account for the initial hold-up of water in the apparatus (23 mL).



**Figure 4.2**. Comparison of Elution Profiles from Cycles 3 and 4 (SL644 resin batch SMC-010319SMC-IV-73, nominal 1 BV/h, ambient conditions)

For instantaneous elution (no mass transfer resistance or chemical equilibrium constraints), plug-flow through the column and apparatus and an even distribution of cesium through the bed, the cumulative fraction of eluted Cs profile would be a step from 0 to 1 at 0 normalized bed volumes. This profile is approached for down-flow elution although the step is centered at approximately 1.5 BV, as shown in Figure 4.3. However, the head of fluid above the bed would act as a reservoir in which the incoming eluant would be mixed and diluted before passing through the bed. Elution only appears to become significant when the acid concentration in the fluid head has presumably reached a critical value since there is a rapid increase in the cumulative fraction of eluted Cs starting at 1 BV. As shown in Figure 4.3, all but ~4% of the total Cs eluted from the resin is eluted after passage of ~1 BV of eluant through the bed when the long tail of the elution profile begins. The non-step wise nature of the profile could be due to improving elution efficiency as the acid concentration increases, mass transfer effects or chemical equilibrium constraints.

In contrast to the down-flow elution profile, the cumulative fraction of eluted Cs resulting from up-flow elution exponentially increases from 0 BV to ~0.98 after ~5.5 BV. For up-flow elution, the acid concentration of the eluant entering the base of the bed would be expected to be nearly at full strength (0.5 M) since there is little opportunity for mixing in the feed lines. However, the eluted Cs would be mixed and diluted in the fluid head above the bed. As first approximations in the analysis, the fluid head above the bed was described as a perfectly mixed vessel being fed and discharged at a constant rate, v, and the Cs was considered to have instantaneously eluted. The cumulative fraction in the effluent, y, of a tracer instantaneously added (as a pulse input) to a vessel of volume V as a function of time, t, is described by the continuously stirred tank mixing model,

$$y = 1 - \exp\left(\frac{-vt}{V}\right) = 1 - \exp\left(\frac{-x}{V}\right)$$
(6)

where v is the volumetric flow rate. An equation of this type was fitted by least squares to the up-flow elution data and is shown in Figure 4.3.



**Figure 4.3**. Comparison of Up- and Down-flow Elution Profiles (SL644 resin batch SMC-010319SMC-IV-73, nominal 1 BV/h, ambient conditions)

In this case, the variable x is the product of v and t and it and V are in terms of bed volumes. The value of V is then the reciprocal of 0.8679, or 1.15 BV, or approximately 12 mL. The volume of the headspace was estimated to be no more than 2 mL and so the model described above appears imperfect. However, imperfect mixing in the headspace and gradual elution of Cs due to mass transfer resistances and chemical equilibrium constraints would artificially enlarge the mixing volume. Note that this analysis is only applicable to the peak region of the elution profile.

When eluting in the up-flow direction there appeared to be some improvement in the volume of eluant required to achieve an eluant Cs concentration 1% of that in the simulated LAW feedup-flow. The Cs concentration in the eluant was 1.2% of that in the simulated LAW feed after  $\sim$ 38 BVs of eluant had been processed in the up-flow direction whereas it was  $\sim$ 2.4% at the same point for down-flow elution. However, this was not considered sufficiently significant to warrant further analysis given the long elution tail and the possibility of resin clumps shielding ion exchange sites holding Cs.

## 5.0 Conclusions

Elution of Cs from the ion exchange resin SL-644 when eluant is pumped in the down-flow direction is characterized by almost complete removal (~95%) of the Cs from the resin in approximately 1 BV of eluant. Mixing of the eluant with residual DI water in the column and a consequent initial reduction in the eluant acid concentration is hypothesized to have delayed elution.

Nearly complete elution (~95%) was achieved in 5 BVs of eluant when it was pumped up-flow through the column. The difference in behavior between up-flow and down-flow elution is considered associated with mixing of the eluate in the head of liquid above the bed and consequent dilution of the eluted Cs. This hypothesis is supported by the good fit obtained by applying the continuously stirred tank mixing model to the data, albeit with qualifications associated with imperfect mixing and mass transfer and chemical equilibrium effects. There is no project formalized concept of the internal design of the WTP ion exchange columns but a head space above the resin bed is normal in such equipment. On this basis, the up and down flow elution characteristics observed in this test would be expected to manifest themselves in the WTP columns.

Eluant flow direction appeared not to have a significant impact on the tail of the elution profile and the total volume to achieve an eluant Cs concentration 1% of that in the simulated LAW feed. However, greater volumes of eluant were required to achieve eluant Cs concentrations 1% of those in the simulated LAW feeds in both cycles than previously required for this resin. Approximately 45 BVs were required for the down-flow elution cycle compared to less than 15 BVs required in previous tests. The poorer performance is considered due to degradation of the resin during storage. The breakthrough performance of the resin with the first batch of simulated LAW was very poor with a breakthrough of 50% achieved after only 8 BVs of material had been processed. However, breakthrough performance improved significantly in subsequent waste processing operations after the resin had been eluted and the bed fluidized, indicating that extended acid conditioning may minimize the impacts of potassium salts leftover from manufacture and degradation. Some characteristic of the bed, though, induced by the degradation that is manifested in the long elution tail is hypothesized to have persisted through subsequent cycles. The storage conditions and nitric acid pre-conditioning of the resin appear, therefore, to have a significant impact on the resin performance throughout its operational life.

## 6.0 References

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Appendix A: Feed Analytical Reports

### Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report PO Box 999, Richland, WA 99352

Client	S. Arm	Charge Code/Project:	W58970 / 42365
			and W58612/ 42365
<b>RPL Numbers:</b>	01-01849 and 01-02132	ASR Number:	6195 and 6215
Analyst:	MJ Steele	Analysis Date:	March 14, 2002

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)

		TIC(1)	ter et de	TOC			TC	
			MDL	Result	RPD&RSD	MDL	Result	RPD&RSD
RPL Number	Sample ID	(ugC/mL)	(ugC/mL)	(ugC/mL)	(%)	(ugC/mL)	(ugC/mL)	(%)
ASR 6215				·			<u> </u>	
01-02132	AN105 C1#1	2,500	500	< 500	<u> </u>	35	2,500	
01-02132 Dup	AN105 C1#1 Dup	2.600				35	2,600	5 & 3
ASR 6195								,
01-01849	AW101 FEED TI70r1 DF#2	2,700	200	< 200		35 .	2,700	
01-01849 Dup	AW101 FEED TI70r1 DF#2 Dup	2,900				35	2,900	8 & 5
Batch QC Sam	ples					<u> </u>		
02-00778 MS (2	) Tc-1X-Effluent MS	:		75%		[		
01-02132 MS	Batch Matrix Spike (TC)					<u> </u>	89%	
BSICS	Blank Spike LCS			103%			96%	

#### TOC/TIC/TC Results

MDL = method detection limit; TIC = total inorganic carbon; TOC = total organic carbon; TC = total carbon

RPD = relative percent difference

(1) TIC is by difference: TC - TOC.

(2) Analysis Batch Total Organic Carbon (TOC) MS performed on Sample 02-00778 (ASR 6281)

The TOC/TIC analyses of the samples submitted under ASRs 6195 and 6215 were to be performed by both the hot persulfate and furnace methods. This report presents the results from the furnace oxidation method and compares the results to those obtained from the hot persulfate method. Determination of total organic carbon (TOC) is performed by combusting an aliquot of the sample in oxygen at 700 °C for 10 minutes. The total carbon is determined on another aliquot of the sample by combusting at 1000 °C for 10 minutes. The total inorganic carbon is obtained by difference.

The table above shows the results, rounded to two or 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

### Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report PO Box 999, Richland, WA 99352

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 TOC was 97% and for TC was 98%.

System blanks were analyzed similarly to the calibration check, averaged, and subtracted from the sample 'raw data' results prior to calculating the final reported result. The TOC determination produced an average blank of 35  $\mu$ gC and the TC determination produced an average blank of 12  $\mu$ gC, which was subtracted from the sample measurements.

The QC for the analyses include sample duplicates, blank spikes (as a laboratory control sample), and matrix spikes. 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 QC Control Parameter Table 12.2 appended to the ASR.

Blank Spike/Laboratory Control Sample: The BS/LCS for both the TOC and TC measurements is well within acceptance criteria of 80% to 120%.

<u>Duplicates</u>: The precision between the duplicates (replicates), as demonstrated by the Relative Percent Difference (RPD), is acceptable for the samples. For those measurements above the MDL, the RPD is well within the QA Plan's acceptance criteria of <20%. The RSD calculated from only two results produced an RSD within the acceptance criteria of Table 12.2 (i.e., <15%)

<u>Matrix Spike</u>: The accuracy of the carbon measurements can be estimated by the recovery results from the matrix spike. The batch MS for TC was prepared from a liquid sample from a different ASR (ASR 6281; Sample 02-00778.) The batch MS for TOC was prepared from sample 02-02132 (AN-105C1 $\pm$ 1). Although somewhat low, both the TC and TOC MS recovery meet the acceptance criteria of 75% to 125%.

·		TIC	· · · · · · · · ·	TOC		TC	
ASR Number	r sample ID	Furn. (1) (ugC/mL)	HP (ugC/nL)	Furn.	HP (ugC/mL)	Furn. (ugC/mL)	HP(2) (ugC/n1L)
ASR 6215			<u>l` 3.                                    </u>	· · · ·			
01-02132	AN105 C1#1	2.500	1,400	< 500	1,100	2,500	2,500
01-02132 Du	p  AN105 C1#1 Dup	2,600	1,400	nm	1,100	2,600	2,500
ASR 6195							
01-018-9	AW101 FEED TI70r1 DF=2	2,700	1,300	< 200	1,600	2,700	2,900
01-01849 Du	p AW101 FEED TI70r1 DF#2 Dup	2.900	1,400	г. <b>п</b> :	1.400	2,900	2,800

Furnace Results Compared to Hot Persulfate Results

HP = hot persulfate method; Furn = Furnace Method

nm = not measured

(1) TIC is by difference: Furnace TC - Furnace TOC

(2) TC is by summation: HP TIC + HP TOC

## Battelle PNNL/RPG/Inorganic Analysis --- TOC/TIC Report PO Box 999, Richland, WA 99352

The two method appear to produce nearly identical results for TC. However, there are significant differences between the TIC and TOC results for some samples. The reason for the discrepancy between the hot persulfate method and furnace method is unknown. It appears that the organic carbon compounds measured by the HP method are not combusted at the 700 °C (as TOC) used for the furnace method. Typically, the furnace method produces the best TC results and the hot persulfate the best TIC results; thus, a better TOC result may be the difference between these measurements (i.e., Furnace TC – HP TIC).

