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Chemical Degradation of SuperLig®644 Ion Exchange Resin

S. T. Arm
D. L. Blanchard
S. K. Fiskum
D. R. Weier

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WTP
Project
Report

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Test specification: None
Test plan: TP-RPP-WTP-091, Rev. 0
Test exceptions: None
R&T focus area: Pretreatment
Test Scoping Statement(s): B-53

Battelle—Pacific Northwest Division
Richland, Washington 99352

Completeness of Testing

This report describes the results of work and testing specified by TP-RPP-WTP-091, Rev. 0. The work and any associated testing followed the quality assurance requirements outlined in the Test Specification/Plan. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Test plan results are reported. Also reported are any unusual or anomalous occurrences that are different from expected results. The test results and this report have been reviewed and verified.

Approved:

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

Summary

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 ^{137}Cs and ^{99}Tc by ion exchange from the liquid portion of the waste. SuperLig® 644 (SL-644) and SuperLig® 639 (SL-639) ion exchange resins were selected by the project to perform ^{137}Cs and ^{99}Tc separations, respectively.

Objectives

The primary objective of this task was to determine the degradation in SL-644 resin performance over repeated cycles of waste processing and elution in a column system. Secondary objectives included:

- Determination of the extent to which the SL-644 resin consumes oxygen by monitoring the oxygen concentrations in the feeds and effluents. Steimke et al. (2001) hypothesized that SL-644 resin oxidation is a major contributor to its chemical degradation.
- Preliminary assessment of some hazardous waste characteristics of the resin to support future development of a spent-resin disposal pathway.
- Determination of the impact of low-activity waste (LAW) processing rate and Cs^+, K^+, and OH^- concentrations on the resin breakthrough performance.

This investigation was conducted according to the test plan prepared by Arm (2001) in response to the test requirements for investigating ion exchange resin chemical degradation delineated by Barnes et al. (2001) in Section 3.7.2.3 of the Research and Technology Plan and test scoping statement B-53. All of the test objectives were achieved.

Conduct of Testing

Tests were performed using a simulated 241-AN-105 (AN-105) LAW. The simulated AN-105 LAW recipe was modified to include the toxicity characteristic (TC) metals (Ag, As, Ba, Cd, Cr, Hg, Pb, and Se) at concentrations corresponding to the highest observed in actual LAW samples so that the hazardous waste characterization of the resin was determined for the worst-case conditions. The K^+, OH^-, and Cs^+ concentrations were also modified for conducting the parametric study investigating the impact of these parameters on breakthrough performance. The simulated LAW was traced with ^{137}Cs so that process samples could be analyzed by gamma energy analysis (GEA).

The test apparatus consisted of an ion exchange column containing nominally 10 mL of SL-644 resin expanded in 0.25 M NaOH, a metering pump, a pressure-relief valve, a pressure gage, and three 3-way valves. Probes positioned on the column feed and effluent lines were linked to a spectrometer that measured dissolved oxygen.

The SL-644 resin was conditioned with 0.5M HNO_3 before commencing cycle testing. The SL-644 resin underwent 25 cycles of simulated waste processing, elution and regeneration to accomplish this objective. A cycle test commenced with converting the resin to the sodium form by pumping 0.25 M NaOH through the bed. The simulated LAW was then processed followed by column rinses of 0.1 M NaOH and de-ionized (DI) water before the resin was eluted with 0.5 M HNO_3. The cycle finished with a rinse of DI water.

Simulated LAW effluent samples were periodically collected by directing the flow into 20-mL vials to collect nominally 5 mL of sample. The bed height and effluent bottle mass were measured during sampling events. For most cycles, eluate was collected in a single bottle, and then the final 5 mL was collected separately for GEA to determine the ^{137}Cs content. To ascertain the elution profile, eluate was collected during cycles 1, 2, 5, 10, 15, 20, and 25 into 20-mL vials that were changed every hour to provide 10-mL fractions. Samples were analyzed by GEA for their ^{137}Cs content. The oxygen concentration in the feed and effluent was periodically measured and automatically logged on a computer.

Cycles 1, 5, 10, 15, 20 and 25 processed simulated LAW at a consistent flow rate with consistent Cs^+, K^+ and OH^- concentrations to assess chemical degradation of the resin. In the remaining cycles, the values of these parameters and eluant flow rate were varied according to a partial factorial test design to ascertain their impacts on performance. The spent resin was examined by scanning electron microscopy and subjected to a modified toxicity characteristic leach procedure before digestion to facilitate determination of its metals content.

Results and Performance against Objectives

The column-distribution coefficient (number of bed volumes processed at 50% Cs breakthrough) progressively decreased by ~4 BV each cycle so that the resin appeared to lose approximately 60% of its effective capacity for Cs^+ over the course of the 25 cycles based on the volume of the bed in 0.25 M NaOH. However, note that ~34% of the resin mass was also lost over this period and the 26% reduction in effective capacity accounting for the mass loss is presumably due to chemical alteration of the ion exchange sites. Greater volumes of eluant were required to elute the same volume of resin with each successive cycle, although this phenomenon was totally masked by the resin mass loss such that elution performance did not apparently deteriorate.

The SL-644 resin was found to dissolve at an average rate of 1.4 wt% per cycle so that the total mass loss from the bed after 25 cycles was 34%. The acid-form resin bed volume also decreased at the same rate, although the fully expanded bed volume in 0.25 M NaOH remained constant such that the bed density also decreased by 34%. The acid-form resin bed volume also decreased at the same rate. These observations are consistent with scanning electron microscopy examination of the resin that showed the used resin to have apparently higher porosity than the fresh material.

In general, the oxygen consumption rate increased with increasing concentration of dissolved oxygen in the feedstock and was greatest for the resin in the Na form. Consistent with this pattern, the oxygen consumption rate processing 0.25M NaOH during resin regeneration was 0.84 $\mu molg^{-1}h^{-1}$ and decreased to 0.32 $\mu molg^{-1}h^{-1}$ when processing LAW of lower dissolved oxygen content. The dissolved oxygen concentration was higher in the 0.1M NaOH feedstock than LAW during feed displacement such that the consumption rate increased to 2.4 $\mu molg^{-1}h^{-1}$. The consumption rate increased further to 3.4 $\mu molg^{-1}h^{-1}$ in the subsequent water rinse. During elution, the consumption rate decreased from 1.1 $\mu molg^{-1}h^{-1}$ to 0.71 $\mu molg^{-1}h^{-1}$ as the resin was converted to the acid form. The consumption rate in the final water rinse was 1.8 $\mu molg^{-1}h^{-1}$, lower than in the water rinse following feed displacement because the resin was in the acid form.

The modified Toxicity Characteristic Leach Procedure (TCLP) indicated that the spent resin would not exhibit toxicity characteristics if a formal TCLP were performed. However, this result may not be appropriate for regulatory purposes or submissions since the TCLP had to be modified from the standard U.S. Environmental Protection Agency (EPA) SW-846 method due to the small sample size.

The parametric study showed that breakthrough performance deteriorated with increasing concentrations of Cs^+ and K^+, apparently according to a Langmuir-type adsorption model. There was no significant impact of OH^- concentration in the range 1.2M to 2.2M. Breakthrough performance improved with decreasing flow rate, indicating that diffusion in the mass-transfer film around the resin particles is significant in the ion exchange process. Eluant flow rate appeared to have no significant impact on the required volume of eluant.

Chemical analysis of the spent resin and TCLP leachate showed the spent resin to have contained Cs^+ at a concentration of 1.71 μg/g of resin air dried at room temperature (0.374 g/m^3). If the resin had been processing waste containing Cs of which a quarter was ^{137}Cs (typical for Envelope A or C LAW) then the residual activity would have been 37 μCi/g (8.1 Ci/m^3). If a value typical of Envelope B LAW of a third were assumed, then the residual ^{137}Cs activity would have been 50 μCi/g (11 Ci/m^3). These concentrations are factors of >1.4 higher than that of the 26 μCi/g[1] found by Kurath and Wagner (2000) on resin previously used to process actual AW-101 and AN-107 LAW samples. In addition, these values are between the Hanford Site Solid Waste Acceptance Criteria (McDowell (2002)) category 1 and 3 limits (5.5×10^{-3} Ci/m^3 and 12,000 Ci/m^3, respectively) indicating that the spent resin from WTP would have to be dispositioned in high integrity containers.

Preliminary analysis of the results presented in this report shows that the ion exchange bed would require replacement after completing the 14th cycle (a cycle consisting of the column processing LAW in the polishing, lag and lead positions and then undergoing elution and regeneration) to maintain plant throughput and consistent with Olson (2001) who assumes the bed would be replaced after the 10th cycle. The analysis assumes that the expected column distribution coefficient provided by Olson (2001) of 100 for processing Envelope A LAW in normal operations is an average over the resin service life and that degradation is assumed to be solely due to reaction with dissolved oxygen. The analysis does not account for degradation arising from irradiation.

Quality Assurance

PNWD implemented the RPP-WTP quality requirements in a quality assurance project plan (QAPjP) as approved by the RPP-WTP quality assurance (QA) organization. Test-preparation activities and the first 5 of 25 test cycles were conducted in accordance with PNWD's quality assurance project plan, CHG-QAPjP, Rev.0, which invoked PNWD's Standards Based Management System (SBMS), compliant with DOE Order 414.1A Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A – Quality Assurance Requirements. Due to a change in the contract QA requirements, the remainder of the cycle tests and analytical activities were conducted in accordance with PNWD's QA project plan, RPP-WTP-QAPjP, Rev.0, which invoked NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Part 2.7. These quality requirements were implemented through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP). Data quality was not affected by the change in QA requirements.

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 that the reported work satisfied the Test Plan objectives.

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[1] The value of 21.3 μCi/g of resin dried at 95°C and including interstitial solution actually reported by Kurath and Wagner (2000) is based on the sodium form resin from a different batch than used here. The value of 26 μCi/g is based on the expected equivalent acid form resin derived by applying the sodium to acid form resin mass conversion factor determined for the resin batch used for this study and presented in Section 2.3.

Issues

Resin life can be improved by minimizing the time the resin spends in the expanded, sodium form and its exposure to dissolved oxygen because test results indicate that resin oxidation is responsible for a significant portion of its chemical degradation and that oxygen consumption is greatest for the expanded resin. For example, an approximately five-fold improvement in the resin life may be realized by removing dissolved oxygen in the elution and regeneration reagents and could be accomplished by sparging the feedstocks with nitrogen.

Gas generation within the resin bed was observed during regeneration operations after cycle 15. Gas bubbles were either entrained into the effluent or accumulated within the bed to eventually achieve sufficient buoyancy to rise and occupy a portion of the headspace above the bed. The source and composition of the gas is unknown but was considered to originate from the resin since air in-leakage would have been manifested in other operations as well as regeneration. Consideration should be given to accommodating the generation and entrainment of gas bubbles in the WTP column design and associated safety analyses.

Acronyms, Abbreviations, and Definitions

AV	apparatus volume
BNI	Bechtel National, Inc.
BV	bed volume
CPM	counts per minute
CVAA	cold vapor atomic absorption
DI	de-ionized (water)
EPA	U.S. Environmental Protection Agency
FMI	Fluid Metering, Inc.
GEA	gamma energy analysis
HLW	high-level waste
HP	hot persulfate
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICP-MS	inductively coupled plasma-mass spectrometry
LAW	low-activity waste
NCAW	neutralized current acid waste
PNWD	Battelle, Pacific Northwest Division
QA	quality assurance
QAPjP	Quality Assurance Project Plan
RPP-WTP	River Protection Project – Waste Treatment Plant
SEM	scanning electron microscopy
SL	SuperLig®
SVOA	semi-volatile organic analysis

TC	toxicity characteristic
TCLP	Toxicity Characteristic Leach Procedure
TIC	total inorganic carbon
TOC	total organic carbon
TWINS	Tank Waste Information Network System
VOA	volatile organic analysis
WTPSP	Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual

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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 ^{137}Cs and ^{99}Tc by ion exchange from the liquid portion of the waste. SuperLig® 644 (SL-644) and SuperLig® 639 (SL-639) ion exchange resins were selected by the project to perform ^{137}Cs and ^{99}Tc separations, respectively, and are available from IBC Advanced Technologies, Inc., American Fork, Utah. Kurath (2000a), Kurath (2000b), and Hassan et al. (2000), for example, have tested these resins with actual waste and shown that they satisfy the performance criteria delineated by the RPP-WTP project.

The deterioration in performance of an ion exchange resin over repeated cycles of waste processing and elution is an important characteristic to understand for design and operational purposes. The rate of deterioration will determine the useful life of the resin and thereby the rate of its consumption and the quantity of spent resin for disposal. Chemical degradation of the SL-639 and SL-644 resins was investigated in a parallel suite of tests and reported separately. This report documents the testing, results, and analysis of the SL-644 chemical degradation.

1.2 Objectives

The primary objective of this task was to determine the deterioration in performance of the ion exchange resin SL-644 over repeated cycles of waste processing and elution in a column system. The SL-644 resin underwent 25 cycles of waste processing and elution to accomplish this objective. Secondary objectives include:

- Determination of the extent to which the SL-644 resin consumes oxygen by monitoring the oxygen concentrations in the feeds and effluents. Steimke et al. (2001) hypothesized that SL-644 resin oxidation is a major contributor to its chemical degradation
- Preliminary assessment of some hazardous waste characteristics of the resin to support future development of a spent resin disposal pathway.
- Determination of the impact of the LAW processing rate and Cs^+, K^+, and OH^- concentrations on the resin breakthrough performance.

This investigation was conducted according to the test plan prepared by Arm (2001) in response to the test requirements to investigate ion exchange resin degradation delineated by Barnes et al. (2001) in Section 3.7.2.3 of the Research and Technology Plan and test scoping statement B-53.

1.3 Purpose

This report documents the testing, results, and analysis associated with the SL-644 chemical degradation investigation. The purpose of the investigation was to provide information for an assessment of the degradation in resin performance over repeated cycles of waste processing and elution. The toxicity characteristics of the spent resin were determined to aid in determining the optimum disposal method for the spent resin arising from the WTP. 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 quality assurance (QA) organization. Test preparation activities and the first 5 of 25 test cycles were conducted in accordance with PNWD's quality assurance project plan, CHG-QAPjP, Rev.0, which invoked PNWD's Standards Based Management System (SBMS), compliant with DOE Order 414.1A Quality Assurance and 10 CFR 830, Energy/Nuclear Safety Management, Subpart A – Quality Assurance Requirements. Due to a change in the contract QA requirements, the remainder of the cycle tests and analytical activities were conducted in accordance with PNWD's quality assurance project plan, RPP-WTP-QAPjP, Rev.0, which invoked NQA-1-1989 Part I, Basic and Supplementary Requirements, and NQA-2a-1990, Subpart 2.7. These quality requirements were implemented through PNWD's Waste Treatment Plant Support Project Quality Assurance Requirements and Description Manual (WTPSP). Data quality was not affected by the change in QA requirements. Note that the TCLP sample analysis was not subject to the WTP QAPjP for environmental/regulatory data due to the research nature of the test.

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 that the reported work satisfied the Test Plan objectives.

2.0 Test Design and Operation

This section describes the process for preparing simulated AN-105 and AW-101 LAW, preparing reagents, and preparing and storing ion exchange resin. It also describes the batch-contact procedure, the ion exchange column test setup, the measurement and interpretation of dissolved oxygen, and the modified toxicity characteristic leach procedure.

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.

2.1.1 Simulated AN-105 LAW Preparation

The LAW currently stored in Tank 241-AN-105 (AN-105) was selected as that to simulate and test since processing of the LAW in this tank is scheduled for the WTP, and it contains a relatively high concentration of Cr (0.0312 M or 1,300 ppm). Kurath and Wagner (2000) have shown that a significant quantity of Cr remained in the SL-644 resin after elution following processing LAW samples from Tanks AW-101 (at a Cr concentration of 8.4E-4 M or 35 ppm) and AN-107 (at a Cr concentration of 8.8E-4 M or 37 ppm). This phenomenon would affect the hazardous waste characteristics of the resin since Cr is a toxicity characteristic (TC) metal. Chromium retention would also affect the resin performance since it would reduce the number of sites available after each cycle by either blocking, by its precipitation, or occupying ion exchange sites. AN-105 LAW is also representative of the envelope A type waste that will constitute the majority of the feed to the WTP.

The simulated AN-105 LAW recipe provided by Eibling and Nash (2001) was modified to include the TC metals (Ag, As, Ba, Cd, Cr, Hg, Pb and Se) at concentrations corresponding to the highest observed in actual LAW samples so that the hazardous-waste characterization of the resin was determined for the worst-case conditions. In addition, U was added at a concentration corresponding to the highest observed in actual LAW samples, since Kurath and Wagner (2000) have shown that a significant quantity remains on the SL-644 resin after elution. The applicable TC metal and U concentrations were determined by interrogating the Tank Waste Information Network System (TWINS). Only samples from the LAW currently scheduled for processing in the WTP (Tanks AN-102, AN-103, AN-104, AN-105, AN-107, AP-101, AW-101, AZ-101, AZ-102 and SY-101) were examined, and the highest concentrations, with respect to Na, were selected for the modified recipe.

The K^+, OH^- and Cs^+ concentration parametric study required selection of three concentrations for each of these constituents. The AN-105 LAW recipe concentrations of K^+ and OH^- detailed by Eibling and Nash (2001) were selected as baselines. The remaining concentrations were required to be symmetric about the baselines to provide minimum and maximum values. Concentrations bounding the range of potential values were determined by again interrogating the TWINS. As before, only samples from the LAW currently scheduled for processing in the WTP (Tanks AN-102, AN-103, AN-104, AN-105, AN-107, AP-101, AW-101, AZ-101, AZ-102 and SY-101) were examined. Appropriate values for the minimum and maximum values were then selected, given the need for symmetry and the bounding concentrations.

In addition, parallel SL-639 resin-degradation tests required the NO_3^- concentration to be varied in a parametric study. The KNO_3, NaOH, $NaNO_3$, and $NaNO_2$ concentrations were therefore optimized to fulfill the requirements of each test and maintain a consistent Na concentration since the effluent from the SL-644 test served as simulated LAW feed to the SL-639 test.

The baseline ^{133}Cs concentration was selected to provide 50% breakthrough when the equivalent of 150 bed volumes (BVs) of waste had been processed through the SL-644 column in the first cycle. Batch-contact data are usually used to determine this ^{133}Cs concentration but prior work by Arm et al. (2002) and Fiskum et al. (2002) showed this method to be unreliable. Instead, a baseline value of 0.122 mM (13 ppm) was selected based on prior work by Arm et al. (2002) and Fiskum et al. (2002). This value was confirmed to provide 50% breakthrough within the prescribed LAW processing period during the test. Appropriate maximum and minimum concentrations were then formulated. In addition, the ^{133}Cs concentration was progressively increased in a series of cycles up to the approximate maximum concentration of all Cs isotopes observed in actual LAW samples. The radioisotope ^{137}Cs was added to the simulated LAW as a tracer to a concentration of 0.1 mCi/L, or ~9×10^{-9} M.

The baseline modified recipe was used for Batches 1 through 4 and 9, and this is provided in Table 2.1, which also compares the modified and original (as provided by Eibling and Nash [2001]) recipes. Table 2.2 details the K^+, OH^-, NO_2^-, and NO_3^- concentrations in each batch prepared for the parametric study; other constituents, except Cs^+, were the same as in the baseline recipe. The minimum and maximum Cs^+ concentrations used in the parametric study were 0.061 mM and 0.235 mM, respectively, although concentrations up to 0.564 mM were also used in some cycles. The Cs^+ concentration used for each cycle is provided in the operations descriptions provided later in the report.

Batches of 5 L or 5.5 L were prepared for the column tests, and each batch was filtered following a week of mixing. The volume of material required for the next cycle was then extracted and further filtered. The requisite quantity of $CsNO_3$ was added, and the cycle batch was spiked with ^{137}Cs tracer to facilitate gamma emission analysis (GEA) immediately before processing.

The simulated AN-105 LAW feeds to cycles 1, 5, 10, 15, 20, and 25 were analyzed by the following methods.

- Ion chromatography (IC).
- Inductively coupled plasma – atomic emission spectrometry (ICP-AES).
- Inductively coupled plasma – mass spectrometry (ICP-MS).
- Carbon oxidation using hot persulfate (HP) for total inorganic and organic carbon (TIC and TOC).
- Carbon oxidation using a furnace for total carbon and TOC.
- Cold vapor atomic absorption (CVAA) spectroscopy for mercury (Batch 1 only).

Table 2.3 compares the target constituent concentrations with those determined by the appropriate analysis methods for the simulated LAW feeds. Batches 1, 2, and 8 served as feeds for Cycles 1, 5, and 25, respectively, while Batch 4 served as feed for Cycles 10, 15, and 20. The overall error for analytical results is estimated to be within 15% except those ICP-AES results in italics that are within 10 times their detection limits with errors likely exceeding 15%.

The presence of a significant quantity of black precipitate after a week of mixing indicates that not all of the reagents dissolved entirely. This is particularly true for the TC metals since they were added at quantities to maximize their individual concentrations in the simulated LAW matrix rather than to collectively simulate the contents of any particular tank. Na and Al appear to be the major constituents of the precipitate, probably as sodium aluminate, with some K. Of the TC metals, Ag, Ba, and Hg were at concentrations a factor of ~100 below their targets, although the others (As, Cr, Pb, and Se) were at concentrations close to their targets given the analytical errors. Ag, Ba and Hg probably precipitated as insoluble Cl^- and SO_4^- compounds.

Table 2.1. Simulated AN-105 LAW Component List

		Final Target Concentration		
Species	**Main Reagent Used**	Modified recipe		Original recipe from Eibling and Nash (2001)
		(mg/L)	(M)	(M)
Metals				
Aluminum	Sodium aluminate	19,900	7.36E-1	7.36E-1
Arsenic	Sodium arsenate	51.8	6.91E-4	not included in recipe
Barium	Barium nitrate	26.5	1.93E-4	not included in recipe
Cadmium	Cadmium nitrate	3.74	3.33E-5	1.47E-5
Calcium	Calcium nitrate	20.0	4.99E-4	4.99E-4
Cesium	Cesium nitrate	16.2	1.22E-4	6.09E-5
Chromium	Sodium chromate	1,620	3.12E-2	1.30E-2
Lead	Lead nitrate	80.8	3.90E-4	1.28E-4
Magnesium	Magnesium nitrate	2.70	1.11E-4	1.11E-4
Mercury	Mercuric (I) nitrate	0.263	1.31E-6	not included in recipe
Molybdenum	Potassium molybdate	41.0	4.27E-4	4.27E-4
Potassium	Potassium nitrate	3,720	9.51E-2	9.51E-2
Selenium	Selenium dioxide	52.9	6.70E-4	6.27E-6
Silicon	Sodium meta-silicate	106	3.76E-3	3.76E-3
Silver	Silver nitrate	22.3	2.07E-4	7.56E-5
Sodium	Various	123,000	5.34E00	5.34E00
Uranium	Uranyl nitrate	260	1.10E-3	not included in recipe
Zinc	Zinc nitrate	5.05	7.72E-5	7.72E-5
Cations				
Ammonium	Ammonium acetate	60	3.33E-3	3.33E-3
Boron	Boric acid	25.5	2.36E-3	2.36E-3
Anions				
Carbonate	Sodium carbonate	6,240	1.04E-1	1.04E-1
Chloride	Sodium chloride	4,540	1.28E-1	1.28E-1
Fluoride	Sodium fluoride	95	5.00E-3	5.00E-3
Hydroxide	Sodium hydroxide	29,200	1.72E00	1.72E00
Nitrate	Sodium nitrate	82,500	1.33E00	1.33E00
Nitrite	Sodium nitrite	55,700	1.21E00	1.21E00
Phosphate	Sodium phosphate	280	3.00E-3	3.00E-3
Sulfate	Sodium sulfate	390	4.01E-3	4.01E-3
Organic compounds				
Glycolic acid	Glycolic acid	830	1.09E-2	1.09E-2
Acetate	Sodium & ammonium acetate	1020	1.75E-2	1.75E-2
Formate	Sodium formate	1410	3.20E-2	3.20E-2
Oxalate	Sodium oxalate	310	3.47E-3	3.47E-3

Table 2.2. K^+, OH^-, NO_2^- and NO_3^- Concentrations in Simulated AN-105 LAW Batches

	Target Concentration (M)			
Species	**Batch 5**	**Batch 6**	**Batch 7**	**Batch 8**
K^+	0.008	0.008	0.80	0.80
OH^-	2.20	1.20	2.20	1.20
NO_3^-	0.10	0.50	1.36	1.36
NO_2^-	1.46	2.46	0.58	1.60

Table 2.3. Comparison of Analyzed and Target Simulated AN-105 LAW Composition

	Analysis	Concentration (mg/L)(1,7)				
Analyte	**Method**	**Target**	**Batch 1**	**Batch 2**	**Batch 4**	**Batch 8**
Ag	ICP-AES	22.3	<0.63	<0.63	<0.63	<0.63
Al	ICP-AES	19,900	15,900	15,600	16,300	16,200
As	ICP-AES	51.8	*54*	64.2	62.9	*61*
B	ICP-AES	25.5	110(5)	82	29.2	20.3(8)
Ba	ICP-AES	26.5	*0.40*	*0.39*	*0.56*	*0.55*
Ca	ICP-AES	20.0	<6.3	<6.3	<0.63	<0.63
Cd	ICP-AES	3.74	*2,2*	*1.3*	*0.91*	*1.4*
Cl	IC	4,540	4,760	6,300	5,500	4,600
Cr	ICP-AES	1,620	1,530	1,470	1,490	1,450
Hg	CVAA	0.263	4.46E-3	(4)	(4)	(4)
F(3)	IC	95	1,160	1,000	1,000	800
K	ICP-AES	3,720	3,610	3,070	3,190	3,350
Mg	ICP-AES	2.70	<2.5	<2.5	<2.5	3.3
Mo	ICP-AES	41.0	41.6	40.4	40.9	39.3(8)
Na	ICP-AES	123,000	121,000(11)	113,000(10)	115,000(10)	115,000
P	ICP-AES	280	152	125	127	93.2
Pb	ICP-AES	80.8	87.7	56.7	56.3	56.5
Se	ICP-AES	52.9	51	51	52	48
Si	ICP-AES	106	780(5)	156	120	89(8)
U	ICP-AES	260	110	<50	<50	<50
Zn	ICP-AES	5.05	5.3	5.0	4.8	17.7(6)
C_2O_4	IC	310	<500	<500	<500	300
NO_2^-	IC	55,700	62,200	56,900	57,900	57,900
NO_3^-	IC	82,500	81,200	82,900	83,500	84,200
PO_4^-	IC	280	<500	<500	1,300	1,300
SO_4^-	IC	390	<500	2,600	2,600	1,100
TIC	HP	1,250	1,390(12)	1,450	1,570	1,490
Total Carbon	Furnace	3,400	2,600	2,420	2,790	2,720
TOC	HP	1,150	<500	1,120	1,140	1,240
	Furnace		1,120	35(9)	45(9)	<170(9)
	Furnace Total Carbon – HP TIC(2)		1,210	970	1,220	1,230
Density (g/mL)	Not applicable	Not applicable	1.25	1.25	1.26	1.25

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus the best TOC result may be the difference between these measurements.
3. F^- results from IC should be considered upper bounds due to significant analytical interference from organic compounds such as acetate.
4. Not measured.
5. Si and B analysis compromised by error in sample preparation, manifesting in poor recoveries from spike samples.
6. Observed Zn concentration in the blank did not satisfy QC acceptance criteria, and this Zn concentration consequently was likely up to 75% over-estimated.
7. Reported results satisfy the WTP project QC criteria unless otherwise noted.
8. B, Mo, and Si achieved recoveries of 72%, 73%, and 59%, respectively, from the matrix spike sample and so did not satisfy the QC acceptance criterion of >75%. No significant impact on results expected.
9. TOC recoveries from the caustic matrix spike lower than the QC acceptance criterion makes this result doubtful. See also Note 2.
10. Relative % difference of 4.0% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
11. Relative % difference of 7.3% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.

2.1.2 Simulated AW-101 LAW Preparation

As described later, the first cycle test on the SL-644 with the modified simulated AN-105 LAW showed unexpectedly poor performance of the resin. Therefore, PNWD and RPP-WTP staff decided to perform a cycle test with a simulated AW-101 LAW for comparison to an earlier test. Golcar et al. (2000) provides the recipe and procedure for preparing this simulated LAW. The recipe, which was used with no modifications, is presented in Table 2.4. The solution was filtered immediately before processing. The test batch was spiked with ^{137}Cs as a tracer.