#### 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.

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- 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 r values for n-1 degrees of freedom to establish the daily MDL. The sample MDL (in ug C/m) 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:

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Review/Approval by:

Date <u>3-20-02</u> Date <u>3-20-02</u>

Raw Data Calculation/Archive Information: ASR 6195, 6215, 6281.xls

Client:	Arm/Burgeson	femp.	ĺ	Ar I	atyzer M& H	F_W013071 701 Balance MRTE: 360-06 01-023
Project :	42365	1000 [	Degree C			
Work Pkg:	CMC / Lut Of	Run time				BS & MS SPIKE: JT BAKER LOTOMS #161359 11 99% Carbon <<[6]
Analyzed:	March 14, 2002 Web 2/15/02	7 5	Minutes	<u> </u>		CAL STD: MALLINCKRODT CMS#161732 11 99% Carbon <<
ASR:	6195, 6215, 6281			-		
		0.1 m-0				12.3 <ss (ug="" average="" blank="" c)<="" td=""></ss>
		(10 C)				0.8 <<< Blank Std Dev (ug C)
Blanks:	Catibration blank (start of batch)	12				3 <<< POOLERISTRIDEV [19] U)
	Calibration blank (start of batch)	12				
<u> </u>	Calibration blank (end of batch)	13				
						t van vvvv [1] Avarana [7 %, Rac
		-	Total Car	1 () ()	1	
		[A] Raw	[8]	[D] Std		
		TC (ug)	Blk (ug)	wt (0)	% Rec	
Standards:	Calibration Slandard (start of batch)	3801	12	0.0321	98.4	
	Calibration Standard (start of batch)	7160	12	0.0603	98.9	
	Calibration Standard (end of batch)	1200	12	0.0103	96.7	
				1 0.010	(15.15	-1-
00	Blank Spike/LCS	1830	15	0.0102	0 00	
	Blank Spike/LCS					
Formulas:	Standard TC % Recovery = ((A-B)/((C/100)*D))	E <sup>-6</sup> *100				
	0C % Recovery = (((A-B)/((C/100)^D))^E * 100)/	(1100)				
_	Sample TC (ug C/ml or ug C/g) = $(1-J)/(K^{+}L/100)$	(				
	MS TC % Recovery = ((((O-R)/(1/100))-S-T)-100					
				a ha chabha a	of due to rol	
Comments:	Dae to the precision carried in the spreadsheet.	some results n the Std Deviati	nay appear t on from the i	o un bier (n) of	blanks time	es the Student's Lyake for the number of degrees of freedom (n-1).
	The weited Detected Limit to the variation is from the first structure of the first structure of the first	al reported "les	ss than" con	centration is c	alculated by	by dividing the Method Detection Limit [M] by [K].
	IF OF ON THE RESULT HIS PRAYED AS A MANAGEMENT AND THE PRAYED AND A MANAGEMENT AND	in the RPD is	not ratculat	ed and displa	ved as "n/a	
	If either the Sample or Duplicate are < px mu, u	er er ivr ann uati				

nical Processing Group: TC Calculations \*\*Review\*\* Report - Furnace Method PNL-ALO-380 4 ÷ ò

PNNL Radiochemical Processing Group: TC Calculations \*\*Review\*\* Report - Furnace Method PNL-ALO-380

					01 000
Client:	Arm/Burgeson	Temp.	Malyzer M&LF - WD13071 701		C20-10-0
		1000 Degree C			
Project :				11 00% Cadwo	22101
Mark Dia.		Run time	THE A MIS SHERE JE INVALIATED AND A DATA		[m]
IVUTA FAG.			CERTIFICATION TO A CAR AND	11 00%, Cadron	ددرن
Analyzad'	March 14 2002	10 Minutes			
Analysee.					
ASR:	6195, 6215, 6281				

		Note: Sample	weights are on	as received.	f" basis; i.e., we	et weight
I Sola olume	s	[I] Raw	[1]	[K] Sam	TC	TC
DI Mumber	Samula (D	TC (ug C)	BIK (ug C)	Vol. (ml)	(m) C/m])	RPD (%)
	ANTOC D #1	259	12	0.1000	2,514	
1 03 43 101 10	AN105 C1#1	270	12	0.1000	2,634	G
1 DJ 132 MIS	AN105 C1#1	606	12	0.1000	see below	
1 01840	AW10111510 11/001 DF#2	2/18	1	0.1000	2,706	
1-01940 Dillo	AW101 FEED TI70r1 DF#2	301	12	0.1000	2,944	8
0.0776	T_c_1X.fluate	82	12	0.5000	141	
01100-2	AN2-Tc.0-C	2074	12	0,100	21,041	
12-00171	Te.tx.Fffhnnt	1955	12	0.100	19.826	
0 00778 DUD	l'fe_1X_l-fllnert	1972	12	0.1000	20,000	-
	AN 102 D104-Cef-Comp1	80	21   21	0.500	139	

ŧ

[O] Raw MS         [R] MS BIK         [S] Sam         [1] MS Sam         [U] Sample         Spike         [U] Spike							· · · · ·	Cuit Caston	N.V.
(iiig C)         (iiig C)         (iiig C)         (iiig C)         (iiig C)         wt (j)         (iiig C)         % Recovery           covery (TC)         606         12         2634         0.10         263         0.0032         384         89.3		FOT RAW MS [FR]	I MS BIK	S] Sam	IJ MS Sam	[V] Sample	spike	avide (n)	
covery (TC)         V = 12         2634         0.10         263         0.0032         384         89.3				ci C/ml)	Vol. (ml)	(ng C)	wt (g)	(rig C)	% Recovery
				2634	0,10	263	0 0032	384	89.3
	covery (1U)	-	   !						

Reviewertdate: MULD Than 3-19-02

Dipol # 11060 0,25ml 19=1,000,1 3/14/02 Balance M&TE: 260-06-01-02-3 m/m Comments J3°C Ene particulate )~ovu – Ch8/11 MY Stleele fine Enno= Pr. 0996 <u>مح ماخدار</u> له 0.0796 0.0995 0,0999 late of 6661 802 19,72ev 13.2 1830 Carbon (ug Zirre 7-160 258.7 30018 1206 380 81.6 1955 Furnace Benchsheet 626 23 270.4 31 Client Norm/ Hugan ASR 6(95, 62/5, 628/ Analyst/Date Matrix Spike - Mallincheelf 16/732 Standards: Calcium Carlonate Cal Verification \_ 577 Bubar # 161359 DISMY Sample Delm J.M. 012 0 hSm Run Time/Tomp: //min//WO C Analyzer M&TE: Σ Þ 01629 ľ. 01035 -0603<u>9</u>-03219 .00325 Standard AWDIFEED TT 70C1 DF4 2 AN-102 Kroy - Cole-amp TC-IX-EFFLent 5-1X-2/1/1402 Te- /X-Eluit AN2-TC-0-1 ANIOS CI #1 Client ID Procedure: PNL-ALO-380 , Z Dlank 96 Hank 0.1-1849 01-2132 Narl Lab ID 0 T C C 540 02-10 021 C C C C R

Carbon Bench sheet.xls

PNNL Radiochemical Processing Group: TOC Calculations \*\*Review\*\* Report - Furnace Method PNL-ALO-380

Clinut	Arnifturgeon	Temp.			Analyzer M&1	E: WD13071 701 Balance M&H	360-06-01-023
Project :	42365	100	Degree C	<u>.</u>			
Work Pkg	C.N.C.	Run Jime		CAL	STD - alpha-1	0.gtucose: Aldrich, lot HY 12603EY, CMS# 53219	40.00% Carbon <<[G]
Analyzed:	March 14, 2002 11, 102	10	Minutes	' <u></u>	BS/MS -alpha	-D-glucose: Sigma Lot 581 (01281, CMS# 161713	40.00% Carbon <<[C]
ASR:	6195, 6215, 6281 V 711						
		TOC				35.3	<<< Blank Average (ug G)
		(nit C)				10,8	<<< Blank Std Dev (ug C)
Blanks:	Calibration blank (start of batch)	40.0					<<< # of Blanks analyzed
	Calibration blank (start of batch)	43.0				46	<<< Method Det. Limit (ug C) [M]
_	Calibration blank (end of batch)	23.0					
			21	20		97.0 <<<< [L] Average 10C % Rec	
		(A) Raw	[8]	[D] Std	TOC		
		TOC (ug)	Bik (ug)	Wt (g)	% Rec		
Standards:	Calibration Standard (start of batch)	000	35	0.0024	100.4		
	Calibration Standard (start of batch)	1478	35	0.0037	97.5		
	Catheorem Standard Cond of batchy	2160	35	0.0057	93.2		
00	fittink Suke/LCS	139.0	35	0.0034	102.7		
		-		ļ			
Formulas:	Slandard TOC % Recovery = ((A-B)/((C/100	0013.((cl.(c					
	OC % Recovery = (((A-B)/((C/100)'D))'E <sup>-6</sup> -1	(00)/(F/100)					
	Sample TOC (ug C/ml or ug C/g) = _(I-1)/(K' MS TOC % Recovery = (((Q-R)/(L100))-S*	T/1001-11					
Comments:	Due to the precision carried in the spreadsh	ieel, some rest a is the Std De	da may app	ear to be slig the number	jhtly off due to (n) of blanks	s rounding. imes the Student's t-value for the number of deg	rees of freedom (n-1).
	Fire nacinal trendent transformer and the transformer of the fired of the transformer of	the final report	iedl seaf ba	n" concentra	tion is calcula	led by dividing the Methed Detection Limit [M] by	įkį.
	If either the Sample or Dupticate are < 5x m	idi, then the 10	IN IS NOT CH	culated and	se oblighted	1. T.	

Client	Arm/Burgeson	femp.			Analyzer M&T	1:: WD13071	701	Balance M& HE:	360-06-01-023	
Droiort	42366	700 E	Degree C							
		Run time		CM	STD - alpha-l	D-glucose: A	drich, tot HY 126036	CMS# 53219	40.00% Carbon	<br !
WOLK 1'KG:				-	         				AD ADV/ Cachen	
- Analyzed:	March 14, 2002	10	Minutes		RS/MS - alpha	n-D-glucoser	Sigma Lot 58H01281	1. CM5# 101/13	40.00% Carpon	
ASR:	6195, 6215, 6281									
		Note: Sample	weights are	on "as reco	cived" basis; i	.e., wet weigh	-			
Carnels Door		til Raw	171	[K] Sam	100					
SAVI SIGNIPS										
RPL Number	Sample 1D	TOC (ug C)	Bik (ug C)	Vol. (ml)	(mic) (mic)	1817 (%)				
02-00776	Tc-1X-Elunte	57	35	0.500	45 ( <mdl)< td=""><td></td><td></td><td></td><td></td><td></td></mdl)<>					
02-00777	AN2-Tc-0-C	2095	36	0.100	21,230					
02-00778	Te-1X-Lifthent	1815	35	0.100	18,344					
02-00778 DUP	[c-1X-Effluent	1904	35	0.100	19,262	ۍ ا				
02-00778 MS	Tc-1X-Effluent	2840	35	0.10	see below					
02-00779	AN-102 C104-CstComp1	- 15	35	0.50	82 ( <mdl)< td=""><td></td><td></td><td></td><td></td><td></td></mdl)<>					
01.09140	AN105 C1#1	97. 197	35	0 10	-72 ( <mdl)< td=""><td></td><td></td><td></td><td></td><td></td></mdl)<>					
01-013441		69	1	67.0	(ipui>) 861			١		

Furnace Method PNL-ALO-380
TOC Calculations **Review** Report -
PNNL Radiochemical Processing Group:

Matrix Sniko Rosults	[U] Raw MS	III WS BIK	[S] Sam	[T] MS Sam	[V] Sample	Spike	[U] Spike	MS
	- () un ()		(un C/ml)	Vol. (ml)	(Ing C)	w( (d)	(0 Gin)	% Recovery
KPL Number Julen Sample in							[ 1 	
				- m -				
no antital Land Chasar Calmin Recovery	2840	35	18344	010	1834	0.0035	1400	75.5
	Ţ							

1/hu 3-19.00-Reviewer/date:\_\_\_\_\_\_\_\_\_

.