Table 2.4. Simulated AW-101 LAW Component List

Species	Final Target Concentration (M)	Formula Weight
EDTA	3.70E-03	292.24
Citric acid	3.70E-03	210.14
$Na_3HEDTA\text{-}2H_2O$	3.70E-03	344.00
Na_3NTA	3.70E-03	257.10
NaGluconate	3.70E-03	218.00
Na_2Iminodiacetate	3.70E-03	177.07
$Fe(NO_3)_3\text{-}9H_2O$	5.00E-05	404.02
$Mg(NO_3)_2\text{-}6H_2O$	1.50E-03	256.40
$Mn(NO_3)_2$, 50%	6.63E-05	4.3M
MoO_3	2.86E-04	143.95
$Ni(NO_3)_2\text{-}6H_2O$	1.33E-04	290.80
SiO_2	2.93E-03	60.08
$BaNO_3$	1.33E-04	261.38
$Ca(NO_3)_2$	4.13E-04	236.16
$Sr(NO_3)_2$	1.30E-05	211.65
$RbNO_3$	1.00E-05	147.47
$CsNO_3$	5.54E-04	194.92
$LiNO_3$	5.51E-04	69.00
KOH	4.30E-01	56.11
NaOH	3.89E+00	40.00
$Al(NO_3)_3\text{-}9H_2O$	5.06E-01	375.15
Na_2CO_3	1.00E-01	105.99
Na_2SO_4	2.36E-03	142.05
$NaHPO_4\text{-}7H_2O$	1.73E-03	268.07
NaCl	6.93E-02	58.45
NaF	1.10E-02	41.99
$NaNO_2$	7.90E-01	69.00

2.2 Reagent Preparation

All reagents were “reagent grade.” Sodium hydroxide solutions were prepared by dissolving the required mass of sodium hydroxide pellets in de-ionized (DI) water. The solution of 0.5 M HNO_3 was prepared by diluting the 68 to 70 wt% HNO_3 commercial stock with DI water.

2.3 Ion Exchange Resin Preparation and Storage

SL-644 Cs ion exchange resin from Batch 010319SMC-IV-73 was received in late March of 2001 in a dry, probably acid-form, with K_2CO_3 or $KHCO_3$ salts residual from its manufacture. The resin was immediately sieved to produce fractions with defined size ranges that were stored in plastic bottles. Table 2.5 presents the weight distribution determined from the sieving operation.

Table 2.5. Dry Weight Distribution of SL-644 Resin Batch 010319SMC-IV-73

Sieve Size(1)	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
1. U. S. standard sieve size corresponding to ASTM E-11 specification.		

The fraction defined by a particle-size range of 212 μm to 425 μm, or 24.09% of the total weight, was used throughout these tests for consistency with the size range used by Kurath et al. (2000a/b). Note that the tests described in Section 3.3 indicate that the resin volume was ~50% larger when submerged in simulated AN-105 LAW and in the Na form than when in the dry, as-received form. Table 2.6 presents various properties of the as-received resin and the resin in the size range of 212 to 425 μm, which was previously determined in March 2001 and reported by Fiskum et al. (2002), and determined in this work in June 2001.

Table 2.6. Physical Properties of Batch 010319SMC-IV-73 SL-644 Resin

Property	Value (Fiskum et al. (2002)	Value (Fiskum et al. (2002)	Value (this work)
Size range	As received	212 – 425 μm	212 – 425 μm
Bulk density (as-received form) (g/mL)	0.84	0.70	0.60
F factor (as-received form)	0.871	0.877	Not determined
F factor (acid form)	0.938	0.858	0.91
L factor (solids fraction remaining after conversion to H^+ form)	0.556	0.538	0.531(1)
I factor (mass increase from H^+ form to Na^+ form)	1.22	1.25	Not determined
1. Assumes the value of F (as-received form) determined by Fiskum et al. (2002)			

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

$$F = \frac{m_d}{m_i} \tag{1}$$

Here m_d is the mass of resin dried at 50°C under vacuum, and m_i is the initial mass of dry resin (dried under ambient conditions).

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

$$L = \frac{(m_H F_H)}{(m_i F)} \tag{2}$$

Here F_H is the F factor for the H^+ form resin, m_i is the initial mass of the as-received resin, and F is the F factor of the as-received resin.

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

$$I = \frac{m_{Na}}{m_H F_H} \tag{3}$$

Here m_{Na} is the dry mass of the Na^+ form resin.

The color of the 212- to 425-µm 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. For example, the dry-bed density was lower in June 2001 than when the resin was received in March 2001. Additionally, Fiskum et al. (2002) reported that resin in the 212- to 425-µm range occupied 20% less volume in 0.5 M nitric acid than in 0.25 M NaOH shortly after it was received. However, results reported herein indicate that it occupied ~50% less volume approximately 4 months after receipt.

2.4 Batch Contact Procedure

Batch-contact tests were performed to ascertain the equilibrium performance of the resin. The batch-contact tests were performed by contacting approximately 0.1 g of acid-form resin, measured to an accuracy of 0.001 g, of resin with 10 mL of simulated AN-105 LAW of baseline composition, but at different Cs^+ concentrations. Only the Na concentration in this batch of simulated LAW was determined at 5.09 M; Cs^+ concentrations were determined by calculation.

The resin was first converted to the H^+ form by contacting it with 0.5 M nitric acid. There was no significant impact on the simulated LAW composition as a result of the resin being in the acid form due to the large excess of solution. The simulated LAW was transferred to the contact vials by pipette, and the actual volume was determined from the net mass and density. The contact vials containing the resin and simulated LAW were shaken in a horizontal shaker for 24 hours at ambient temperature.

After contacting with the resin, the simulated LAW was filtered, and the ^{137}Cs concentrations were determined by GEA. The batch-distribution coefficients (K_d) were determined from the relationship,

$$K_d = \frac{(C_0 - C_1)}{C_1} \frac{V}{MF} \tag{4}$$

where,

C_0 and C_1 = initial and final ^{137}Cs concentrations
V = volume of simulated LAW (mL)
M = mass of ion exchange resin (g)
F = ratio of resin mass before and after drying.

Drying was performed at 50°C and used resin samples weighed at the same time as those for the batch contacts to assure applicability at the same atmospheric humidity.

2.5 Ion Exchange Column Test Setup

Figure 2.1 provides a process schematic of the apparatus. The apparatus consisted of an ion exchange column containing nominally 10 mL of SL-644 resin expanded in 0.25 M NaOH, a metering pump, pressure relief valve, pressure gage (indicated by ‘P’) and three 3-way valves. Probes positioned on the column feed and effluent lines were linked to a spectrometer that analyzed for dissolved oxygen.

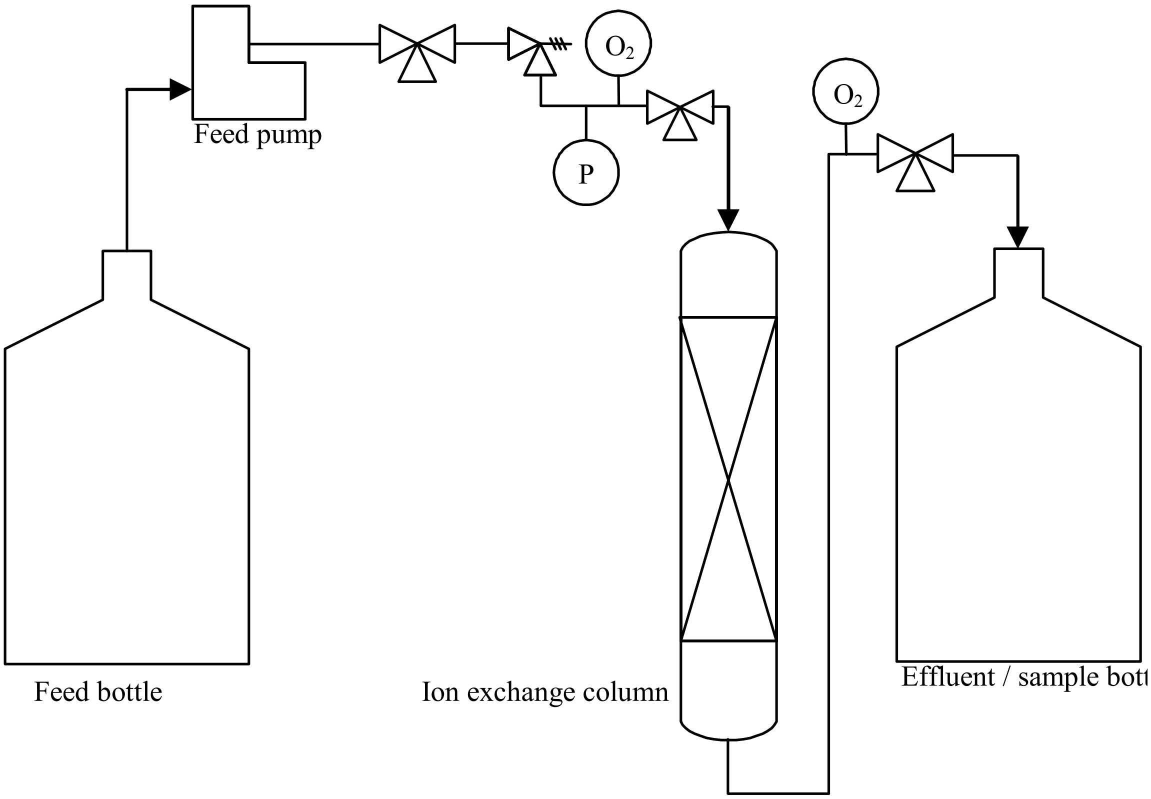

Figure 2.1. SL-644 Chemical Degradation Column Test Process Schematic

The column was a Spectrum Chromatography Spectra/Chrom® column manufactured from glass with plastic plungers on the ends that could be adjusted to control the distance between the top of the resin bed and the column feed. The internal diameter of the column was 1.5 cm.

The pump was a Fluid Metering, Inc. (FMI) piston pump with the flow rate controlled from outside of the fumehood using an FMI stroke-rate controller. The pump was pre-calibrated using water, and it provided 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 was below the maximum operating pressure of the column. Valves 1 and 2, placed between the pump outlet and the column, were used to eliminate air from the system or isolate the column from the pump. Valve 3 was used to prevent the column from draining while the pump was stopped. The equipment and fittings were connected using $^{1}/_{16}$-in. internal-diameter polyethylene tubing.

2.6 Dissolved Oxygen Measurement and Interpretation

The dissolved-oxygen concentration in the feed and effluent streams was monitored with a FOXY fiber-optic oxygen-sensor system manufactured by Ocean Optics Inc. The FOXY probes were inserted into the process stream using Swagelok® tee-piece fittings. They contain a ruthenium complex encapsulated in a sol-gel matrix that is further protected by a silicone overcoat. Oxygen dissolved in the solution to be analyzed diffuses into the sol-gel matrix such that the oxygen in the sol-gel matrix and solution are in dynamic equilibrium. A pulsed blue-light-emitting diode transmits light at ~475 nm through an optical fiber to the probe. The blue light excites the ruthenium complex, which fluoresces, emitting energy at ~600 nm. The fluorescence signal decreases when the excited ruthenium complex encounters an oxygen molecule because the excess energy is transferred. Hence the fluorescence signal strength is directly proportional to the oxygen partial pressure in the sol-gel film. The fluorescence energy is carried back to a spectrometer by an optical fiber. The system was calibrated in air and in a pure nitrogen gas stream before use in each cycle as recommended by the manufacturer.

The FOXY system measures and reports the partial pressure of oxygen at equilibrium with that dissolved in solution. Therefore, Bunsen coefficients were used to determine the concentration of oxygen dissolved in each reagent. The Bunsen coefficient, α (units of m^2/N) , is the equivalent volume of gas at standard temperature and pressure dissolved in a unit volume of solution existing in equilibrium with a pure atmosphere of the gas. By assuming ideal gas and Henry's Law applicability, the dissolved oxygen concentration, $[O_2]$, was calculated from the Bunsen coefficient and the partial pressure, p (units of Nm^{-2}), given by the FOXY system according to the equation

$$[O_2] = \frac{\alpha p}{R}\left(\frac{P}{T}\right)_{STP} \tag{5}$$

where R is the universal gas constant (8.3144 J/mol K), P is the standard pressure (1.013×10^5 Nm^{-2}), and T is the standard temperature (298 K).

Bunsen coefficients were extrapolated for the appropriate reagent concentrations, [R], from data provided by Lange and Zander (1986, pp 778-779) according to Setchenov's law,

$$Log_{10}\left(\frac{\alpha_0}{\alpha}\right) = k_{Sc\alpha}[R] \tag{6}$$

where α_0 is the Bunsen coefficient for water (0.025 atm^{-1}), and $k_{Sc\alpha}$ is the Setchenov constant for the reagent. Values for $k_{Sc\alpha}$ were also available from Lange and Zander (1986, pp 778-779).

Oxygen solubility data for LAW are not available, but were estimated using Young's first mixing rule. For this estimate, the LAW composition was approximated to 2.15 M NaOH and 3.05 M $NaNO_3$, assuming

- the contributions of other constituents to oxygen solubility is negligible
- $NaNO_3$ and $NaNO_2$ are equivalent in their contributions to oxygen solubility since values of α for $NaNO_2$ are not available.

Values of α for 5.2 M NaOH and 5.2 M $NaNO_3$ were derived using Equation 5, and then the Bunsen coefficient for the mixture was derived using Young's first law,

$$\alpha_{LAW} = \left(\frac{2.15}{5.2}\alpha_{NaOH}\right) + \left(\frac{3.05}{5.2}\alpha_{NaNO3}\right) \tag{7}$$

Table 2.7 provides the values of α used in this analysis for each reagent.

Table 2.7. Bunsen Coefficients Derived for the Reagents Used in this Test

Reagent	Bunsen Coefficient ($\times 10^5$ m^2N^{-1})
0.25 M NaOH	0.0225
LAW (5.2M Na)	0.0052
0.1 M NaOH	0.0240
DI water	0.0250
0.5 M HNO_3	0.0243

2.7 Modified Toxicity Characteristic Leach Procedure

A Toxicity Characteristic Leach Procedure (TCLP) was to be performed on the spent resin to provide a preliminary indication of its toxicity. However, the TCLP was modified from the official U.S. Environmental Protection Agency (EPA) SW-846 procedure to accommodate the very small sample of resin available. The deviations from the EPA SW-846 method are outlined below.

The SW-846 method requires that the leach, or extraction, fluid be selected according to the sample acidity. To choose the extraction fluid, according to the procedure, 1 g of sample is stirred with 20 mL of water. If the pH of the water is less than 5, then Extraction Fluid #1 (a mixture of acetic acid and NaOH at a pH of 4.93) is used. Otherwise, 0.5 mL of 1 M HCl is added to the solution, and it is stirred more. Then, if the pH is less than 5, Fluid #1 is used; otherwise, Fluid #2 (an acetic acid solution of pH 2.88) is used. There was insufficient sample to perform this test. However, there was little material adhering to the sample that may have reacted and affected the pH of water or 1 M HCl, and the resin itself was considered inert in 1 M HCl. Therefore, pHs of nearly neutral and <5 in water and 1 M HCl, respectively, were assumed to have been generated and extraction fluid #1 was selected.

Approximately 1 g of sample was available for testing, and this was much less than the 100 g required by the EPA SW-846 procedure. The extraction-fluid volume was proportionately scaled according to the mass of available resin. No duplicate extractions were performed since splitting the small sample would have made the extraction tests impractically small.

The results from the modified TCLP only provide a preliminary assessment of the toxicity characteristic of the spent resin and may not be appropriate for regulatory submissions.

3.0 Column Test Operation and Results

This section defines BV and then describes resin conditioning and the process of cycle operation for 25 cycles.

3.1 Bed Volume Definition

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

3.2 Resin Conditioning

The air-dried, as-received SL-644 resin of mass 4.101 g had a dry BV of 6.8 mL and was converted from the as-received form to the sodium form by contacting it with 1 M NaOH for one hour in a beaker. The 1 M NaOH was de-canted through a filter that captured 0.135 g of resin. The remaining resin was then transferred to the column using DI water to form a bed of volume 11.5 mL. The ion exchange resin was conditioned in the column with 0.5 M HNO_3 and 0.25 M NaOH solutions, before cycle testing, to remove any potassium salts remaining from its manufacture and conversion. Table 3.1 outlines in detail the conditioning operation. A conditioning operation more extensive than that recommended by Hassan et al. (1999) was conducted since prior work by Arm et al. (2002) indicated that it was insufficient to completely wash the K-salts residual from manufacturing from this batch of resin leading to poor breakthrough performance. Hassan et al. (1999) recommended conditioning with 3 BVs of 0.5 M HNO_3 and 6 BVs of 0.25 M NaOH, whereas 49 BVs of 0.5 M HNO_3 and 6.1 BVs of 0.25 M NaOH were used in this work.

Table 3.1. Column Resin-Bed Conditioning Details

	Total Volume of Reagent		Flow Rate of Reagent	
Reagent	**mL**	**BV**	**mL/h**	**BV/h**
1. 0.5 M HNO_3	57.6	5.0	30	2.6
2. DI water	70.9	6.2	30	2.6
3. 0.5 M HNO_3	348	30	10	0.87
4. DI water	60	5.2	30	2.6
5. 0.25 M NaOH	70	6.1	10	0.84
6. DI water	55	4.8	30	2.6
7. 0.5 M HNO_3	156	14	10	0.87
8. DI water	60	5.2	30	2.6

A total apparatus volume (AV) of 23 mL was measured at Steps 1 and 2 by monitoring the pH of the effluent and the volume of collected effluent. The resin bed was observed to pull away from the walls of the column during the nitric acid contact described by Step 7. The bed was stirred with a glass rod between the last two operations, and the bed settled across the full diameter of the column such that the bed height reduced from 61 mm to 46 mm.

3.3 Cycle Operation

3.3.1 Test Schedule

Table 3.2 presents the test schedule. The schedule shows 25 cycles with cycles performed on nearly consecutive weeks with breaks for holidays. Every fifth cycle was performed under baseline conditions while different LAW feed compositions and flow rates were investigated in the intermediate cycles according to a partial factorial experimental design. The test variables studied were Cs^+, K^+, and OH^- concentrations and a simulated LAW feed rate. The logic behind the values of the composition variables is described in Section 2.1.1. The baseline LAW feed rate of 3 BV/h was selected since it is the WTP design value for processing LAW of Envelopes A and C provided by Olson (2001). The value of 1.5 BV/h was selected as the lower rate since it is the WTP design value for processing LAW from Envelope B. The maximum value of 6 BV/h was selected with WTP personnel to provide a reasonable range of flow rates expected in the WTP.

Table 3.2. SL-644 Chemical Degradation Test Schedule

		Nominal Concentrations (M)			LAW Feed Rate (BV/h)	
Cycle	**Operation Dates**	**Cs^+**	**K^+**	**OH^-**		**Comments**
1	8/20/01 – 8/23/01	1.22E-4	0.095	1.7	3	Baseline
2	8/27/01 – 8/30/01	6.40E-5	0.43	4.32	3	Simulated AW-101 LAW
3	9/3/01 – 9/6/01	6.09E-5	0.095	1.7	3	None
4	9/17/01 – 9/20/01	9.38E-5	0.095	1.7	3	None
5	9/24/01 – 9/27/01	1.22E-4	0.095	1.7	3	Baseline
6	10/1/01 – 10/4/01	2.35E-4	0.095	1.7	3	None
7	10/8/01 – 10/12/01	3.48E-4	0.095	1.7	3	None
8	10/15/01 – 10/18/01	4.61E-4	0.095	1.7	3	None
9	10/22/01 – 10/25/01	5.64E-4	0.095	1.7	3	None
10	10/29/01 – 11/1/01	1.22E-4	0.095	1.7	3	Baseline
11	11/5/01 – 11/8/01	2.35E-4	0.008	2.2	6	None
12	11/12/01 – 11/15/01	6.11E-5	0.008	2.2	1.5	None
13	11/26/01 – 11/29/01	2.35E-4	0.008	2.2	3	None
14	12/3/01 – 12/6/01	2.35E-4	0.008	1.2	1.5	Elution performed at 2 BV/h
15	12/10/01 – 12/13/01	1.22E-4	0.095	1.7	3	Baseline
16	1/7/02 – 1/10/02	6.11E-5	0.008	1.2	6	None
17	1/14/02 – 1/17/02	2.35E-4	0.008	1.2	3	None
18	1/21/02 – 1/24/02	2.35E-4	0.8	2.2	1.5	None
19	1/28/02 – 1/31/02	6.11E-5	0.8	2.2	6	None
20	2/4/02 – 2/7/02	1.22E-4	0.095	1.7	3	Baseline
21	2/11/02 – 2/14/02	6.11E-5	0.8	2.2	3	None
22	2/18/02 – 2/21/02	2.35E-4	0.8	1.2	6	None
23	2/25/02 – 2/28/02	6.11E-5	0.8	1.2	1.5	None
24	3/4/02 – 3/7/02	6.11E-5	0.8	1.2	3	None
25	3/11/02 – 3/14/02	1.22E-4	0.095	1.7	3	Baseline

3.3.2 Cycle Operation Overview

A cycle test commenced with converting the resin to the sodium form by pumping the equivalent of nominally 6 BVs of 0.25 M NaOH through the bed. The simulated LAW was then processed, followed by column rinses of nominally the equivalent of two AVs, each of 0.1 M NaOH solution and DI water before the resin was eluted with up to 20 BVs of 0.5 M HNO_3 solution. A Cs^+ concentration in the final

BV of eluate of 1% of that in the simulated LAW feed was targeted. The cycle finished with a rinse of nominally 2 AVs of DI water.

Cycles 1, 5, 10, 15, 20, and 25 were performed with simulated LAW of baseline composition and baseline flow rates to be able to monitor resin performance at consistent conditions. Other cycles were used for the parametric study. The contact time of resin with each reagent was maintained constant for all cycles since exposure time was considered an important variable on chemical degradation characteristics.

Simulated LAW effluent samples were periodically collected by directing the flow into 20-mL vials to collect nominally 5 mL of sample. These samples were then analyzed by GEA for their ^{137}Cs content. The bed height and effluent bottle mass were measured during sampling events. Samples of the simulated LAW feed and effluent of ~20-mL volume were also collected for potential semi-volatile and volatile organic analysis (SVOA/VOA) in Cycles 1, 5, 10, 15, 20, and 25. Note that BNI determined during the course of this work that SVOA and VOA were to be performed on samples collected in another task.

For most cycles, eluate was collected in a single bottle and then the final 5 mL was collected separately for GEA to determine the ^{137}Cs content. In order to ascertain the elution profile, eluate was collected during Cycles 1, 2, 5, 10, 15, 20, and 25 into 20-mL vials that were changed every hour to provide 10-mL fractions. Appropriately sized aliquots were then extracted from each vial for GEA. Eluant and eluate samples of ~20-mL volume were also collected for potential SVOA/VOA in Cycles 1, 5, 10, 15, 20, and 25.

Activity-balance integrity was assessed on Cycles 1, 2, 5, 10, 15, 20, and 25 by performing GEA on 5-mL samples of all effluent composites except regeneration. The oxygen concentration in the feed and effluent was measured every 10 or 15 minutes and automatically logged on a personal computer.

Appendix A contains the operational data for each cycle.

3.3.3 Operational Details for Cycle 1

Table 3.3 provides the details regarding the operation of the first cycle.

Table 3.3. Cycle 1 Process Operation Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	11.9	55	4.7	9.2	0.8
Waste processing	Batch 1 simulated AN-105 LAW	11.0	1500	126	30	2.5
Feed displacement	0.1 M NaOH	Not measured	44	3.7	29	2.5
Rinse	DI water	Not measured	44	3.7	29	2.5
Elution	0.5 M HNO_3	Not measured	192	16	9.9	0.8
Rinse	DI water	9.6	81	6.8	29	2.4

The effluent from regeneration was discolored a deep red-brown. Figure 3.1 presents the Cs^+-breakthrough profile for Cycle 1. Cesium breakthrough is defined as the Cs^+ concentration in the effluent as a fraction of the Cs^+ concentration in the feed. Probability and linear scales are used for Cs^+ breakthrough and effluent volume, respectively, to facilitate analysis since the profile on such a plot is linear for ideal ion exchange performance. The breakthrough profile is approximately linear past 20 BV with ~5% and then ~50% breakthrough after ~8 BVs and ~120 BVs, respectively.

Breakthrough performance was poorer than expected from the results of Fiskum et al. (2002), although Arm et al. (2002) observed poor performance on the first cycle before improvement in subsequent cycles. The poor performance may be indicative of either incomplete chemical conditioning or bed packing, the latter resulting in the LAW channeling through the bed, despite conditioning the resin more extensively than Arm et al. (2002). Indeed, the top 10 mm of the bed was observed to have pulled away from the column wall during LAW processing and may be responsible for channeling.

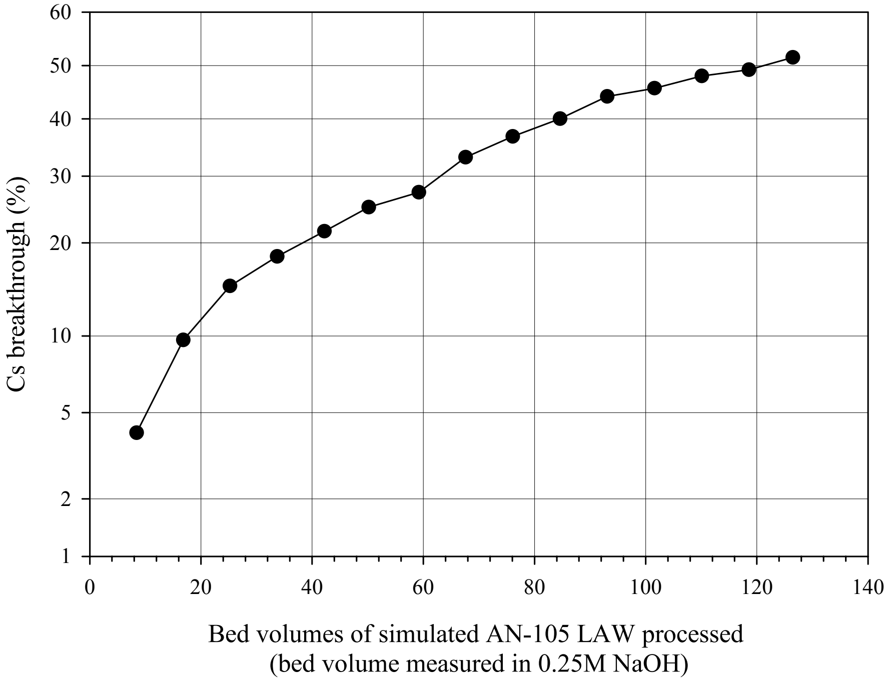

Figure 3.1. Cycle 1 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.5 BV/h, 5.26 M Na, 1.16E-04 M Cs^+, 0.092 M K^+, 1.72 M OH^-, 1.35 M NO_3^-, 1.31 M NO_2^-, ambient conditions, BV = 11.9 mL in 0.25 M NaOH and 11.0 mL in simulated AN-105 LAW)

Figure 3.2 presents the elution profile on logarithmic and linear axes. The Cs^+ concentration in the eluate peaked when ~3 BVs of eluate had been generated and was 1% of that in the simulated LAW feed after ~10.4 BVs of eluate had been generated. Approximately 20 mm of the top of the bed was observed to have pulled away from the column wall.

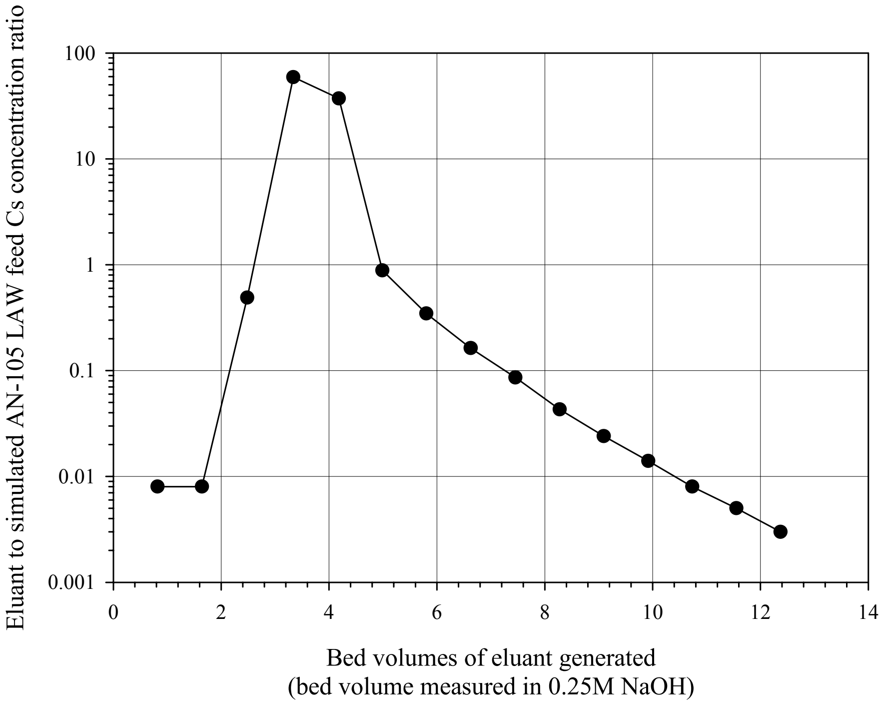

Figure 3.2. Cycle 1 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 0.8 BV/h, Ambient Conditions, BV = 11.9 mL in 0.25 M NaOH)

The activity balance for Cycle 1 is presented in Table 3.4 and shows that approximately 66% of the influent Cs was separated onto the resin and recovered by elution. The balance is considered good with 98.3% of the activity fed to the system accounted for in the effluents.

Table 3.4. Cycle 1 Activity Balance

Process Stream	Total Count Rate (CPM)	Fraction of Feed (%)
Simulated LAW Feed	1.05E6	100
Simulated LAW effluent	3.17E5	31
Feed displacement effluent	1.13E4	1.1
Rinse effluent	3.04E2	0.0
Elution effluent	6.94E5	66
Rinse effluent	5.97E1	0.0
Total recovery of feed ^{137}Cs in effluents	1.03E6	98

Figure 3.3 presents the dissolved-oxygen-concentration profiles. The probe on the feed line failed early in the test and gave spuriously high results. Probe failure was found later to be due to breakdown of the silicone overcoat and chemical attack of the detection system. The feed reagents were, therefore, assumed to be saturated with oxygen to facilitate analysis. This assumption is considered good since later measurements with a new probe and, albeit new, reagents showed them to be saturated with oxygen. The profiles show that oxygen was removed from each reagent at constant rates for each operation except during elution when the oxygen consumption rate appeared to increase.