	Arm/Burgeson	Temp		<u>~ !</u>	Vnotyzer M& 1	E: WD1307	1 701	£	atance M&TL:	360-06-01-023		Ī
Project :	42365	200	Degree C									
Work Pkg:		Run time		CVL	STD - alpha-f	-glucose: A	Ndrich, Jol 1D	r12603EY,	CMS# 53219	40.00% Carbon	<[0]	
Analyzed:	March 14, 2002	10	Minutes		BS/MS -alpha	-D-glucose:	Sigma Lot 5i	8H01281, C	MS# 161713	40.00% Carbon	c]</td <td></td>	
ASR:	6195, 6215, 6281											
		Note: Sample	: weights arc	aon "as rece	ived" hasis; io	e., wet weigt	ħ					
Sample Res	sults	wey [1]	[7]	[K] Sam	TOC				:		• •	
RPL Number	Sample ID	TOC [ug C]	Bik (ug C)	Vol. (ml)	(III) (III)	RPD (%)	ן כ	تر ن	19 19	コミント	ا۔ ا	
02-00776	Tc-1X-Isluate	57	35	0.100	225 ( <mdt)< td=""><td>ł</td><td>5</td><td></td><td></td><td></td><td></td><td></td></mdt)<>	ł	5					
02-00777	AN2-16-0-C	2005	35	0.100	21,230							
02-00778	Tc-1X-E.Itluent	1815	35	0,100	18,344							
02-00778 DUF	7 1 c-1X-1-Muent	1904	35	0.100	19.262	S.						
02-00778 MS	1.c-1X-Efftuent	2840	35	0.10	see below							
02-00779	AN-102.0104-0515-Comp1	75	35	0.50	82 ( <mdl)< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></mdl)<>							
01-02132	ANHS (21#1	82	35	0.10	-72 ( <mdl)< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></mdl)<>							
01-01849	AVVIO 111.0 11/01 01 #2		36	0.25	138 (smdl)				ŧ			
		COLOSED MC	RPL MS RIK	rst Sam	ITT MS Sam	IVI Sample	Spike	UI Spike	SM			
Matrix Spit	(e kesuits											
RPL Number	Client Sample ID	[ug_C]	() (i) (i) (i) (i) (i) (i) (i) (i) (i) (	(mg_C/ml)	Vol. (ml)		(6)		74 KGGGVGLY			
						10,00	0.000	1 400	75.5			
02-00778 MD	Total Organic Carbon Recovery	2840	8	11.344	0.10	FC01	CC00 0					

Parge 2 of 2

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File: asr6105,6215,6281Fur.zls

	MAXELLE STRICT	ANN-ICHECSE TREESE	Side C	C MS# INDIZ	Comments				milel we for ne ally to breek up therease		11/10/60 CHIER 1/1342	0.25 ml 0.1 ml	0.2471 0.0997	0.2003 0.1003	0,0010 4010			
urnace Benchsheet	Analyst/Date W1	yzer M&TE: (√ ○   ⊃ ()	405/ <u>85</u>		Sample Carbon (ug)	, 0037 1478	10	C.72 (1m)	· 1. 0 - 2.2.2 - 2.0.9.	13 40	1 ml 1250 2840	1.1.1 /1253 2C1 3	-120 0218-15m/2m/2	001/6 1/10-1 mil-	73,7			
	Micsen ASR	Run Time/Temp: $\int \hat{U}_{\rm min} \int \partial \hat{v} \int \hat{V} = \int \hat{V} = \hat{V}$	6 HUCUS C	1. 5.32.19	Vient ID Standard					- CO 34	1, 0035			6900				
	Stient David B	rocedure: PNL-ALO-380	Standards: $\infty - 1$ ).					<u>                                     </u>	$\frac{(2,2-1)}{(2,2-1)}$	<u></u>	$\frac{1}{2} \frac{1}{2} \frac{1}$			02-178 Dup				

Carbon Report sheet xis

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

Project / WP#:	42365 / W58970
ASR#:	6195
Client:	S.T. Arm
Total Samples:	6

RPL #	Client ID
BLK-1850 '	Reagents Only
01-1848	AW101 Feed TI70R1DF#1
01-1850	AW101 Feed TI70R1UF
01-1850-DUP	AW101 Feed TI70R1UF-DUP
MS-1850 (ICP/MS)	AW101 Feed TI70R1UF & ICP/MS Spk.
BS-1850 (ICP/MS)	Reagents & ICP/MS Spk.

**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

- Analysis Date: 13FEB02
- Analysis Files: Experiment 13FEB02 Procedure - 13FEB02 Element Menu - 13FEB02a
- Point of Contact: Orville Thomas Farmer III
- Analyst: James P Bramson, Teresa C Wilson

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

Jult 7 Reviewed By

De 3.15.02 DCu

Concur

Page 1 of 2 3/15/2002 The samples (AW-101 Feed) submitted for analysis were analyzed on a radioactive-materialcontained ICP/MS for the requested analyte(s), Cs and Rb.

### 1. Analysis

See attached ICP/MS data reports for Cs and Rb 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.

#### 2. Quality Control

Duplicate (DUP). Duplicate samples were submitted and analyzed and met the QC success criteria of  $\pm 25\%$ . A Post replicate was analyzed and met the QC success criteria of  $\pm 25\%$ .

<u>Matrix Spike (MS)</u>. A matrix spike was submitted and analyzed and met the QC success criteria of  $\pm 25\%$ . A post spike analysis was performed and met the QC success criteria of  $\pm 25\%$ .

<u>Preparation Blank (PB) and Laboratory Control Standard (LCS BS).</u> A Process Blank was submitted and analyzed and meets the success criteria of being <10 EQL. The LCS/BS met the success criteria of + 25%.

*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.

<u>Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV).</u> The ICV/CCV standards met the QC criteria of  $\pm$  10%.

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

1.ոբ.լո	Sample	Client	ICP/MS	MDL	Cs-133		Rec/RPD MDL	RI;		Rec/RPD
Number	ID	G	e	2/224	∓ ઝ/તંધ	15D	3/ari •/•	на/н +	15D	*
	t-'I¢I		SOL-2	V	2.28E-06			< 2.35E-06		
	5~10H		SOL-3	V	2.27E-06			< 2.34E-06		
	8-101 		SOL-4	V	2.27E-06			< 2.33E-06		
	IDL-4		SOL-5	V	2.33E-06		·	< 2.40E-06		
	1D1-5		SOL-6	V	2.31E-06			< 2.38E-06		
	1016		SOL-7	V	2.27E-06			< 2.34E-06		
	2~10H		SOL-8	<b>V</b>	2.31E-06			< 2.38E-06		
	STD BLK		801-9	V	2.34E-06			< 2.41E-06		
	ICB		SOL-18	V	2.51E-06			< 2.58E-06		
	CCB1		SOL-39	V	2.73E-06		-	< 2.81E-06		
	CCIE		SOL-59	~	2.75E-06			< 2.83E-06		
True Value			1		2.50E-03		-	2.50E-03		
	ICV		91-10S		2.47E-03 ±	1.98E-05	99 <u>%</u>	2.32E-03 ± 2	2.41E-06	93%
	CCVI		201-37		2.48E-03 ±	7.19E-06	%66	2.54E-03 ± 3	.87E-05	%101
	CCV2		SOL-57		2.555-03 ±	3.48E-05	102%	2.73E-03 ± 5	.77E-05	%601
BLK-1850	Reagents Only	Reagents Only	SOL-19	5,33E-04 <	5,33E-04		5.4XE-04	< 5.48E-04		
BLK-1850	Reagents Only post spike	Reagents Only	SOL-45	5.24E-04	4.94E-01 ±	4.04E-03	99% 5.39E-04	5.09E-01 ± 1	.27E-02	102%
Post Spike C	oncentration Expected				2,50E-03			2.50E-03		
I3S-1850	Reagents + spike	Reagents + spike	SOL-49	5.21E-04	[.11E+00 ±	2.55E-03	92% 5.36E-04	1.92E-01 ± 9	,88E-04	96%
Spike Conce	ntration Expected				6.00E-03		.e.;	1.00E-03		
01-1850	AW 101 Feed T170K1DF	AW 101 Feed T170R1UF	SOL-23	1.92E-02	2.41E+01 ±	1.20E-01	1.97E-02	1.82E+00 ± 3	.10E-02	
01-1850	AW 101 Feed T170R1UF replicate	AW 101 Feed T170R1UF	SOL-25	1.94E-02	2.41E+01 ±	2.42E-01	0.01% 1.99E-02	1.79E+00 ± 3	.86E-02	1.57%
01-1850-DU	II AW 101 Feed T170R1UF dup	AW 101 Feed T170R1UF dup	SOL-27	2.00E-02	2,43E+01 ±	1.12E-01	0.79% 2.06E-02	$1.82E+00 \pm 3$	.57E-02	0.18%
01-1850 Post Snike C	T1708.005 post spike oreentration Eveneted	T170R1UF post spike	SOL-35	2.03E-02	4.38E+01 ±	1.92E-01	98% 2.09E-02	2.13E+01 ± 2 2.50E_03		97%
0]-1848	AW 101 Feed T170R1DF#1	AW 101 Feed T170R11)F#1	SOL-29	2.01E-02	2.32E+01 ±	1.64E-01	2.06E-02	1.82E+00 ± 3	.08E-02	
							÷			
MS-1850 Spike Concer	AW 101 Food T170R10F+spike ntration Expected	AW 101 Feed T170R1UF+spike	SOL-53	2.29E-02	4.56E+01 ± 3.00E-03	1.34E-01	89% 2.35E-02	5.38E+00 ± 2 5.00E-04	.29E-01	89%