Table 3.5 presents the oxygen-consumption rate and the total quantity removed for each operation of Cycle 1. The oxygen-consumption rate generally increased with increasing dissolved oxygen-feed concentration. The greatest proportion (57%) of the total quantity of oxygen removed during all operations occurred during elution.

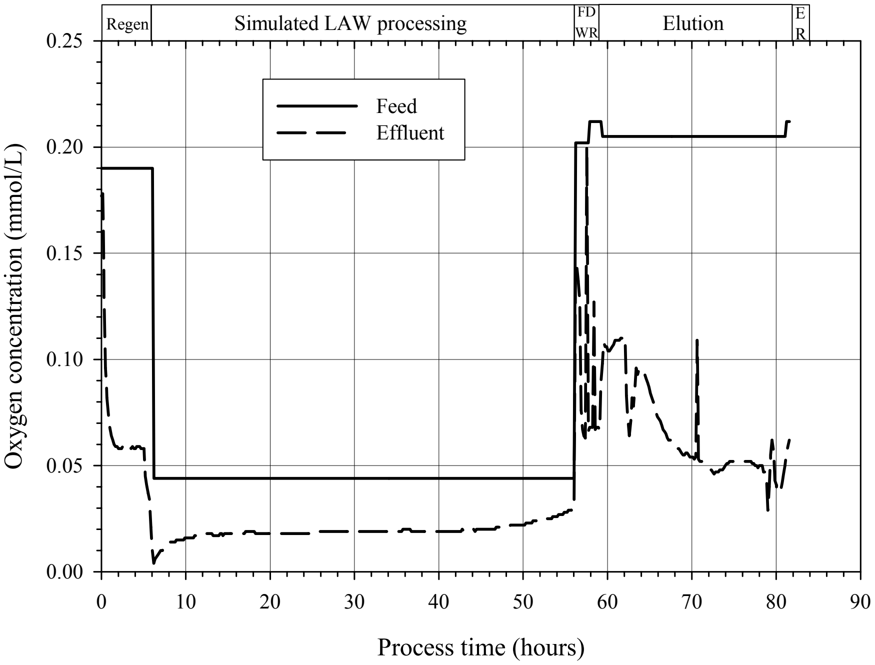

Figure 3.3. Cycle 1 Dissolved-Oxygen-Concentration Profiles (Regen – regeneration, FD – 0.1 M NaOH feed displacement, WR – water rinse, ER – elution rinse)

Table 3.5. Dissolved-Oxygen-Consumption Characterization in Cycle 1

Operation	Oxygen-Consumption Rate (μmol/h)	Total Oxygen Removed (μmol)	Fraction of Total Oxygen Removed (%)
Regeneration	3.5	21	13
LAW processing	0.69	35	22
0.1 M NaOH rinse	2.7	4.1	2.5
Water rinse	5.9	8.9	5.5
Elution	4.0	80	50
Water rinse	4.4	12	7.5

Table 3.6 presents the chemical analysis of the composite eluate and simulated LAW feed. A total of 8.07 meq of metal was eluted from the resin. Significant fractions (>10%) of the feed TC metals Ba, Cd and Pb as well as Fe and U were separated onto the resin and eluted in addition to Cs. Note that Fe was presumably a contaminant of the reagents used to prepare the simulated LAW.

Table 3.6. Chemical Analysis of LAW Feed and Composite Eluate from Cycle 1

Analyte	Analysis Method	Concentration (mg/L)(1,10)		Total mass (mg)		Fraction Separated (%)(3)
		LAW feed	Eluate	LAW feed	Eluate	
Ag	ICP-AES	<0.625	0.349	<0.938	0.067	>7
Al	ICP-AES	15,900	15.8	23,850	3.0	0.01
As	ICP-AES	*54*	<0.25	*81*	<0.05	<0.06
B	ICP-AES	110(8)	10.4	165	2.0	1.0
Ba	ICP-AES	*0.40*	1.35	*0.60*	0.26	40
Ca	ICP-AES	<6.25	3.77	<9.38	0.72	>8
Cd	ICP-AES	*2.2*	2.75	*3.3*	0.53	16
Cl	IC	4,760	<13	7,140	<2.5	<0.04
Cr	ICP-AES	1,530	29.8	2,300	5.7	0.2
Cs	ICP-MS	15.4	80.0(4)	23.1	15.3	66
F	IC	1,160	<13	1,740	<2.5	<0.1
Fe	ICP-AES	*3.0*	8.67	*4.5*	1.7	38
K	ICP-AES	3,610	89.1	5,420	17.1	0.3
Mg	ICP-AES	<2.5	1.27	<3.8	0.24	>6
Mo	ICP-AES	41.6	<0.05	62.4	<0.001	<0.01
Na	ICP-AES	121,000(11)	1,190(9)	181,500	228	(5)
P	ICP-AES	152	*0.17*	228	*0.03*	0.01
Pb	ICP-AES	87.7	67.6	132	13.0	10
Se	ICP-AES	*51*	<0.25	*77*	<0.05	<0.06
Si	ICP-AES	780(8)	27.8	1170	5.34	0.5
U	ICP-AES	*110*	250	*170*	48	28
Zn	ICP-AES	*5.3*	*0.27*	*8.0*	*0.05*	0.6
C_2O_4	IC	<500	<25	<750	<4.8	<0.64
NO_2^-	IC	62,200	<25	93,300	<4.8	<0.01
NO_3^-	IC	81,200	28,100	121,800	5,380	(6)
PO_4^-	IC	<500	<25	<750	<4.8	<0.64
SO_4^-	IC	<500	<25	<750	<4.8	<0.64
TIC	HP	1,390(12)	<10	2,090	<1.9	<0.09
Total Carbon	Furnace	2,600	(7)	3,900	Indeterminate	Indeterminate
TOC	HP	<500	<40	<750	<7.7	<19
	Furnace	1,120	(7)	1,680	Indeterminate	Indeterminate
	Furnace Total Carbon – HP TIC(2)	1,210	Indeterminate	1,820	Indeterminate	Indeterminate

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus, the best TOC result may be the difference between these measurements.
3. Assumed to be fraction of the feed constituent appearing in the eluate.
4. Value is the product of the feed concentration and fraction of ^{137}Cs determined in the eluate by GEA.
5. Not applicable since resin in the Na form.
6. Not applicable since eluate is 0.5 M HNO_3.
7. Not measured due to insufficient sample.
8. Si and B analysis compromised by error in sample preparation manifesting in poor recoveries from spike samples.
9. Relative % difference of 4.0% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
10. Reported results satisfy the WTP project QC criteria unless otherwise noted.
11. Relative % difference of 7.3% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
12. Recovery of 69% from the matrix spike does not satisfy the QC acceptance criterion of >75%. Value may be under-estimated.

3.3.4 Operational Details for Cycle 2

Table 3.7 details the operation of the second cycle with simulated AW-101 LAW. The effluent from regeneration was again discolored a deep red-brown. An off-white precipitate was observed on the top of the bed during LAW processing, but this disappeared during elution. In addition, the simulated LAW feed appeared cloudy during the final 30 min of processing. The precipitate appeared to have been removed from the column during elution since the third eluate fraction (corresponding to the passage of a complete AV) contained solid material, which dissolved shortly after collection. This eluate fraction was also pale yellow whereas other fractions were colorless. The top 15 mm of the resin bed was observed to have pulled away from the column wall during elution only. The oxygen probe on the effluent line failed early in this test, and results are not reported.

Table 3.7. Cycle 2 Process Operation Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	11.7	66	5.6	9.4	0.8
Waste processing	Simulated AW-101 LAW	10.6	1720	147	29	2.5
Feed displacement	0.1 M NaOH	11.0	42	3.6	28	2.4
Rinse	DI water	11.0	42	3.6	28	2.4
Elution	0.5 M HNO_3	9.4	150	13	10	0.9
Rinse	DI water	9.4	46	3.9	31	2.6

The poor result from Cycle 1 caused some doubt as to whether chemical-degradation effects would be discernible in the breakthrough profile. However, whether the relatively poor breakthrough performance was attributable to the resin or to the simulated AN-105 matrix was unclear. Tests reported by Arm et al. (2002) seemed to indicate that the resin had degraded in storage from when tests were performed by Fiskum et al. (2002) shortly after its receipt, and this was manifested in giving poorer breakthrough performance. However, this was uncertain since the tests by Arm et al. (2002) used higher Cs^+ concentrations than those by Fiskum et al. (2002). Therefore, Cycle 2 was performed with simulated AW-101 LAW and a Cs^+ concentration equal to that in the test by Fiskum et al. (2002) to evaluate if the resin had already degraded and to what extent. The results of this test were used to decide if a new batch of resin was required, and the test was re-started so chemical-degradation effects could be discernible in the breakthrough profiles.

The Cycle 2 breakthrough profile presented in Figure 3.4 is sharper than that observed in Cycle 1, although the λ value was approximately the same at ~130 BVs. This result appears to indicate that the resin underwent some degradation during storage since the Cycle-2 breakthrough performance is appreciably poorer than experienced in the tests by Fiskum et al. (2002). However, the Cycle-2 breakthrough profile was similar to that experienced with actual AW-101 LAW tested by Kurath et al. (2000a) and a previous batch of resin. The sharper shape of the simulated AW-101 breakthrough profile provided the confidence that performance degradation would be discernible in subsequent cycles with this resin batch.

The elution profile presented in Figure 3.5 shows a Cs^+ concentration peak centered at ~4 BVs of eluate more broad than observed by Fiskum et al. (2002), Arm et al. (2002), or in Cycle 1 and may be associated with channeling as a result of the resin pulling away from the walls. Despite the broad peak, the target Cs^+ concentration was achieved, reducing to 1% of that in the simulated LAW feed after~11 BVs of eluate had been generated.

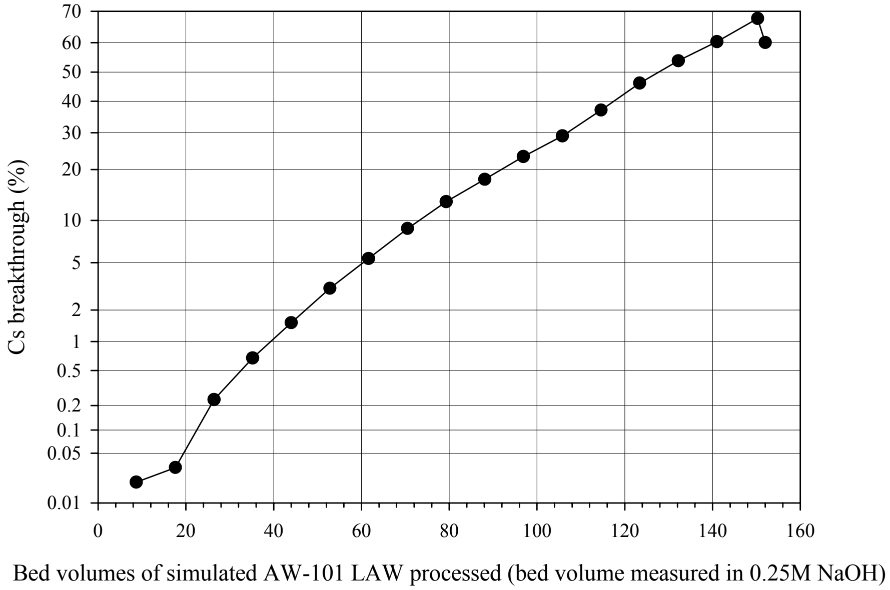

Figure 3.4. Cycle 2 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.6 BV/h, ~5 M Na, 12.2 mM Cs^+, 0.43 M K^+, 3.89 M OH^-, 1.52 M NO_3^-, 0.79 M NO_2^-, ambient conditions, BV = 11.7 mL in 0.25 M NaOH and 10.6 mL in simulated AW-101 LAW)

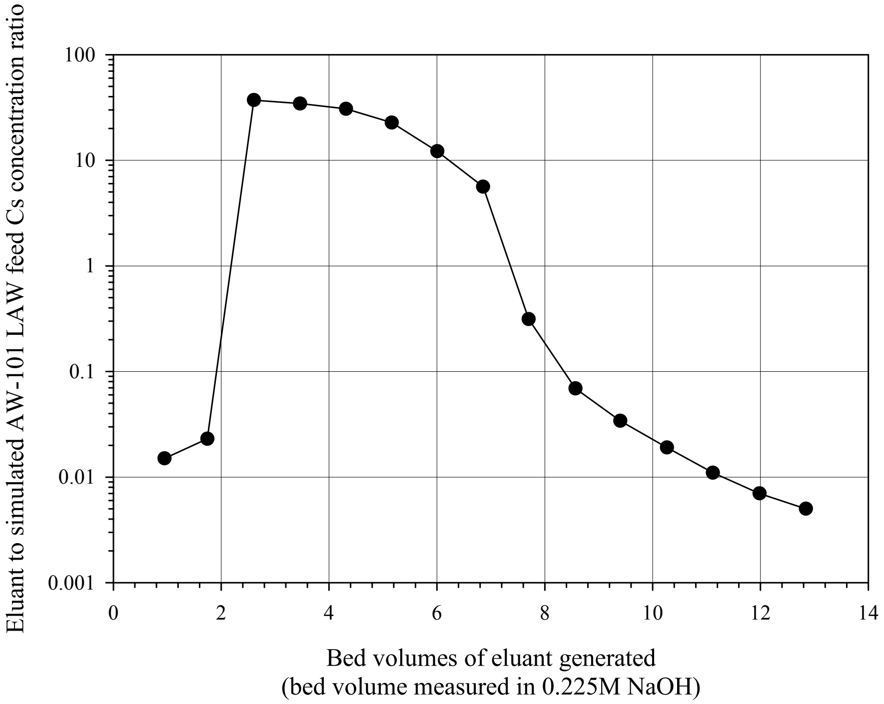

Figure 3.5. Cycle 2 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 0.9 BV/h, ambient conditions, BV = 11.7 mL in 0.25 M NaOH)

The activity balance for Cycle 2 is presented in Table 3.8 and shows that approximately 75% of the influent Cs was separated onto the resin and recovered by elution. The balance is considered good with 92% of the activity fed to the system accounted for in the effluents.

Table 3.8. Cycle 2 Activity Balance

Process Stream	Total Count Rate (CPM)	Fraction of Feed (%)
Simulated LAW Feed	1.06E6	100
Simulated LAW effluent	1.80E5	17
Feed displacement effluent	2.80E3	0.3
Rinse effluent	8.99E2	0.1
Elution effluent	7.95E5	75
Rinse effluent	4.51E1	0.0
Total recovery of feed ^{137}Cs in effluents	9.79E5	92

3.3.5 Operational Details for Cycle 3

The operational details are presented in Table 3.9 for Cycle 3 and indicate that the system was operated without deviating from the plan outlined in Section 3.3.2.

Table 3.9. Cycle 3 Process Operation Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	12.2	60	4.9	9.4	0.8
Waste processing	Batch 2 simulated AN-105 LAW	10.8	1494	122	30	2.5
Feed displacement	0.1 M NaOH	Not measured	45	3.7	30	2.5
Rinse	DI water	Not measured	44.3	3.6	30	2.4
Elution	0.5 M HNO_3	Not measured	142	12	9.4	0.8
Rinse	DI water	Not measured	46.5	3.8	31	2.5

The effluent from regeneration was again discolored a deep red-brown. This test was conducted with simulated AN-105 LAW at the Cs^+ concentration of 6.09E-5 M required for the recipe provided by Eibling and Nash (2001) as the lowest value in determining the concentration giving 50% breakthrough with the LAW processing period. Performance of the resin was good from a separations viewpoint, as shown in Figure 3.6, with breakthrough below 2% upon termination of the simulated LAW feed. The column-distribution coefficient is ~200 based on extrapolation of the last three data points to 50% breakthrough.

The Cs^+ concentration in the last 5 mL of eluate was 0.8% of that in the simulated LAW feed, satisfying the target of 1%.

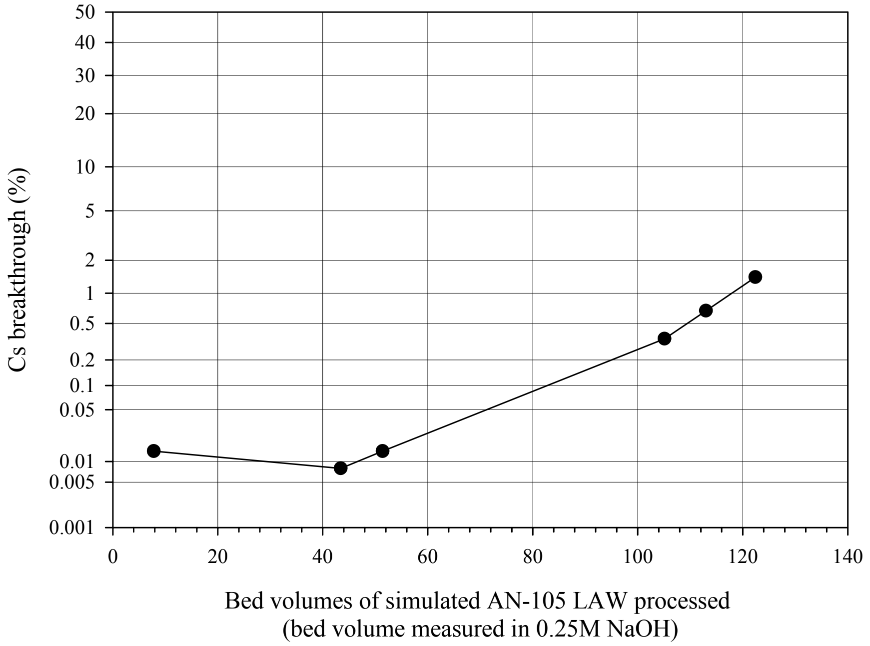

Figure 3.6. Cycle 3 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.5 BV/h, 4.91 M Na, 6.09E-5 M Cs^+, 0.079 M K^+, 1.72 M OH^-, 1.34 M NO_3^-, 1.24 M NO_2^-, ambient conditions, BV = 12.2 mL in 0.25 M NaOH and 10.8 mL in simulated AN-105 LAW)

3.3.6 Operational Details for Cycle 4

The operational details are presented in Table 3.10 for Cycle 4 and indicate that the system was operated as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2. The effluent from regeneration was again discolored a deep red-brown.

Table 3.10. Cycle 4 Operational Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	9.9	61	6.1	10	1.0
Waste processing	Batch 2 simulated AN-105 LAW	9.0	1506	152	30	3.0
Feed displacement	0.1 M NaOH	Not measured	46	4.7	30	3.1
Rinse	DI water	Not measured	43.0	4.3	29	2.9
Elution	0.5 M HNO_3	Not measured	147	15	9.8	1.0
Rinse	DI water	Not measured	46.6	4.7	31	3.1

Following regeneration, 0.325 g of Na-form resin was removed from the column to reduce the BV to 10 mL for consistency with the test-plan requirements. This test was conducted with simulated AN-105 LAW at a Cs^+ concentration of 9.38E-5 M in an effort to achieve 50% breakthrough within the prescribed LAW processing period of 150 h. As shown in Figure 3.7, a breakthrough of ~20% was achieved upon termination of the simulated LAW feed. The column-distribution coefficient is ~190 based on extrapolation of all but the first data point to 50% breakthrough.

The Cs^+ concentration in the last 5 mL of eluate was 1% of that in the simulated LAW feed, matching the target of 1%.

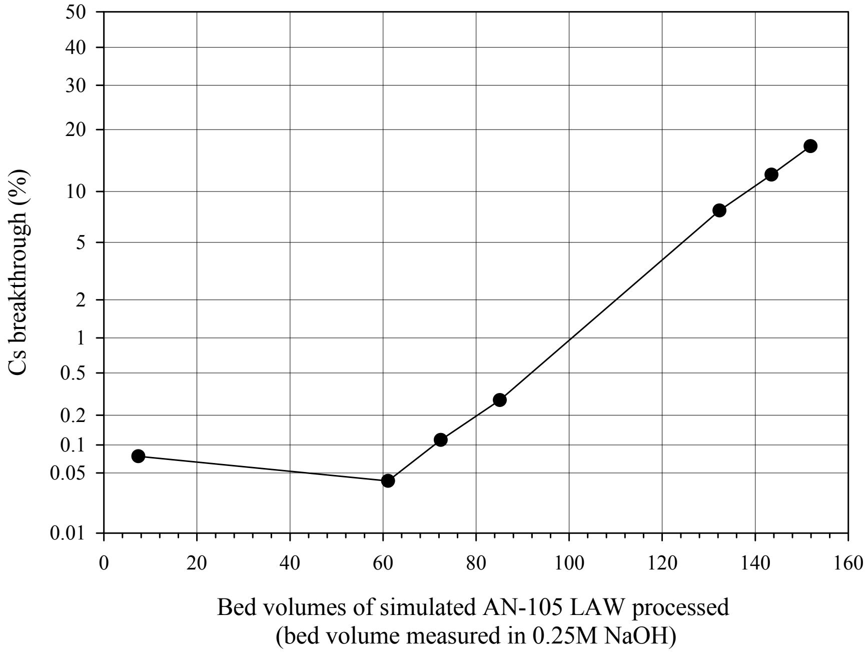

Figure 3.7. Cycle 4 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 3.0 BV/h, 4.91 M Na, 0.938 mM Cs^+, 0.079 M K^+, 1.72 M OH^-, 1.34 M NO_3^-, 1.24 M NO_2^-, ambient conditions, BV = 9.9 mL in 0.25 M NaOH and 9.0 mL in simulated AN-105 LAW)

3.3.7 Operational Details for Cycle 5

The operational details are presented in Table 3.11 for Cycle 5 and indicate that the system was operated as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2. The effluent from regeneration was again discolored a deep red-brown.

Table 3.11. Cycle 5 Operational Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	9.7	59	6.1	9.7	1.0
Waste processing	Batch 2 simulated AN-105 LAW	8.9	1502	155	30	3.1
Feed displacement	0.1 M NaOH	Not measured	43	4.4	29	2.4
Rinse	DI water	Not measured	44	4.5	29	3.0
Elution	0.5 M HNO_3	Not measured	193	20	9.8	1.0
Rinse	DI water	6.7	45	4.6	30	3.1

The simulated AN-105 LAW feed contained Cs^+ at a concentration identical to that used in Cycle 1 at 1.22E-4 M. The breakthrough profile presented in Figure 3.8 shows that resin breakthrough performance had improved over that observed in Cycle 1. For example, 1% breakthrough was not observed until ~85 BVs of feed had been processed in Cycle 5, whereas breakthrough was already 5% after 10 BVs in Cycle 1. The breakthrough profile was essentially linear on the probability scale after 50 BVs and extrapolated to a column distribution coefficient of 150. The concentration of Cs^+ used in this test was therefore confirmed as the baseline value.

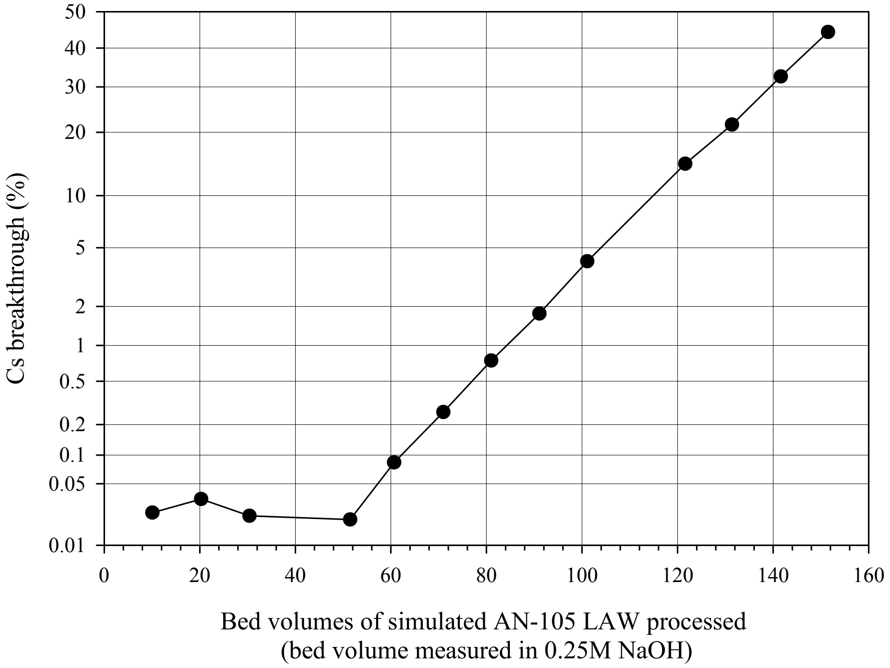

Figure 3.8. Cycle 5 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 3.1 BV/h, 4.91 M Na, 0.122 mM Cs^+, 0.079 M K^+, 1.72 M OH^-, 1.34 M NO_3^-, 1.24 M NO_2^-, ambient conditions, BV = 9.7 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)

Figure 3.9 presents the elution profile. The Cs^+ concentration in the eluate peaked when ~4 BVs of eluate had been generated and was 1% of that in the simulated LAW feed after ~13 BVs of eluate had been generated.

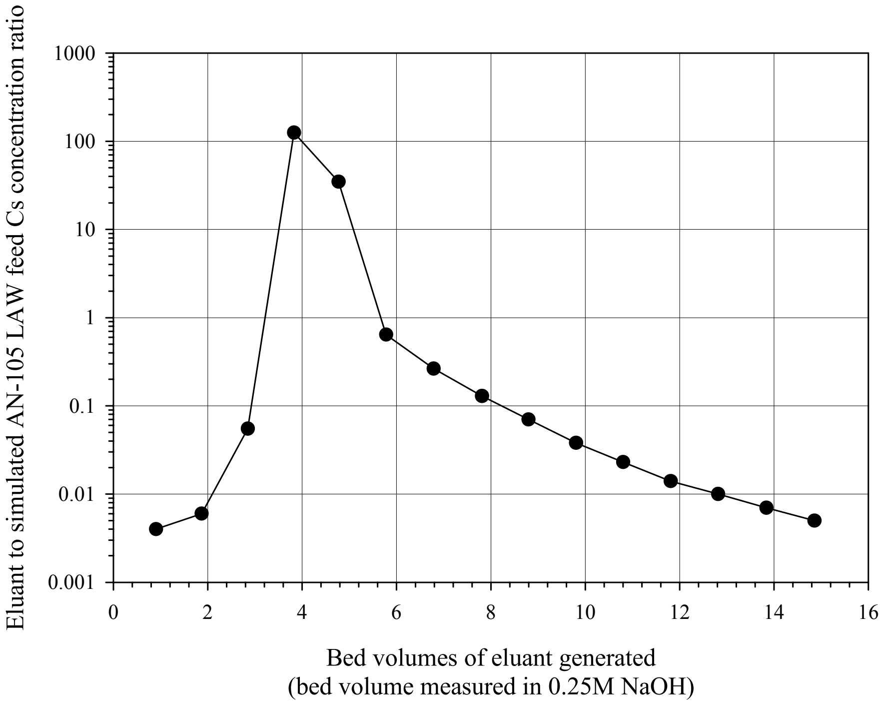

Figure 3.9. Cycle 5 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 1.0 BV/h, ambient conditions, BV = 9.7 mL in 0.25 M NaOH)

The activity balance for Cycle 5 is presented in Table 3.12 and shows that approximately 86% of the influent Cs was separated onto the resin and recovered by elution. The balance was considered good with 90% of the activity fed to the system accounted for in the effluents.

Table 3.12. Cycle 5 Activity Balance

Process Stream	Total Count Rate (CPM)	Fraction of Feed (%)
Simulated LAW Feed	1.00E6	100
Simulated LAW effluent	3.50E4	3.5
Feed displacement effluent	9.29E3	0.9
Rinse effluent	4.05E2	0.0
Elution effluent	8.55E5	86
Rinse effluent	3.28E1	0.0
Total recovery of feed ^{137}Cs in effluents	9.01E5	90

Table 3.13 presents the results of the composite simulated LAW effluent and eluate chemical analyses. There were 6.54 meq of metal in the eluate. As in cycle 1, significant fractions (>10%) of the feed TC metals Ba, Cd and Pb but also Ag as well as Fe and U were separated onto the resin and eluted in addition to Cs. Note again that Fe was presumably a contaminant of the reagents used to prepare the simulated LAW.