•

		15D	1.84E-07 0.02E-07	4.10E-06	-6.18E-07	-6.15E-07	-2.32E-07	2.77E-07	2.29E-06	5.85E-05	9.99E-05	7,44E-05	4.79E-05	-4.56E-07	1.00E-04	-1.82E-04	3.10E-02	7.59E-05	3.86E-02 3.72E-05	3.57E-02	2.33E-05 3.08E-02	7.73E-05	9.20E-04	1.44E-03	1.21E-04	7.83E-05	3.8/E-UD
	i	Rb ± 0/01	2.35E-06 ±	2.33E-06 ±	2.40E-06 ±	2.34E-06 ±	2.38E-06 ±	2.41E-U5 ± 3.81E-06 ±	3.84E-05 ±	4.9/E-04 ± 2.55E-03 ±	4.98E-03 ±	2.95E-04 ±	2.32E-03 ±	2.58E-06 ±	3.62E-04 ±	5,14E-04 ±	0.03E+04 I 1.82E+00 ±	3.72E-04 ±	1./9E+00 ± 4.28E-04 ±	1.82E+00 ±	2.84E-04 ±	4.35E-04 ±	4,60E-02 ±	3.86E-02 ±	4.68E-04 ±	9.36E-04 ±	2.54E-U3 ±
	1 34E-05	в <sub>/бл</sub>	2.35E-06 <	2.33E-06 <	2.40E-06 <	2.34E-06 <	2.38E-06 <	2.41E-06 < 2.39E-06	2.40E-06	2.41E-05 2.41E-06	2.46E-06	2.95E-04 <	2.55E-U0 3.24E-04 <	2.58E-06 <	5.48E-04 < 3.62E-04 <	5.14E-04 <	3.64E-04 1.97E-02	3.72E-04 <	1.99E-02 3.84E-04	2.06E-02	2.84E-04 < 2.06E-02	4.35E-04 <	1.05E-02	4.09E-04 1.06E-02	4.88E-04 <	2.096-02 3.346-04	2.58E-Ub
B822.6497 39.3342 39.3342 39.3342 0 173 73 73 73 73 73 73 73 73 73 73 73 838	1.34E-06	Conc. µg/g	-4.46E-06	-4.33E-06	-5,22E-06	-5.08E-06	-5.43E-06	-5.32E-06 3.81E-06	3.84E-05	4.9/E-04 2.556-03	4.98E-03	2.54E-04	2.32E-03 2.50E-04	-5.61E-06	-1.10E-03 2.33E-04	-1.13E-03	6.03E-04 1.82E+00	2.77E-04	1.79E+00 4.28E-04	1.82E+00	2.24E-04	2.10E-04	4.60E-02	3.86E-02	4.02E-04	2.128E+01 9.36E-04	2.54E-03
lope Nercept Std 0.000 0.000 0.000 0.000 0.000 2.500 5.000	(IDL)	Std Dev	010	2	ы с	N	~- i	C1 V0	23	131	882	573	332	2	2 580	4	542 35	507	43	40	165 26	336	300	5 202	886	694 694	342
07 <u>–</u>	Ϋ́	Avg acps bkg-sub	01	<b>∩</b> ≁-	<u>~</u> ∙	ትሳ	ဗို	8- 73	378	4425 22533	43938	2277	20479 2245	-10	-9 2095	-	5358 2045	2487	2013 3814	2053	2013	1891	144	121c	3583	23548 8294	22427
		ICP/MS DIL			<b></b> ,	<b>**</b> ***	<b></b> -	• <b>•-</b>	-					-	200	200	400	-	400 400	400		+00 -	200	200		400	-
		Client Dit.			·		-	• •-	-	·- ·		-		-	 		1 1 7 19 97	-	7 19.97	3 19.92	1	cu.uz 1	19.42	1 1 1 2 1 2 1 2 1 2 1 2 1 2 1 2 1 2 1 2	-	7 19.97 1 1	-
	3.000	Totat Díl.			• ·		-					•		•	500	20(	79.8		798	796	0	00	388	382		798	
	200	ICPANS	sol-2	sol-3 sol-4	sol-5	sol-6 sol-7	SOL-8	SOL-9	SOL-11	SOL-12	SOL-14	SOL-15	SOL-16 SOL-17	SOL-18	SOL-19	SOL-21	SOL-22	SOL-24	SOL-25	SOL-27	SOL-28	SOL-20	SOL-31	SOL-32	SOL-34	SOL-35 SOL-36	SOL-37
al sample.	2.000 2	Client													Reagents Only	Process Blank	111 101 E 1 11 700 11 16		AW 101 Feed T170R1UF	AW 101 Feed T170R1UF dup		AW 101 Feed T170R1DF#1	2133			T170R1UF post spike	
cd in Lig analytic/g of origin	000 1.500	sample ID													-	_			70R1UF replicate	70R1UF dup	•	70R1DF#1				spike	
led, the results are roport	0.500 1.		101-1	1DL-2 1DL-3	IDL-4	5-10 1-5-	IDI-7		0.00005 ppm	0.0005 ppm	0.0025 ppm 0.0050 nom	wash	ICV	iCB	Reagents Only	wasn Process Blank	wash	wash wash	AW 101 Feed T1	wash AW 101 Feed T1	hash	AW 101 Feed T1 wash	2133	wash	dup cc 12 wash	T170R1UF post wash	CCV1
Rubidium Unless otherwise specif 25000 15000 15000 5000 5000	0.000	Log-In Number													BLK-1850	01-2133-PB		0001-10	01-1850	01-1850-DUP		01-1848	01-02133		100 20120-10	01-1850	

	hash		SOL-38	-		-	3951	645	4.43E-04	5.36E-04 <	5.36E-04 ±	8.76E-05
	CCB1		SOL-39	•	۰.	-	-11	4	-5.69E-06	2.81E-06 <	2.81E-06 ±	-9,33E-07
01-02133	DT	2133	SOL-40	23306	19,42	1200	12	2	-7.15E-02	6.58E-02 <	6.58E-02 ±	2.68E-02
01-02133	tcs	2133	SOL-41	3884	19.42	200	125	2	3.79E-02	1.16E-02	3.79E-02 ±	2.00E-03
01-02133	ICSD	2133	SOL-42	3884	19.42	200	134	ო	4.19E-02	1.14E-02	4.19E-02 ±	8.90E-04
01-02133	ICSDS	2133	SOL-43	3884	19.42	200	25615	252	1.13E+01	1.26E-02	1.13E+01 ±	1.11E-01
	ysew		SOL-44	-	-	•	5934	355	6.68E-04	6.53E-04	6.68E-04 ±	4.00E-05
BLK-1050	Reagents Only post spike	Reagents Only	SOL-45	200	-	200	22505	562	5.09E-01	5.39E-04	5.09E-01 ±	1.27E-02
	wash		SOL-46	-	-	***	4460	354	5.01E-04	6.31E-04 <	6.31E-04 ±	5.01E-05
01-2133-PB	Process Blank post spike	Process Blank	SOL-47	200	-	200	22162	704	5.01E-01	5.40E-04	5.01E-01 ±	1.59E-02
	wash		SOL48	•	<b>*-</b>	<b>-</b> -	2311	611	2.57E-04	6.16E-04 <	G.16E-04 ±	1.63E-04
BS-1850	Reagents + spike	Reagents + spike	SOL-49	200	-	200	8525	44	1.92E-01	5.36E-04	1.92E-01 ±	9.88E-04
	wash		SOL-50	-	-	-	5580	434	6.28E-04	6.10E-04	6.28E-04 ±	4.88E-05
01-02133 BS	Blank Spike	Blank Spike	SOL-51	200	-	200	8293	108	1.87E-01	5.41E-04	1.87E-01 ±	2.44E-03
	wash		SOL-52			-	3043	179	3.40E-04	6.37E-04 <	6.37E-04 ±	3.76E-05
MS-1850	AW 101 Feed T170R1UF+spike	AW 101 Feed T170R1UF+spike	50L-53	8022	20.05	400	5952	254	5.38E+00	2.35E-02	5.38E+00 ±	2.29E-01
	wash		SOL-54	•	<b>+</b>	-	4917	686	5.53E-04	5.72E-04 <	5.72E-04 ±	7.99E-05
01-02133 MS	Matrix Spike	Matrix Spike	SOL-55	3819	19.1	200	7885	203	3.40E+00	1.200E-02	3.40E+00 ±	8.72E-02
	wash		SOL-56		•••	-	3663	606	4.11E-04	6.98E-04 <	6.98E-04 ±	1.15E-04
	CCV2		SOL-57	**	*-	-	24154	510	2.73E-03	2.84E-06	2.73E-03 ±	5.77E-05
	Wash		SOL-58	+	•	-	5773	1095	6.50E-04	6.84E-04 <	6.84E-04 ±	1.30E-04
	CCB2		SOL-59	-	-	-	-13	-	-5.93E-06	2.83E-06 <	2.83E-06 ±	-2.01E-07
	wash		SOL-1	-	-	•	1889	289	2.10E-04	1.01E-04	2.10E-04 ±	3.20E-05
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Uniess otherwse spe 	crited, the results are reprinted in µg analyte/g of	r ongenal sample		:								
25000				ŗ			ωE	lope lercept	8924 3721 -11 6151			
20000								2				
15000			4					(1000)	Ċ.			
10000				 				0000	76 402			
2000				· · · · ·				888	4437 22369 4457/			
000.0	0.500 1.500 1.5	00 2.000 2.5	500 3	- 8								
Log in Number	Sample 10	Client	ICP.MS	Total Di	Cilent Dii,	ICPANS DI.	Avg æps btg-sub	Dev Dev	Conc. µg/g	5jāri Tow	Сs-133 µg/g ±	15D
	DL-1		SOL-2			⊷ •	01	20	1.30E-06	2.28E-06 <	2.28E-06 ±	2.83E-07
	04.2 04.5		501-3 201-4			- •	Ņ,	ə -	1.10E-06 0.60E-07	2.2/E.06 <	2.2/E-06 ±	-3.14E-07
			50L-5				ò vò		7.966-07	2.33E-06 <	2,335-06 ±	3.866-07
	101 -5 		501-6 501-7		<b>.</b>		မှဖ		6.68E-07	2 31E-06 <	2 31E-06 ±	-3.75E-07
	2-10		SOL-8		• ••		γ <b>ιγ</b>	- 14	7.66E-07	231E-06 <	2.31E-06 ±	-7.74E-07
	STULK COMPOSITION		SOL-9	·			 75	- c	5,39E-07	2.34E-06 <	2.34E-06 ±	-2.30E-07
	0 00005 ppm		SOL-11	•			2 <del>6</del>	<u>ہ</u>	4.63E-05	2.33E-06	4.63E-05 ±	1.148-06
	mdd 5000 g		SOL-12	-	-		4437	57	4.99E-04	2.34E-06	4.99E-04 ±	2.73E-06
	0.0025 ppm 0.0050 ppm		SOL-13 SOL-14			• •	22369 44577	<b>3</b> 8	2.51E-03 5.00E-03	2.35E-06 2.39E-06	2.51E-03 ± 5.00E-03 ±	9.44E-06 2.57E-06
	uter		SOL-15	-	-	-	1588	120	1.79E-04	2.87E-04 <	2.87E-04 ±	1.30E-04
	ICV week		SOL-16				22027	12	2,47E-03 1 7RE-04	2.486-06 3.156-04 ×	2.47E-03 ±	1.98E-05
	KGB		SOL-18	-	-	• •-	L-	P	5.40E-07	2.51E-06 <	2.51E-06 ±	-1.54E-07
BLK-1850	Reagonts Only	Reagents Only	SOL-19	ន្តី		8.	-6 775	212	1.15E-04 8 826 05	5.33E-04 ×	5.33E-04 ±	-1.98E-04
01-2133-PB	wasn Process Blank	Process Blank	sol-21	8		8	<u>.</u>	2	1.056-04	5.00E-04 A	5.005-04 ±	-9.89E-05
01-1850	wash AW 101 Feed T120R11JF	AW 101 Feed T170R1UF	SOL-23 SOL-23	1 7987	19.97	- 00	2060 26930	붱	2.32E-04	3.54E-04 < 1.92E-02	3.54E-04 ± 2.41E+01 +	5.93E-05
	vash		SOL-24	1005	- 10	÷ξ	1634	318	1.84344E-04	3.62E-04 <	3.62E-04 ±	7.05E-05
0001-10	Wash wash	AW 101 FEED 11/0K10F	SOL-26			- -	5396 2396	318	2.697E-04	3,73E-04 ×	2.41E+01 ± 3.73E-04 ±	4.95E-05
01-1850-DUP	AVV-101 Feed 1170R1UF dup wash	AW 101 Feed T170R1UF dup	501-27 501-28	2068	19 92 1	<u></u>	27209	126 52	2.430E+01 2.600E-04	2.00E-02 2.76E-04 ≤	2.43E+01 ± 2.76E-04 +	1.12E-01 7.24E-06
01-1848	AW 101 Feed 11/0R1DF#1	AW TOT Feed 11 /OR (UF #1	SOL-29	8011	20.03	400	25789	183	2.316E+01	2.01E-02	2.32E+01 ±	1.64E-01
	wash eest		SOL-30	1	- 9	- 5	2176 79675	445 245	2.451E-04	4.23E-04 ×	4.23E-04 ±	8.66E-05
CC1 70-10	2133 Wash	<b>CC</b> 17	SOL-32	+000	1	3-	2562	193 193	2.883E-04	4.76E-04 <	4.76E-04 ±	3.586-05
01-02133 DUP	2133 dup	2133 dup	SOL-33	3823	19.12	8	28207	356	1.209E+01	1.03E-02	1.21E+01 ±	1.535-01
01-1850	wasn T170R1UF post spike	T170H1UF post spike	SOL-35	7967	19.97	- 8	48897	215	4.377E+01	4.74E-04 < 2.03E-02	4.74E-04 ±	1.92E-01
	hash		SOL-36			<b>.</b> . ,	5193 22133	693 6	5.83E-04	3.25E-04	5.83E-04 ±	7.785-05
	U.C.V.1 Wash		sol-38				1319	8 <u>8</u>	2 48E-03	5.22E-04 <	2.485-03 ± 5.225-04 ±	1.446-04
	CCH1		50L-39	-	-	-	01-	-	1.42E-07	2.73E-06 <	2.73E-06 ±	-1.84E-07
01-02133	DT	EE12	SOL-40	23306	19.42 10.47	82 £	4813 2835.8	8 Ê	1.26E+01	6.40E-02	1.26E+01 ±	1.70E-01
01-02133	CSD CSD	5612 5612	SOLA2	3684	19.42	38	28286	151	1.232E+01	1.10E-02	1.23E+01 ±	6.62E-02