Table 3.13. Chemical Analysis of Composite Simulated LAW Effluent and Eluate from Cycle 5

		Concentration (mg/L)$^{(1,9)}$			Total Mass (mg)			Fraction
Analyte	**Analysis Method**	**LAW Feed**	**LAW Effluent**	**Eluate**	**LAW Feed**	**LAW Effluent**	**Eluate**	**Separated (%)** $^{(3)}$
Ag	ICP-AES	<0.63	<0.63	0.807	<0.94	<0.94	0.156	>17
Al	ICP-AES	15,600	15,600	16.4	23,430	23,430	3.17	0.01
As	ICP-AES	64.2	*60*	<0.25	96.4	*90*	<0.05	<0.05
B	ICP-AES	82	39.8	10.8	123	59.8	2.08	2.0
Ba	ICP-AES	*0.39*	*0.43*	3.20	*0.59*	*0.65*	0.618	100
Ca	ICP-AES	<6.3	<6.3	6.18	<9.4	<9.4	1.19	>13
Cd	ICP-AES	*1.3*	*0.97*	1.34	*2.0*	*1.5*	0.259	13
Cl	IC	6,300	5,700	<13	9,500	8,600	<2.5	<0.03
Cr	ICP-AES	1,470	1,430	6.37	2,210	2,150	1.23	0.06
Cs	ICP-MS	16.3$^{(4)}$	2.33$^{(5)}$	109 $^{(5)}$	24.5	3.5	21.0	86
F	IC	1000	1000	<13	1,500	1,500	<2.5	<0.2
Fe	ICP-AES	*2.3*	*0.72*	6.30	*3.5*	*1.08*	1.22	35
K	ICP-AES	3,070	3,030	53.8	4,610	4,550	10.4	0.2
Mg	ICP-AES	<2.5	<2.5	*0.83*	<3.8	<3.8	0.16	>4
Mo	ICP-AES	40.4	39	<0.05	60.7	59	<0.001	<0.01
Na	ICP-AES	113,000$^{(8)}$	111,000$^{(8)}$	951$^{(8)}$	170,000	167,000	184	$^{(6)}$
P	ICP-AES	125	122	*0.17*	188	183	*0.033*	0.02
Pb	ICP-AES	56.7	41.4	49.3	85.2	62.2	9.51	11
Se	ICP-AES	*51*	*49*	<0.25	*77*	*74*	<0.05	0.06
Si	ICP-AES	156	138	14.5	234	207	2.80	1.0
U	ICP-AES	<50	<50	42.7	<75	<75	8.24	>11
Zn	ICP-AES	*5.0*	*4.7*	*0.15*	*7.5*	*7.1*	*0.029*	0.4
C_2O_4	IC	<500	<500	<25	<800	<800	<4.8	<0.6
NO_2^-	IC	56,900	56,200	<25	85,600	84,500	<4.8	<0.01
NO_3^-	IC	82,900	82,300	22,400	125,000	124,000	4,330	$^{(7)}$
PO_4^-	IC	<500	1,400	<25	<800	2,100	<4.8	<0.6
SO_4^-	IC	2,600	2,600	<25	3,900	3,900	<4.8	<0.1
TIC	HP	1,450	1,610	<10	2,180	2,420	<1.9	<0.01
Total Carbon	Furnace	2,420	2,710	34	3,640	4,080	6.6	0.18
TOC	HP	1,120	2,750	<40	1,680	4,140	<7.7	<0.46
	Furnace	45$^{(10)}$	180$^{(10)}$	30	68	270	5.8	9
	Furnace Total Carbon – HP TIC$^{(2)}$	970	1,100	<34, >24$^{(11)}$	1,460	1,650	<6.6, >4.6$^{(11)}$	<0.45, >0.32$^{(11)}$

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus the best TOC result may be the difference between these measurements.
3. Assumed to be the fraction of the feed constituent appearing in the eluate.
4. Nominal value.
5. Value is the product of the feed concentration and fraction of ^{137}Cs determined in the solution by GEA.
6. Not applicable since resin in the Na form.
7. Not applicable since eluate is 0.5 M HNO_3.
8. Relative % difference of 4.0% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
9. Reported results satisfy the WTP project QC criteria unless otherwise noted.
10. TOC recoveries from the caustic matrix spike lower than the QC acceptance criterion makes this result doubtful. See also note 2.
11. Upper and lower bounds given since the HP TIC result was below detection.

3.3.8 Operational Details for Cycles 6 through 9

The operational details are presented in Table 3.14 for Cycles 6 through 9 and indicate that the system was operated as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2. All effluents from regeneration were again discolored a deep red-brown. Cycle-6 processed simulated LAW from Batch 2 while the Batches 7, 8, and 9 processed that from Batch 3.

Cycles 6 through 9 investigated the effect on breakthrough of increasing Cs^+ concentration from 0.235 mM (31.3 mg/L) to 0.564 mM (75.0 mg/L), and Figure 3.10 through Figure 3.13 provide the respective breakthrough profiles. Breakthroughs of 1% were not observed until after processing ~30 BVs of simulated LAW for Cycles 7 through 9 while it was observed after 60 BVs for Cycle 6. The profiles are generally linear up to 90% breakthrough on the probability scale with some scatter thereafter. The column-distribution coefficients (the number of BVs processed at 50% breakthrough) are recorded in Table 3.15 and are between 112 and 58, decreasing with increasing Cs^+ concentration.

Table 3.14. Operational Details for Cycles 6 Through 9

Cycle	Measurement	Unit	Regeneration (0.25 M NaOH)	Simulated AN-105 Processing	Feed displacement (0.1 M NaOH)	Rinse (DI Water)	Elution (0.5 M HNO_3)	Rinse (DI Water)
6	Bed volume	mL	9.7	8.9	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	59	1497	45	44	145	43
		BV	6.1	154	4.6	4.6	15	4.5
	Reagent flow rate	mL/h	9.8	30	30	30	9.3	29
		BV/h	1.0	3.1	3.1	3.0	0.96	3.0
7	Bed volume	mL	10	8.7	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	56	1488	45	44	145	49
		BV	5.6	149	4.5	4.4	15	4.9
	Reagent flow rate	mL/h	9.3	29.9	30	29	9.3	30
		BV/h	0.93	3.0	3.0	2.9	0.93	3.0
8	Bed volume	mL	10.1	8.9	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	58	1492	46	45	143	45
		BV	5.7	148	4.6	4.4	14	4.4
	Reagent flow rate	mL/h	9.7	29.9	31	30	9.5	30
		BV/h	0.96	3.0	3.1	3.0	0.94	2.9
9	Bed volume	mL	10.3	8.9	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	58	1309	45	45	143	44.2
		BV	5.6	128	4.5	4.4	14	4.3
	Reagent flow rate	mL/h	9.7	29.9	30	30	9.6	29
		BV/h	0.94	2.9	3.0	2.9	0.93	2.9

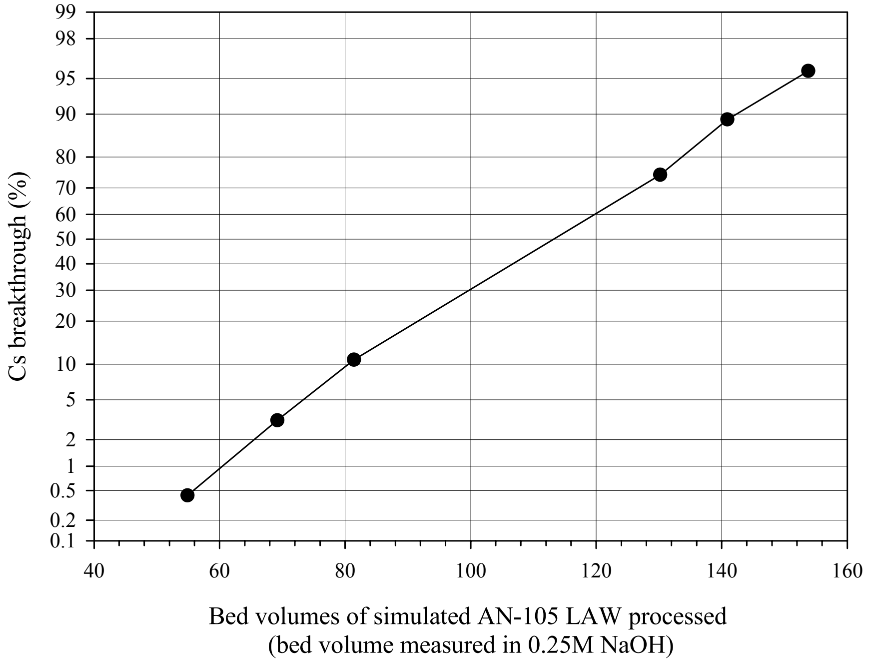

Figure 3.10. **Cycle 6 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 3.1 BV/h, 4.91 M Na, 0.235 mM Cs^+, 79 mM K^+, 1.72 M OH^-, 1.23 M NO_3^-, 1.21 M NO_2^-, ambient conditions, BV = 9.7 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)**

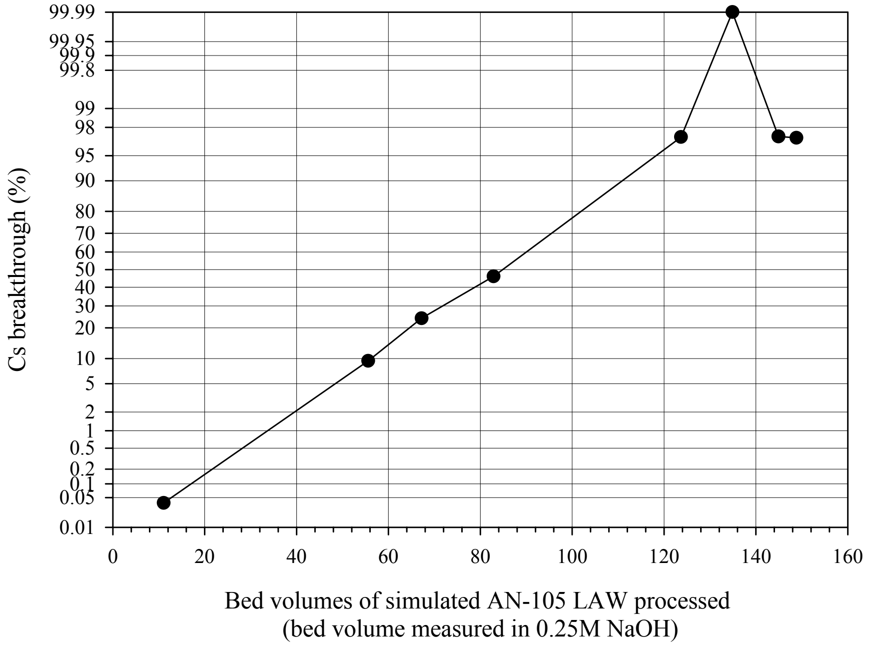

Figure 3.11. **Cycle 7 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 3.0 BV/h, ~5 M Na, 0.348 mM Cs^+, 95.1 mM K^+, 1.72 M OH^-, 1.23 M NO_3^-, 1.21 M NO_2^-, ambient conditions, BV = 10 mL in 0.25 M NaOH and 8.7 mL in simulated AN-105 LAW)**

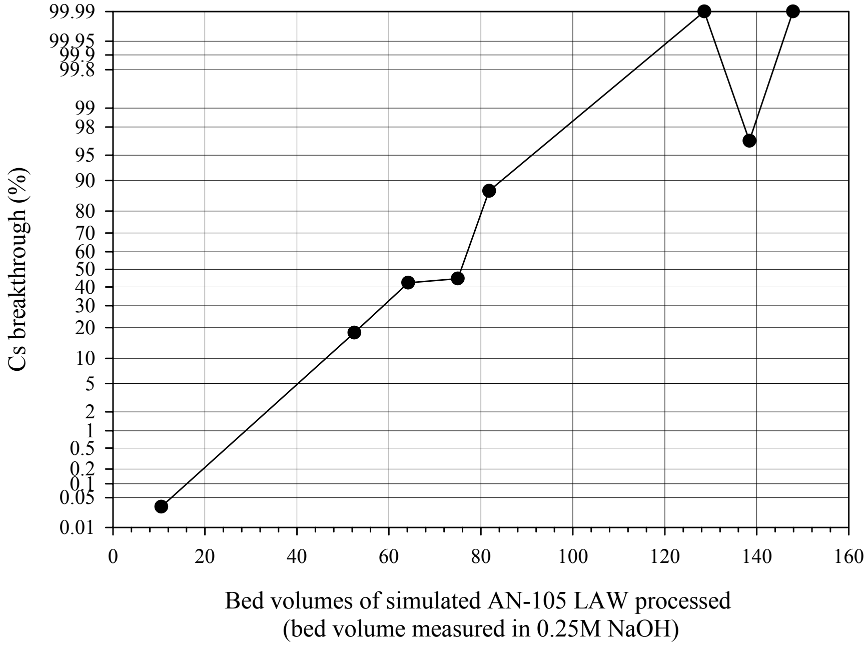

Figure 3.12. **Cycle 8 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 3.0 BV/h, ~5 M Na, 0.461 mM Cs^+, 95.1 mM K^+, 1.72 M OH^-, 1.23 M NO_3^-, 1.21 M NO_2^-, ambient conditions, BV = 10.1 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)**

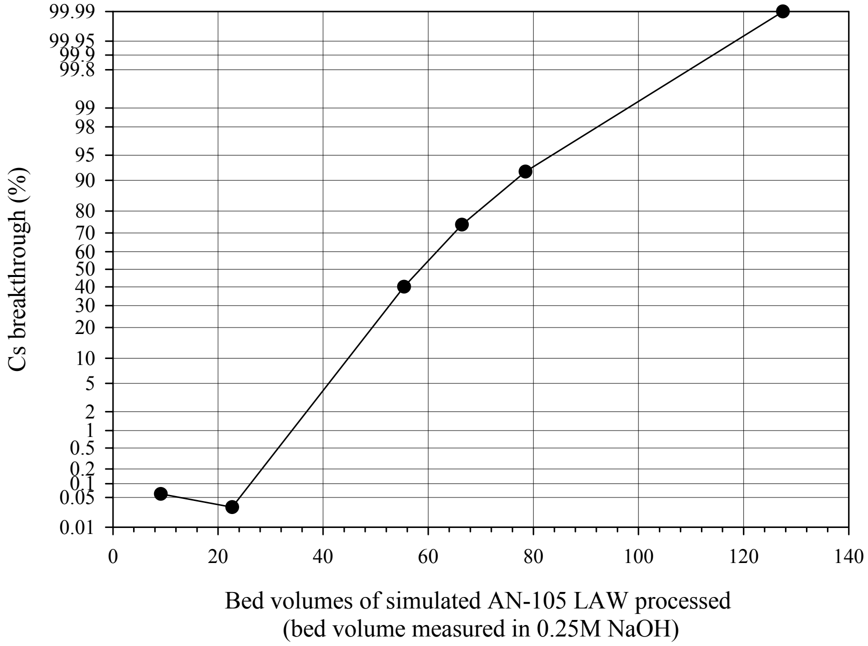

Figure 3.13. **Cycle 9 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.9 BV/h, ~5 M Na, 0.564 mM Cs^+, 95.1 mM K^+, 1.72 M OH^-, 1.23 M NO_3^-, 1.21 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)**

Table 3.15. Column-Distribution Coefficients for Cycles 6 through 9

Cycle	Simulated LAW Batch #	Concentration			Column Distribution Coefficient
		Cs^+ (mM)	K^+ (M)	OH^-(M)	
6	2	0.235	0.0951	1.72	112
7	3	0.348	0.0951	1.72	84
8	3	0.461	0.0951	1.72	68
9	3	0.564	0.0951	1.72	58

Table 3.16 shows that only in Cycle 6 was the target eluate to simulated LAW feed Cs^+ concentration of 0.01 in the last 5 mL of eluate achieved. Contamination of the final 5-mL sample with the bulk eluate is considered responsible for the failure to achieve the target in Cycles 7, 8, and 9 since it was achieved in all cycles where eluate fractions were collected.