,

<b>Sample</b> 10 KCSCS wash	Client ID 2333	ICPIMS SOL-43 SOL-43	7ofsi Dir. 3884	Cllent DI. 19.42	ICPANS DIL 200	Avg acps bkg-sub 3317 3317	sid Dev 556 1213	соне. µg/g 2.397E+01 3.73E-04	MDL 122E-02 6.35E-04 <	са-133 идиа ± 2.40Е+01 ± 6.35Е-04 ±	<b>tsp</b> 2.42E-01 2.32E-04
Reagents Only post spike wash Process Blank post spike wash Reanparts + spike	Reagents Only Process Blank Ruseuents + surke	SOL-45 SOL-45 SOL-45 SOL-45 SOL-45 SOL-45	8-8-8	~~~~ <b>~</b>	8-8-8	22050 3787 22063 1489 49296	180 245 125 153	4.946-01 4.266-04 4.966-01 1.686-04 1.116-00	5.246-04 6.146-04 < 5.256-04 < 5.996-04 < 5.216-04 <	4.94E-01 ± 6.14E-04 ± 4.95E-01 ± 5.99E-04 ± 1.11E+00 ±	4.046.03 3.976.05 2.816.05 6.176.05 6.176.05
Annonius spino Wash Wash Spike Wash AW 101 Feed 1120A1UF+sc Wash	robgens + spake Black Spake AVV 101 Facel 1170R11JF+spake	sol-53 sol-53 sol-52 sol-54 sol-54	8027 - 00 - 20 8053 - 00 - 20		3-0-9-	2575 2575 2675 2675 50728 5815	376 376 1195 149 2230	2:90E-04 1.14E+00 3.01E-04 4.56E+01 6.53E-04	5.266-04 × 5.266-04-04-04-04-04-04-04-04-04-04-04-04-04-	5.93E-04 ± 5.93E-04 ± 6.19E-04 ± 6.19E-04 ± 4.56E+01 ± 6.53E-04 ±	2.505-00 9.47E-06 8.43E-03 2.77E-04 1.34E-01 2.50E-04
MS Natus Soke vash CCV2 vesh vesh CC82 vesh	Matrix Spie	sol-55 sol-55 sol-57 sol-57 sol-56 sol-1	3819 0 E -	<u>.</u>	See	78913 3085 22729 2829 2829 68 1323	1126 439 310 1066 1 61	3.385+01 3.475-04 2.5555-03 3.186-04 4.226-07 1.506-04	1.17E-02 6.79E-04 < 2.76E-06 6.65E-04 < 2.75E-06 < 9.81E-05	3.38E+01 ± 6.79E-04 ± 2.55E-03 ± 6.65E-04 ± 2.75E-06 ± 1.50E-04 ±	4.826-01 9.666-05 3.486-05 2.516-05 2.516-04 5.348-07 6.946-06

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

Project / WP#:	42365/W58970
ASR#:	6195
Client:	S. Arm
Total Samples:	3

	First	Last						
RPL#:	* 01-01848	01-01850						
Client ID:	lient ID: AW101 Feed TI70r1 AW101 Feed TI70r1							
	DF#1	UF						
Sample Prepar	ration: PNL-ALO-106.							
AOI: Al, Ba, Ca	a, Cd, Cr, Fe, K, La, Mg, N	Na, Ni, P, Pb, and U.						

Procedure: <u>PNN</u> Inducti	Procedure: <u>PNNL-ALO-211</u> , "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry" (ICPAES).										
Analyst: <u>D.R. Sande</u>	ers										
Analysis Date (File):	<u>12-07-01</u> (A07	45)									
See Chemical Measurement (Calibra	: Center 98620 file ation and Mainte	e: <u>ICP-325-405-1</u> enance Records)									
M&TE Number:	<u>WB73520</u> <u>360-06-01-029</u>	(ICPAES instrument) (Mettler AT400 Balance)									

MW/m 3-27-02 Reviewed by Rene's Russold 3/28/02

3/28/02

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

Three liquid samples (RPL# 01-01848...01-1850) submitted under Analytical Service Request (ASR) 6195 were prepared by acid digestion procedure PNL-ALO-106. Analytes of interest (AOI) were specified in a table attached to the ASR as Al, Ba, Ca, Cd, Cr, Fe, K, La, Mg, Na, Ni, P, Pb, and U. All other analytes that were not requested are reported, but have not been fully evaluated for QC performance.

A summary of the ICPAES analyses of the samples, including the QC performance, is given in the attached ICPAES Data Report (2 pages). ICPAES measurement results are reported in  $\mu$ g/mL.

The three process blank samples had detectable amounts of AOIs Al, Ca, Fe, and Na present. However, all of their concentrations were below estimated quantitation limits (EQL). These analytes likely originated from the glassware used to digest the samples or (less likely) from the digestion reagent, nitric acid. Boron was also detected in the process blank above the EQL. Copper and zinc were detected in the process blank below the EQL.

Quality control check-standard results met tolerance requirements for the specific AOIs except as noted below. Following is a list of quality control measurement results relative to ICPAES analysis tolerance requirements of the controlling QA plan.

#### Process Blanks:

The concentrations of the AOIs measured in the process blank samples were within the tolerance limit of  $\leq$  EQL.

#### Duplicate RPD (Relative Percent Difference):

For those AOIs measured above the EQL, the RPDs were within the acceptance criteria of <15% (<3.5% for Na).

#### <u>Blank Spike:</u>

The blank spike recoveries for the AOIs were within the acceptance criteria of 80% to 120%. However, B (not an AOI) failed to meet the acceptance criteria in two of the three BS samples analyzed with 39% and 61% recoveries.

#### Matrix Spiked Sample:

The matrix spike recoveries for the AOIs were within the acceptance criteria of 75% to 125% except for Al, K, and Na, which were not recovered. The matrix spike analysis uses a general spiking solution intended to be usable on the majority of samples analyzed by ICPAES. However, for the sample selected for matrix spiking, the spike concentration for Al, K, and Na were less than 20% of the sample concentration and the recovery results are considered meaningless. For Al, K, and Na, the use of serial dilution results is used to evaluate potential matrix interferences. However, Cu and Zn (not AOIs) failed to meet the acceptance criteria with recoveries of 43% and 60%, respectively.

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

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#### Post-Spiked Samples (Group A):

All AOIs were within the acceptance criteria of 75% to 125%. Neither Al nor Na were recovered from this sample as the spike concentrations were less than 20% of the sample concentrations.

#### Post-Spiked Samples (Group B):

All analytes recovered in the Post-Spiked Sample (Group B) were within the tolerance limit of 75% to 125%.

#### Five fold serial dilution:

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

#### Other QC Samples:

The Na was outside the tolerance limit of  $\leq 10\%$  in the mid-range calibration check standard at 13% and 18%. The K was outside the tolerance limit of  $\leq 5\%$  in the high-range calibration check standard at 7.5%.

#### Comments:

- 1) "Final Results" have been corrected for all laboratory dilution performed on the sample during processing and analysis unless specifically noted.
- 2) Detection limits (DL) shown are for acidified water. Detection limits for other matrices may be determined if requested.
- 3) Routine precision and bias is typically  $\pm 15\%$  or better for samples in dilute, acidified water (e.g. 2% v/v HNO<sub>3</sub> 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/RSE/Inorganic Analysis.... ICPAES Report

		r						10/7/0004	4017/0004	49/7/0004
	Run Date=	12/7/2001	12/7/2001	12/7/2001	12/7/2001	12/7/2001	12///2001	12/7/2001	12/7/2001	12///2001
	Multiplier=	25.0	25.0	25.0	25.0	25.0	125.0	25.0	125.0	125.0
	RPL/LAB #=	BLANK 1	BLANK 1	BLANK 2	BLANK 3	01-01848	01-01848 @5	01-01850	01-01850 @5	DUP @5
								414/404	A14/4.04	A14/407
					<b>_</b>	<u>AW101</u>	AWIU1	<u>AW101</u>	AWIUT Food T170r1	AWIUI Food T170r1
		process black	process blank	<u>process</u>	black	DE#1	DF#1	VI	Vf	Vf•Dun
Det. Limit	Client ID=	plank	Didik	<u>prank</u>				<u>vi</u>		(us(mL)
(ug/mL)	(Analyte)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mc)	(ug/mL)	(ug/mc)	(ug/mc)
0.060	IA	[8.5]	[8.3]	[7.6]	[9.8]	9,900	·	13,100		13,600
0.010	Ba		**		••	16.4		16.8		18.1
0.250	Ca	[12]	[12]	[13]	[21]	[37]	ļ	[30]		[36]
0.015	Cd									••
0.020	Cr	••				[0.58]		[0.66]	·	
0.025	Fe	[2.2]	[2.1]	[1.3]	8.23	12.8	ļ	13.4		[4,4]
2.000	<u>к</u>	•-				15,100	l	14,600		15,600
0.050	La			•- <sup>1</sup>	••					
0.100	Mg				••		ļ	[5.4]	L	
0.150	Na	[26]	[27]	61.1	43.1	over range	119,000	over range	117,000	118,000
0.030	Ni	<u> </u>				8.17	ļ	7.65	L	[8.6]
0.100	P				**	65.5		64.4		[65]
0.100	Pb					[9.4]		[12]	· ·-	
2.000	U						<u> </u>	<u> </u>	<u> </u>	<u> </u>
Other Analyt	es		-							
0.025	Ag					· ••		· · ·		•-
0.250	As									••
0.050	В	18.6	18.7	41.1	27.8	591		110		98.2
0.010	Be									
0.100	Bi									
0.200	Ce		••				•			
0.050	Co	•-				••			•	
0.025	Cu	[1.7]	[1.6]	[1.0]	[2.9]			[3.0]		
0.050	Dy							••		
0.100	Eu									
0.030	Li		••			[4.2]	1	[4.3]		[4.3]
0.050	Mn					[6.5]		[3.2]		
0.050	Mo					28.6		27.1		[29]
0,100	Nd									
0.750	Pd									••
0.300	Rh		•-							
1.100	Ru									
0,500	Sb									
0,250	Se	••								
0,500	Si			[13]		264	1	261		[260]
1 500	Sn						<u> </u>			
0.015	Sr					[2,0]	<u> </u>	[1.9]		[2.0]
1 500								··· ··	<u> </u>	
1 000	ть Тh						<u>+</u>		1	
0.025	ті Ті		·			[0.68]	· [			
0.025	TI						1			
0.000										
2 000							<u> </u>			
0.050		+							1	
0.050	70	[5.5]	(5.6)	[6 1]	[4 4]	[7.7]	<u> </u>	[8.9]	+	
0.030	   									·
,				1	1		4			

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 12/07/2001

·····		<b>I </b>	l .		(		[		
Criteria>	<15% <sup>(ə)</sup>	80% - 120%	80% - 120%	80% - 120%	75%-125%	75%-125%	75%-125%	< +/-10%	< +/•10%
QC ID=	01-01850 &	1.00/00	1.00/00	1.00/00	04 04050 0	01-01850 +	01-01850 +	01-01848	01-01848 @5(@25
	01-01850-D (@5)	ECS/85 85#1	BS#2	RS#3	01-01850 &	Post Spike A	Post Spike B (@5)	Serial Dil	@or@25 Secial Dit
Applytos	PPD (%)	%Rec	%Rec	%Rec	%Rec	%Bec		%Diff	%Diff
Analytes	NFD (70)	00	101	100	701100	701XEC	741160	2.0	2.8
	1.6	101	101	102	0.1	07		53	2.0
	1.0	101	103	102	100	101		<u> </u>	
	·	103	103	102	100	100			
		103	104	104	07	100			
		102	107	103	97	100			
Fe		103	107	105		102		6.2	59
N	0.4	104	100	100	1 111	30	100	0.4	0.0
La		107	* 0.0	100	+04	106	100		
Mg		110	109	109	104	100		0.000 100000	7.4
Na	0.9	118	105	107	over range	nr 102		uver range	7.4
		105	107	107	100	103			
P		102	103	103	103	99			
РБ	·	110	113	113	104	108		-	
0		101	103	103			104		
Other Analyt	es	1	1	i					
Ag						98			·
As						103			
8	13.9	96	39	61	99	99		<b>3</b> .1	3.2
Be						98			
Bi						99			
Ce							98		·
Co		104	106	106	103	103			
Cu		97	100	95	43	98			
Dy						ļ	101		
Eu						<u> </u>	100		
Li		101	101	100	87	95		<b>-</b>	······
Mn		104	105	106	nr I	107			
Ma				<u> </u>		100			
Nd						1	99		
Pd							93		
Rh						· · · · -	96	·	
Ru									
Sb				 					
Se						103	L		
Si		<u></u>	<u> </u>	· ·		109			
Sn					·		9-		·····
Sr		102	104	104	98	100			
Te		ļ		ļ			105		
Th					ļ		100	<u> </u>	
Ti		ļ				95	· · · · · · · · · · · · · · · · · · ·	<u> </u>	
TI	l	ļ		<u>.</u>	[	99		<u> </u>	
V			<u> </u>		ŀ	95	<u> </u>		
W	ļ				ļ		ļ		· · ·
Y		<u> </u>				98		<u> </u>	
Zn	ļ	96	99	102	60	103		· · · ·	
Zr					ļ	100	I	<u> </u>	

Shaded results exceed acceptance criteria

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

<sup>(a)</sup>=RPD <3.5% for Na (only)

Appendix B: Test Operation Data and Calculations

### Cycle 1 Breakthrough Performance

Effluent bottle tare	320.8	g
Feed density	1.248	g/mL
Cs concentration	5.54E-04	М
Bed height in 0.25M NaOH	6.9	cm
Bed volume in 0.25M NaOH	12.213	mL
Time between samples	3.17	hours

	Counts	Count time (s)	Sample mass	Sample activity (CPM/mL)
Feed 1	35799	600	6.368	701.588
Feed 2	13533	300	6.368	530.439
Feed 3	158392	3000	6.386	619.083
			Average	617.037

Effluent	Mass of feed	Volume flow	Volume of	Bed volumes	Counts	Count time	Sample	Sample volume	Sample activity	C/C0 (%)
bottle mass	processed (g)	rate (mL/hr)	feed	of feed		(s)	mass (g)	(mL)	(CPM/mL)	
(g)			processed	processed						
			(mL)							
442.7	121.9	30.8	102.7	8.4	15769	600	6.298	5.046	312.476	50.641
565.0	244.2	30.9	205.7	16.8	21369	600	6.275	5.028	424.996	68.877
687.1	366.3	30.9	308.5	25.3	23379	600	6.190	4.960	471.357	76.390
809.6	488.8	31.0	411.8	33.7	24745	600	6.418	5.143	481.174	77.981
931.5	610.7	30.8	514.6	42.1	25610	600	6.396	5.125	499.707	80.985
1053.1	732.3	30.8	617.2	50.5	26073	600	6.366	5.101	511.139	82.838

#### Cycle 1 Elution Performance (down-flow)

Feed density	1.248 g/mL
0.5M HNO3 density	1.015 g/mL
Bed height in 0.25M NaOH	6.900 cm
Bed volume in 0.25M NaOH	12.213 mL

Eluant mass processed	Cumulative eluant mass	Cumulative eluant volume	Volume flow rate (mL/h)	Bed volumes of eluant	Counts	Count time (s)	Sample mass	Sample volume	Sample activity (CPM/mL)	C/C0	Total CPM in collected fraction	Cs concentration in
	processed	processed		generated							indetion	fraction (mg/L)
4.335	4.335	4.270	8.540	0.350	737	3000	4.335	4.270	3.452	0.006	14.740	4.12E-01
4.372	8.707	8.576	8.612	0.702	818	3000	4.372	4.307	3.799	0.006	16.358	4.54E-01
4.461	13.167	12.970	8.787	1.062	884	3000	4.461	4.394	4.023	0.007	17.678	4.80E-01
3.229	16.396	16.150	6.361	1.322	928	3000	3.229	3.181	5.835	0.009	18.558	6.97E-01
4.440	20.836	20.524	8.747	1.680	4348	3000	4.440	4.374	19.883	0.032	86.960	2.37E+00
4.412	25.247	24.869	8.691	2.036	34359	3000	4.412	4.346	158.120	0.256	687.149	1.89E+01
4.377	29.624	29.181	8.623	2.389	261523	3000	1.016	1.001	5226.342	8.470	22533.192	6.24E+02
8.953	38.577	37.999	8.819	3.111	386051	3000	1.012	0.997	7745.434	12.553	68303.561	9.25E+02
8.747	47.324	46.615	8.616	3.817	366160	3000	1.013	0.998	7339.104	11.894	63233.266	8.76E+02
8.683	56.007	55.169	8.553	4.517	79515	3000	5.067	4.991	318.625	0.516	2725.260	3.80E+01
8.758	64.765	63.795	8.626	5.224	40836	3000	5.065	4.989	163.699	0.265	1412.127	1.95E+01
8.743	73.508	72.407	8.612	5.929	13666	1800	5.065	4.989	91.305	0.148	786.359	1.09E+01
9.020	82.528	81.292	8.885	6.656	8529	1800	5.065	4.989	56.983	0.092	506.301	6.80E+00
8.604	91.132	89.767	8.475	7.350	5343	1800	5.065	4.989	35.697	0.058	302.527	4.26E+00
8.726	99.858	98.362	8.595	8.054	3775	1800	5.065	4.989	25.221	0.041	216.784	3.01E+00
8.735	108.593	106.967	8.605	8.758	2616	1800	5.066	4.990	17.474	0.028	150.361	2.09E+00
9.033	117.626	115.865	8.898	9.487	1208	1800	5.066	4.990	8.069	0.013	71.796	9.64E-01
9.058	126.684	124.787	8.923	10.218	1732	1800	4.984	4.909	11.760	0.019	104.928	1.40E+00
8.873	135.557	133.528	8.740	10.933	999	1800	5.065	4.989	6.674	0.011	58.338	7.97E-01
9.223	144.781	142.613	9.085	11.677	701	1800	5.062	4.986	4.686	0.008	42.576	5.60E-01

### Cycle 3 Breakthrough Performance

								Sample activity
								(CPM/mL)
Effluent bottle tare	306.2	g			Counts	Count time (s)	Sample mass	
Feed density	1.257	g/mL	Fee	ed 1	14833	300	6.384	584.119
Cs concentration	2.18E-04	Μ	Fee	ed 2	14880	300	6.323	591.670
Bed height in 0.25M NaOH	6.5	cm	Fee	ed 3	13900	300	6.384	547.378
Bed volume in 0.25M NaOH	11.505	mL	Fee	ed 4	15235	300	6.323	605.786
Time between samples	3.17	hours	Fee	ed 5	155085	3000	6.384	610.720
								587.934

Effluent bottle	Mass of feed	Volume flow	Volume of feed	Bed volumes of	Counts	Count time (s)	Sample mass	Sample volume	Sample activity	C/C0 (%)
mass (g)	processed (g)	rate (mL/hr)	processed (mL)	feed processed			(g)	(mL)	(CPM/mL)	
426.8	120.6	30.3	100.9	8.8	29	3000	6.288	5.002	0.116	0.020
548.7	242.5	30.6	203.0	17.6	138	3000	6.389	5.083	0.543	0.092
659.8	353.6	27.9	296.3	25.8	703	3000	6.119	4.868	2.888	0.491
780.9	474.7	30.4	397.3	34.5	2574	3000	5.895	4.690	10.977	1.867
902.1	595.9	30.4	498.3	43.3	5941	3000	5.750	4.574	25.975	4.418
1023.4	717.2	30.5	599.8	52.1	13086	3000	6.359	5.059	51.735	8.799
1143.8	837.6	30.2	700.5	60.9	4224	600	6.072	4.831	87.443	14.873
1262.3	956.1	29.8	799.9	69.5	7005	600	6.549	5.210	134.452	22.869

#### Cycle 3 Elution Performance (down-flow)

Feed density	1.257 g/mL
0.5M HNO3 density	1.015 g/mL
Bed height in 0.25M NaOH	6.500 cm
Bed volume in 0.25M NaOH	11.505 mL

Eluant mass	Eluant flow rate	Cumulative	Cumulative	Bed volumes of	Counts	Count time (s)	Sample mass	Sample volume	Sample activity	C/C0	Error C/C0	Total CPM in
processed	(mL/h)	eluant mass	eluant volume	eluant					(CPM/mL)			collected
		processed	processed	generated								fraction
4.993	9.836	4.993	4.918	0.427	211	3000	4.993	4.918	0.858	0.001	0.002	4
4,183	8.241	9.176	9.039	0.786	179	3000	4,183	4.120	0.869	0.001	0.002	4
4 725	9.309	13 901	13 693	1 190	197	3000	4 725	4 655	0.846	0.001	0.002	4
4 692	0.000	10 502	10.000	1 501	222	2000	4 692	4 612	1.006	0.002	0.002	-
4.002	9.224	18.363	18.305	1.091	232	3000	4.002	4.012	1.000	0.002	0.002	5
5.199	10.242	23.782	23.426	2.036	1138	3000	5.199	5.121	4.445	0.008	0.004	23
4.960	9.771	28.742	28.312	2.461	2019	3000	4.960	4.886	8.265	0.014	0.005	40
5.182	10.209	33.924	33.416	2.904	1077	3000	5.182	5.104	4.220	0.007	0.003	22
5.032	9.913	38.956	38.373	3.335	10829	900	0.020	0.020	36463.021	62.019	0.325	180735
5.182	10.209	44.138	43.477	3.779	15356	900	0.020	0.019	53025.208	90.189	0.392	270663
5.032	9.913	49.170	48.434	4.210	25383	300	6.123	6.031	841.692	1.432	0.049	4172
5 669	11 168	54 839	54 018	4 695	3485	300	5 669	5 584	124 818	0.212	0.019	697
8 391	8 265	63 230	62 283	5 414	3236	300	5.972	5.883	110 020	0.187	0.018	909
0.051	0.203	72 402	72.007	0.414	0200	300	4.004	3.005	00.507	0.107	0.017	505
9.953	9.604	73.163	72.087	0.200	2308	300	4.961	4.906	90.527	0.164	0.017	940
9.975	9.826	83.158	81.913	7.120	2116	300	4.923	4.849	87.270	0.148	0.016	857
9.994	9.844	93.152	91.757	7.975	1930	300	4.989	4.914	78.554	0.134	0.015	773
9.851	9.704	103.003	101.461	8.819	1769	300	4.991	4.916	71.965	0.122	0.014	698
10.022	9.872	113.025	111.333	9.677	1520	300	4.874	4.801	63.326	0.108	0.014	625
10.154	10.002	123.179	121.335	10.546	1370	300	4.954	4.880	56.150	0.096	0.013	562
9,989	9.839	133,168	131.174	11.401	1287	300	4,906	4.832	53,269	0.091	0.012	524
0.034	0.785	143 102	140.050	12 252	1248	300	4 022	4 848	51 482	0.088	0.012	504
10.020	0.990	152 122	140.939	12.232	7671	2006	4.017	4.040	47.260	0.000	0.012	469
10.030	9.000	103.132	150.859	13.111	7071	2000	4.917	4.043	47.309	0.081	0.012	400
9.856	9.708	162.988	160.548	13.955	3134	900	5.067	4.991	41.861	0.071	0.011	406
10.006	9.856	172.994	170.404	14.811	3128	900	5.081	5.005	41.666	0.071	0.011	411
10.276	10.122	183.270	180.526	15.691	2773	900	5.095	5.019	36.835	0.063	0.010	373
10.101	9.950	193.371	190.476	16.556	2776	900	4.985	4.910	37.689	0.064	0.010	375
10.015	9.865	203.386	200.341	17.413	2476	900	5.058	4.982	33.131	0.056	0.010	327
9.997	9.847	213.383	210.188	18.269	2155	900	5.100	5.024	28.598	0.049	0.009	282
10.367	10.212	223,750	220,400	19.157	2105	900	5.067	4,991	28.117	0.048	0.009	287
9 907	9 759	233 657	230 159	20.005	1878	900	5.009	4 934	25 375	0.043	0.009	248
10.012	0.962	242.670	240.022	20.000	1902	000	4.092	4.007	25.515	0.040	0.000	254
10.013	9.003	243.070	240.022	20.802	1092	900	4.962	4.907	25.705	0.044	0.009	204
10.145	9.993	253.815	250.015	21.731	1664	900	5.725	5.639	19.672	0.033	0.008	197
10.113	9.962	263.928	259.976	22.597	1809	900	4.916	4.842	24.905	0.042	0.008	248
10.105	9.954	274.033	269.930	23.462	1633	900	4.822	4.750	22.920	0.039	0.008	228
10.089	9.938	284.122	279.868	24.326	1587	900	4.923	4.849	21.818	0.037	0.008	217
10.090	9.939	294.212	289.807	25.190	1486	900	4.947	4.873	20.330	0.035	0.008	202
10.093	9.942	304.305	299.749	26.054	2818	1800	4.826	4.754	19.760	0.034	0.008	196
10.166	10.014	314.471	309.763	26.924	2216	1200	4.950	4.876	22.724	0.039	0.008	228
10 113	9 962	324 584	319 724	27 790	2975	1200	4 832	4 760	31 252	0.053	0.010	311
10 169	10.017	334 753	329 741	28 661	2847	900	5 196	5 1 1 8	37 083	0.063	0.010	371
10.056	0.005	344 000	330 646	20.001	2351	000	4 974	4 201	32 646	0.056	0.010	300
10.000	9.905	344.009	339.040	29.522	2301	900	4.0/4	4.001	32.040	0.000	0.010	323
10.186	10.033	354.995	349.680	30.394	1967	900	4.882	4.809	27.269	0.046	0.009	2/4
10.087	9.936	365.082	359.616	31.257	1613	900	4.751	4.680	22.978	0.039	0.008	228
10.018	9.868	375.100	369.484	32.115	1433	900	4.788	4.716	20.256	0.034	0.008	200
10.051	9.901	385.151	379.384	32.976	1738	1200	4.926	4.852	17.909	0.030	0.007	177
9.918	9.770	395.069	389.154	33.825	1200	900	4.723	4.652	17.196	0.029	0.007	168
10.082	9.931	405.151	399.085	34.688	1276	900	4.768	4.697	18.112	0.031	0.007	180
10.091	9,940	415.242	409.025	35.552	1440	900	5.027	4,952	19.388	0.033	0.007	193
10.024	9 874	425 266	418 899	36 410	1359	900	5.076	5 000	18 120	0.031	0.007	179
10 246	10 101	135 612	420.000	37 206	1659	1200	5.044	4.066	16 605	0.020	0.007	170
0.047	0.191	433.012	429.090	31.290	1036	1200	5.041	4.300	10.090	0.028	0.007	170
9.847	9.700	445.459	438.789	38.139	2054	1800	5.060	4.984	13./3/	0.023	0.006	133
10.907	10.744	456.366	449.533	39.073	1913	1800	5.083	5.007	12.736	0.022	0.006	137
9.258	9.119	465.624	458.652	39.865	1404	1800	5.098	5.022	9.320	0.016	0.005	85
10.191	10.038	475.815	468.691	40.738	1322	1800	5.035	4.960	8.885	0.015	0.005	89
10.096	9.945	485.911	478.636	41.602	1057	1800	4.994	4.919	7.162	0.012	0.005	71
9.961	9.812	495.872	488.448	42.455	535	1200	5.087	5.011	5.338	0.009	0.004	52
7.949	7.830	503.821	496.278	43.136	678	1800	5.092	5.016	4.506	0.008	0.004	35

Cycle 3 Operational Details and A	Activity Bala	nce (down-	flow elutio	on)		
Density of 0.25M NaOH	1.0095	g/mL				
Density of 0.1M NaOH	1.0039	g/mL				
Density of 0.5M HNO3	1.0152	g/mL				
Regeneration						
Tare mass of effluent bottle	24.5	g				
Final mass of effluent bottle	81.6	g				
Mass of feed processed	57.1	g, or	56.6	mL		
Start date and time	7/23/01 7:15					
Average flow rate	8.70	mL/h or	0.8	BV/h		
Bed height	6.5	cm				
Bed volume	11.5	mL				
Food						
Starting mass of feed bottle	2422 1	a				
Final mass of feed bottle	1366.5	g				
Mass of feed processed	1055.6	g or	840	mL		
Average flow rate	30.02	mL/h or	2.6	BV/h		
Acivity concentration in feed	467.7	CPM/g				
l otal activity processed	4.94E+05	CPM				
Bed volume	5.7	ml				
Dearbhanno	10.1					
Simulated LAW Effluent						
Total acitvity in samples	1572.3	CPM				
Activity in bulk composite sample	4081	counts in	1800	seconds of mass	6.253	g
Composite bulk activity concentration	21.8	CPIM/g				
Total activity in bulk effluent	20799.9	9 CPM				
Total activity in simulated LAW effluent	2.24E+04	CPM, or	4.5%	of feed		
Feed Displacement						
l are mass of effluent bottle	24.4	g				
Mass of feed processed	93.4	g	68.7	ml		
Start date and time	7/24/01 17:23	g 0.	00.1			
Finish date and time	7/24/01 19:23					
Average flow rate	34.37	mL/h or	3.0	BV/h		
Activity in bulk composite sample	16485	counts in	3000	seconds of mass	5.605	g
Composite bulk activity concentration	58.8 4 06E±03	CPM/g	0.8%	of feed		
Bed height	4.00 <u><u></u>+03</u> 6.3	cm	0.0 %	orieed		
Bed volume	11.2	mL				
Water Rinse						
l are mass of effluent bottle	24.4	g				
Mass of feed processed	62.1	y a or	62 1	ml		
Start date and time	7/24/01 19:26	3,				
Finish date and time	7/24/01 21:26					
Average flow rate	31.05	mL/h or	2.7	BV/h		
Activity in bulk composite sample	627	counts in	3000	seconds of mass	5.026	g
Composite bulk activity concentration	2.5 1.55E±02	CPM/g	0.0%	of feed		
Bed height	6.5	cm	0.070	orieed		
Bed volume	11.5	mL				
Elution	500.004		400.0			
l otal mass of eluant processed	503.821	g or ml /b or	496.3	BV/b		
Activity in bulk composite sample	73306	counts in	900	seconds of mass	4.971	a
Composite bulk activity concentration	983.1	CPM/g				5
Total activity in effluent	4.95E+05	CPM, or	100.3%	of feed		
Bed height	4.4	cm				
Bed volume	7.8	mL				
Water Rinse						
Tare mass of effluent bottle	59.2	g				
Final mass of effluent bottle	296.4	g				
Mass of feed processed	237.2	g, or	237.2	mL		
Start date and time	7/26/01 23:52					
Finish date and time	//2//01 19:40	ml /h or	10	B\//b		
Activity in bulk composite sample	153	counts in	1800	seconds of mass	5.068	a
Composite bulk activity concentration	1.0	CPM/g				5
Total activity in effluent	2.39E+02	CPM, or	0.0%	of feed		
Bed height	4.4	cm				
Bed volume	7.8	mL				
Total						
Total activity in all effluents	522138.7	CPM				
Total activity in feed	493734.0	CPM				
Activity recovery, as fraction of feed	105.8%					

## Cycle 4 Breakthrough Performance

Effluent bottle tare	128.2	g
Feed density	1.257	g/mL
Cs concentration	2.27E-04	Μ
Bed height in 0.25M NaOH	6.5	cm
Bed volume in 0.25M NaOH	11.505	mL
Time between samples	3.17	hours

	Counts	Count time (s)	Sample mass	CPM/mL
Feed 1	16771	300	6.924	608.930
Feed 2	16263	300	6.412	637.635
Feed 3	16859	300	6.924	612.125
Feed 4	14127	300	6.412	553.888
				603.144

Effluent bottle mass (g)	Mass of feed processed (g)	Volume of feed processed (mL)	Flow rate (mL/h)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	CPM/mL	C/C0 (%)	Total CPM broken through
248.5	120.3	100.635	31.779	8.747	10	3000	6.198	4.931	0.041	0.007	2.041
370.2	242.0	202.363	32.125	17.589	23	3000	6.172	4.910	0.094	0.016	6.828
490.7	362.5	303.134	31.823	26.348	39	3000	6.170	4.909	0.159	0.026	12.727
610.9	482.7	403.732	31.768	35.092	223	1800	6.251	4.973	1.495	0.248	83.177
733.8	605.6	506.531	32.463	44.027	1355	1800	6.318	5.026	8.986	1.490	538.712
852.6	724.4	605.727	31.325	52.649	4273	1800	5.890	4.686	30.397	5.040	1953.338
973.1	844.9	706.421	31.798	61.401	4224	600	6.072	4.831	87.443	14.498	5932.901
1093.8	965.6	807.653	31.968	70.200	7005	600	6.549	5.210	134.452	22.292	11231.513

#### Cycle 4 Elution Performance (up-flow)

Feed density	1.257 g/mL
0.5M HNO3 density	1.015 g/mL
Bed height in 0.25M NaOH	6.500 cm
Bed volume in 0.25M NaOH	11.505 mL

Eluant mass	Eluant flow rate	Cumulative	Cumulative	Bed volumes of	Counts	Count time (s)	Sample mass	Sample volume	Sample activity	C/C0	Total CPM in
processed	(mL/h)	eluant mass	eluant volume	eluant					(CPM/mL)		collected
		processed	processed	generated							fraction
4.528	8.919	4.528	4.460	0.388	140	1800	4.518	4.450	1.049	0.002	5
4.906	9.665	9.433	9.292	0.808	391	1800	4.906	4.832	2.697	0.004	13
4.954	9.760	14.388	14.172	1.232	508	1800	4.954	4.880	3.470	0.006	17
4.951	9.753	19.338	19.049	1.656	5620	1800	4.951	4.877	38.415	0.064	187
5.510	10.855	24.848	24.476	2.127	3536	1200	0.020	0.020	8974.368	14.879	48708
6.480	12.766	31.328	30.859	2.682	5090	600	0.019	0.019	27196.674	45.091	173596
5.016	9.882	36.344	35.800	3.112	3586	600	0.018	0.018	20225.040	33.533	99930
5.342	10.524	41.686	41.062	3.569	3266	900	0.018	0.018	12280.160	20.360	64618
4.804	9.464	46.490	45.794	3.980	2498	900	0.017	0.017	9944.979	16.489	47060
4.917	9.687	51.407	50.638	4.401	901	900	0.019	0.019	3209.457	5.321	15545
5.098	10.043	56.505	55.659	4.838	1326	1200	0.019	0.019	3542.514	5.873	17789
9,975	9.826	66.480	65,485	5.692	18931	100	5.064	4,988	2277.103	3,775	22374
10.022	9.872	76 502	75.357	6 550	54851	300	5 064	4 988	2199 239	3 646	21711
10.042	9.892	86 544	85 248	7 410	77653	900	5.041	4 966	1042 562	1 729	10313
10.122	9.970	96.666	95 219	8 276	8756	900	5.089	5.013	116 448	0.193	1161
10.122	0.020	106 727	105 120	0.270	4410	900	5.003	5.013	E9 672	0.195	E02
0.002	0.942	116 720	114 092	0.004	4907	900	5.007	5.011	65 216	0.097	642
9.993	9.043	116.730	114.962	9.994	4097	900	5.062	5.006	65.210	0.106	642
10.195	10.040	120.923	125.023	10.007	0141	1200	5.096	5.020	01.109	0.101	014
10.165	10.013	137.088	135.036	11.737	7025	1200	5.084	5.008	70.139	0.116	702
10.126	9.974	147.214	145.010	12.604	6360	1200	5.081	5.004	63.544	0.105	634
9.956	9.807	157.170	154.817	13.456	4598	1200	5.095	5.019	45.809	0.076	449
10.253	10.099	167.423	164.916	14.334	3616	1200	5.093	5.017	36.039	0.060	364
9.954	9.805	177.377	174.721	15.187	3188	1200	5.076	5.000	31.880	0.053	313
10.206	10.053	187.583	184.775	16.060	3129	1200	5.069	4.993	31.333	0.052	315
10.016	9.866	197.599	194.641	16.918	3180	1200	5.074	4.998	31.813	0.053	314
10.099	9.948	207.698	204.588	17.783	2670	1200	4.952	4.878	27.369	0.045	272
9.974	9.825	217.672	214.413	18.637	2117	1200	4.844	4.771	22.184	0.037	218
10.238	10.085	227.910	224.498	19.513	6068	1200	5.082	5.006	60.608	0.100	611
9.930	9.781	237.840	234.279	20.363	2335	1200	5.089	5.013	23.290	0.039	228
10.180	10.028	248.020	244.307	21.235	1212	1200	5.067	4.991	12.142	0.020	122
10.001	9.851	258.021	254.158	22.091	2013	1200	5.083	5.007	20.102	0.033	198
10.248	10.095	268.269	264.253	22.968	1171	1200	5.158	5.081	11.524	0.019	116
10.061	9.910	278.330	274.163	23.830	1494	1200	5.083	5.007	14.919	0.025	148
10.041	9.891	288.371	284.054	24.690	1450	1200	5.097	5.021	14.440	0.024	143
10.129	9.977	298.500	294.031	25.557	1426	1200	5.099	5.023	14.196	0.024	142
9.927	9.778	308.427	303.809	26.407	1169	1200	5.090	5.014	11.658	0.019	114
10.161	10.009	318.588	313.818	27.277	1131	1200	5.094	5.018	11.270	0.019	113
10.098	9,947	328,686	323,765	28,141	1207	1200	5,103	5.027	12.006	0.020	119
9,988	9.838	338.674	333.603	28,996	1145	1200	5.047	4.971	11.516	0.019	113
10.071	9 920	348 745	343 524	29.859	1117	1200	5.071	4 995	11 181	0.019	111
9 988	9.838	358 733	353 362	30 714	1056	1200	5.033	4 958	10.650	0.018	105
10.016	9,866	368 749	363 228	31 571	911	1200	5.062	4 986	9 135	0.015	90
10.010	10.020	378 921	373 248	32 442	836	1200	5.058	4.982	8 390	0.013	84
0.002	0.754	300.021	383 003	33 200	000	1200	5.000	5.014	0.000	0.015	04
9.902	9.704	200.023	202.002	33.290	920	1200	5.090	5.014	9.204	0.015	90
9.973	9.624	390./90	392.825	34.144	950	1200	5.082	5.006	9.489	0.016	93
10.085	9.934	408.881	402.759	35.007	1018	1200	5.047	4.9/1	10.238	0.017	102
10.034	9.884	418.915	412.643	35.866	899	1200	5.031	4.956	9.070	0.015	90
9.969	9.820	428.884	422.462	36.720	1312	1800	5.091	5.015	8.721	0.014	86
10.024	9.874	438.908	432.336	37.578	1087	1800	5.057	4.981	7.274	0.012	72

Cycle 4 Operational Details and Activity Balance (up-flow elution) Data Density of 0.25M NaOH 1.0095 g/mL Density of 0.1M NaOH 1.0039 g/mL Density of 0.5M HNO3 1.0152 g/mL Regeneration Tare mass of effluent bottle 24.6 g Final mass of effluent bottle 83.6 g Mass of feed processed 59 g, or 58.4 mL 8/6/01 7:15 Start date and time Finish date and time 8/6/01 13:15 Average flow rate 9.7 mL/h or 0.8 BV/h 6.4 cm Bed height Bed volume 11.3 mL Feed Starting mass of feed bottle 2424.3 g Final mass of feed bottle 1399.3 g Mass of feed processed 1025 g or 815 mL 31.9 mL/h or BV/h Average flow rate 10.1 479.8 CPM/g Acivity concentration in feed 4.92E+05 CPM Total activity processed Bed height 5.6 cm Bed volume 9.9 mL Simulated LAW Effluent Total acitvity in samples 1319.4 CPM Activity in bulk composite sample 1190 counts in 1800 seconds of mass 6.286 g Composite bulk activity concentration 6.3 CPM/g Total mass of effluent 956.1 g Total activity in bulk effluent 6033.3 CPM Total activity in simulated LAW effluent 7.35E+03 CPM, or 1.5% of feed Feed Displacement Tare mass of effluent bottle 24.6 g Final mass of effluent bottle 93.2 g Mass of feed processed 68.6 68.3 mL g or Start date and time 8/7/01 16:05 Finish date and time 8/7/01 18:05 34.2 mL/h or BV/h Average flow rate 3.0 Activity in bulk composite sample 5.676 g 11737 counts in seconds of mass 1800 68.9 CPM/a Composite bulk activity concentration 4.73E+03 CPM, or Total activity in effluent 1.0% of feed Bed height NM cm Water Rinse Tare mass of effluent bottle 24.4 a Final mass of effluent bottle 84.8 g Mass of feed processed 60.4 60.4 mL g, or Start date and time 8/7/01 18:07 Finish date and time 8/7/01 20:07 Average flow rate 30.2 mL/h or 2.6 BV/h Activity in bulk composite sample 360 5.055 counts in 1800 seconds of mass g 2.4 CPM/g Composite bulk activity concentration Total activity in effluent 1.43E+02 CPM, or 0.0% of feed Bed height NM cm Elution 438.9077 g or Total mass of eluant processed 432.3 mL 0.9 Average flow rate 9.96 mL/h, or BV/h Activity in bulk composite sample 25842 counts in 300 seconds of mass 5.087 g Composite bulk activity concentration 1016.0 CPM/g 4.46E+05 CPM, or Total activity in effluent 90.7% of feed Bed height NM cm Water Rinse Tare mass of effluent bottle 24.4 g Final mass of effluent bottle 87.1 g Mass of feed processed 62.7 62.7 mL g, or Start date and time 8/9/01 15:59 Finish date and time 8/9/01 18:00 Average flow rate 31.1 mL/h or BV/h 2.7 Activity in bulk composite sample 4236 counts in 1800 seconds of mass 5.062 g Composite bulk activity concentration 27.9 CPM/g Total activity in effluent 1.75E+03 CPM, or 0.4% of feed Bed height NM cm Total Total activity in all effluents 459904.4 CPM

#### B.8

491824.2 CPM

93.5%

Total activity in feed

Activity recovery, as fraction of feed

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