Table 3.16. Elution Performance Data for Cycles 6 through 9

Cycle	Eluate to Simulated LAW Feed Cs^+ Concentration Ratio in Last 5 mL of Eluate
6	0.005
7	0.016
8	0.026
9	0.011

3.3.9 Operational Details for Cycle 10

The operational details are presented in Table 3.17 for Cycle 10 and indicate that the system was operated as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2. The effluent from regeneration was again discolored a deep red-brown.

Table 3.17. Cycle 10 Operational Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	10.4	78	7.5	10	1.0
Waste processing	Batch 4 simulated AN-105 LAW	8.9	1471	141	29	2.8
Feed displacement	0.1 M NaOH	Not measured	45	4.3	30	2.9
Rinse	DI water	Not measured	45	4.3	30	2.8
Elution	0.5 M HNO_3	Not measured	202	19	10	1.0
Rinse	DI water	6.2	46	4.4	30	2.9

The simulated AN-105 LAW feed contained Cs^+ at a concentration identical to that used in Cycles 1 and 5 at 1.22E-4 M. The breakthrough profile presented in Figure 3.14 shows that resin-breakthrough performance was still better than in Cycle 1 but had deteriorated over that observed in Cycle 5. For example, 1% breakthrough was observed after ~85 BVs and ~72 BVs of feed had been processed in

Cycles 5 and 10, respectively. The breakthrough profile was essentially linear on the probability scale after 50 BVs and provided a column distribution coefficient of 132.

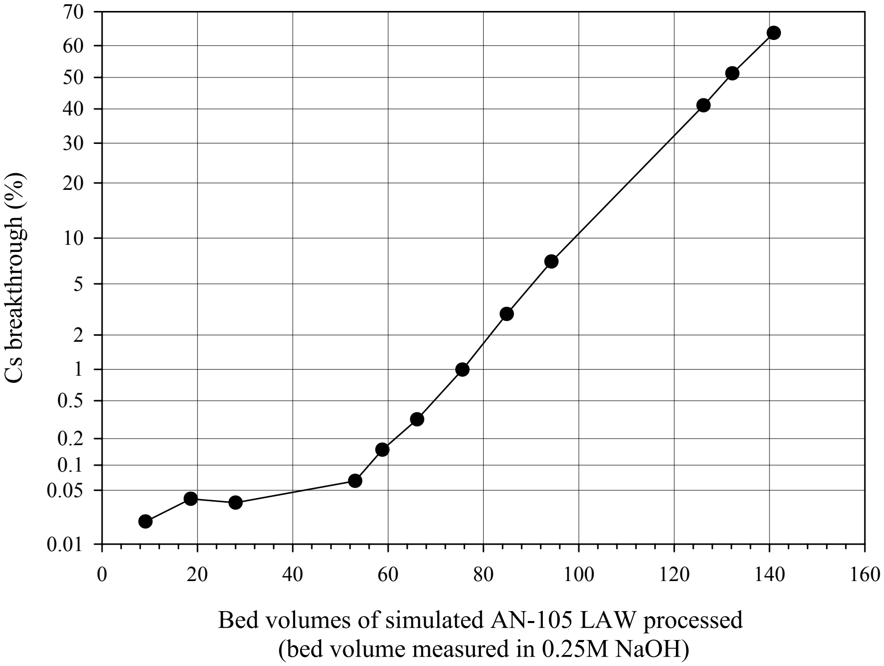

Figure 3.14. Cycle 10 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.8 BV/h, 4.91 M Na, 0.122 mM Cs^+, 79 mM K^+, 1.72 M OH^-, 1.34 M NO_3^-, 1.24 M NO_2^-, ambient conditions, BV = 10.4 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)

Figure 3.15 presents the elution profile. The Cs^+ concentration in the eluate peaked when ~3 BVs of eluate had been generated and was 1% of that in the simulated LAW feed after ~10 BVs of eluate had been generated. The first three eluate fractions were light tan in color, and a “fluffy” brown precipitate was observed to settle from the third fraction to occupy the bottom 3 mm of the sample of total height of 22 mm.

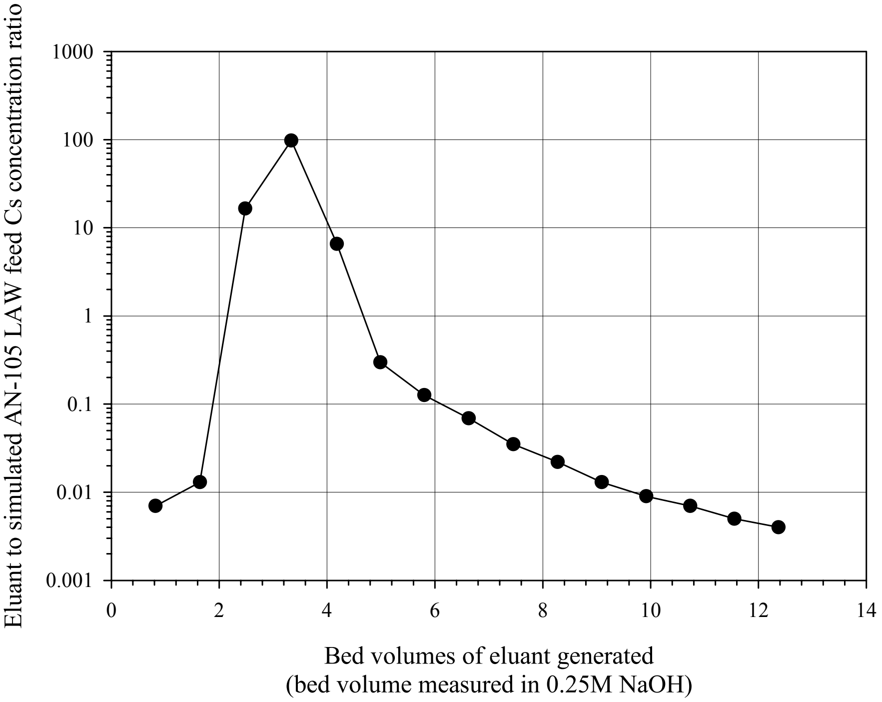

Figure 3.15. Cycle 10 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 1.0 BV/h, ambient conditions, BV = 10.4 mL in 0.25 M NaOH)

The activity balance for Cycle 10 is presented in Table 3.18 and shows that nearly all of the influent Cs^+ was separated onto the resin and recovered by elution. The balance was considered good with 4.1% more of the activity fed to the system accounted for in the effluents.

Table 3.18. Cycle 10 Activity Balance

Process Stream	Total Count Rate (CPM)	Fraction of Feed (%)
Simulated LAW Feed	9.36E5	100
Simulated LAW effluent	2.85E4	3.0
Feed displacement effluent	1.16E4	1.2
Rinse effluent	5.50E2	0.1
Elution effluent	9.34E5	100
Rinse effluent	2.52E1	0.0
Total recovery of feed ^{137}Cs in effluents	9.75E5	104

Table 3.19 presents the results of the composite simulated LAW effluent and eluate chemical analysis. There were 6.52 meq of metal in the eluate. As in previous cycles, significant fractions of the feed Ag, Ba, Fe and U were separated onto the resin and eluted. Note again that Fe was presumably a contaminant of the reagents used to prepare the simulated LAW.

Table 3.19. Chemical Analysis of Composite Simulated LAW Effluent and Eluate from Cycle 10

Analyte	Analysis Method	Concentration (mg/L)(1,9)			Total Mass (mg)			Fraction Separated (%)(3)
		LAW Feed	LAW Effluent	Eluate	LAW feed	LAW Effluent	Eluate	
Ag	ICP-AES	<0.63	<0.63	0.452	<0.919	<0.919	0.091	>10
Al	ICP-AES	16,300	15,400	15.5	24,000	22,700	3.13	0.01
As	ICP-AES	62.9	*60*	<0.25	92.5	*88*	<0.05	<0.05
B	ICP-AES	29.2	39.7	6.65	43.0	58.4	1.34	1.0
Ba	ICP-AES	*0.56*	<0.25	0.925	*0.82*	<0.368	0.187	33
Ca	ICP-AES	<0.63	<6.3	3.20	<0.93	<9.19	0.646	>7
Cd	ICP-AES	*0.91*	*1.1*	0.662	*1.3*	*1.6*	0.134	7
Cl	IC	5,500	5,500	<13	8,100	8,100	<2.6	<0.03
Cr	ICP-AES	1,490	1,420	4.58	2,190	2,090	0.925	0.04
Cs	ICP-MS	16.3(4)	0.07(5)	118 (5)	24.0	0.1	23.9	99.6
F	IC	1,000	1,000	<13	1,500	1,500	<2.6	<0.2
Fe	ICP-AES	*1.6*	*1.5*	3.64	*2.4*	*2.2*	0.735	31
K	ICP-AES	3,190	3,000	46.0	4,690	4,410	9.29	0.2
Mg	ICP-AES	<2.5	<2.5	*0.27*	<3.7	<3.7	0.055	>1
Mo	ICP-AES	40.9	38.6	<0.05	60.2	56.8	<0.010	<0.02
Na	ICP-AES	115,000(8)	110,000(8)	911(8)	169,000	162,000	184	(6)
P	ICP-AES	127	127	*0.14*	187	187	*0.028*	0.02
Pb	ICP-AES	56.3	51.6	38.7	82.8	75.9	7.82	9.4
Se	ICP-AES	*52*	*49*	<0.25	*76*	*72*	<0.05	<0.07
Si	ICP-AES	*120*	*120*	8.83	*180*	*180*	1.78	0.8
U	ICP-AES	<50	<50	35.5	<74	<74	7.17	>10
Zn	ICP-AES	*4.8*	*4.7*	*0.12*	*7.1*	*6.9*	*0.024*	0.3
C_2O_4	IC	<500	<500	<25	<700	<700	<5.0	<0.71
NO_2^-	IC	57,900	56,900	<25	85,200	83,600	<5.0	<0.01
NO_3^-	IC	83,500	81,900	27,100	123,000	120,000	5,470	(7)
PO_4^-	IC	1,300	1,400	<25	1,900	2,100	<5.0	<0.7
SO_4^-	IC	2,600	2,300	<25	3,800	3,400	<5.0	<0.1
TIC	HP	1,570	1,640	<10	2,300	2,410	<2.0	<0.1
Total Carbon	Furnace	2,790	2,680	45	4,100	3,940	9.1	0.22
TOC	HP	1,140	1,070	<40	1,680	1,570	<8.1	<0.48
	Furnace	45(10)	<90(10)	<20	66	<130	<4.0	<6.1
	Furnace Total Carbon – HP TIC(2)	1,220	1,040	<45, >35(11)	1,790	1,530	<9.1, >7.1(11)	<0.51, >0.40(11)

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus the best TOC result may be the difference between these measurements.
3. Assumed to be the fraction of the feed constituent appearing in the eluate.
4. Nominal value.
5. Value is the product of the nominal feed concentration and fraction of ^{137}Cs determined in the solution by GEA.
6. Not applicable because resin is in the Na form.
7. Not applicable because eluate is 0.5 M HNO_3.
8. Relative % difference of 4.0% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
9. Reported results satisfy the WTP project QC criteria unless otherwise noted.
10. TOC recoveries from the caustic matrix spike lower than the QC acceptance criterion makes this result doubtful. See also Note 2.
11. Upper and lower bounds given since the HP TIC result was below detection.

3.3.10 Operational Details for Cycles 11 through 14

Table 3.20 presents the operational details for Cycles 11 through 14. Cycles 11 through 13 used simulated LAW from Batch 5 while Batch 6 simulated LAW was used in Cycle 14. All effluents from regeneration were again discolored a deep red-brown.

Table 3.20. Operational Details for Cycles 11 Through 14

Cycle	Measurement	Unit	Regeneration (0.25 M NaOH)	Simulated AN-105 Processing	Feed Displacement (0.1 M NaOH)	Rinse (DI water)	Elution (0.5 M HNO_3)	Rinse (DI water)
11	Bed volume	mL	10.3	8.6	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	59	1563	57	44	243	150
		BV	5.7	152	5.4	4.3	24	15
	Reagent flow rate	mL/h	9.9	60 / 10(1)	30	29	9.7 / 28(1)	29
		BV/h	0.96	5.9 / 2.9(1)	2.9	2.9	0.94 / 2.7(1)	2.9
12	Bed volume	mL	10.3	8.9	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	59	720	45	44	147	47
		BV	5.7	70	4.3	4.3	14	4.6
	Reagent flow rate	mL/h	9.8	15	30	30	9.8	31
		BV/h	0.95	1.4	2.9	2.9	0.95	3.0
13	Bed volume	mL	10.4	8.7	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	61	1504	46	45	149	46
		BV	5.8	144	4.4	4.3	14	4.4
	Reagent flow rate	mL/h	10	30	31	30	9.9	31
		BV/h	0.97	2.9	3.0	2.9	0.96	2.9
14	Bed volume	mL	10.3	8.9	Not measured	Not measured	Not measured	Not measured
	Reagent volume	mL	60	734	46	51	220	48
		BV	5.8	72	4.5	4.9	21	4.6
	Reagent flow rate	mL/h	9.9	15	30	30	20 / 9.3(2)	32
		BV/h	0.96	1.4	2.9	2.9	1.9 / 0.90(2)	3.1

1. Refer to text for explanation of double values.
2. First 15 BVs of eluant pumped at 20 mL/h (1.9 BV/h) and then the final 6 BVs pumped at 9.3 mL/h (0.90 BV/h).

Large pressure drops in excess of 8 psi were observed during waste processing in Cycle 11, and the flow rate was gradually reduced to 10 mL/h after 20.5 h running at 60 mL/h. The large pressure drop was later found to be due to accumulation of a black precipitate on the membrane covering the feed into the bed chamber of the column.

Five bubbles of diameter ~1 mm were observed approximately a third of the way up from the base of the bed during regeneration in Cycle 13, but these appeared to dissipate before the operation was terminated. However, the bed was fluidized after the final DI water rinse to assure that any air pockets were removed before commencing Cycle 14. Approximately 20 mL of brown cloudy solution were collected from this operation, and ~1 mL of a fine brown powder, presumably resin fines, eventually settled in the collection vial. The mass of fines was later determined to be 0.009 g.

As indicated in Table 3.21, the last 5 mL of the eluate failed to satisfy the target eluate to LAW feed Cs^+ concentration ratio of 0.01. This result in previous cycles had been attributed to contamination of the sample with bulk effluent. However, elution in this cycle may have been incomplete due to channeling of the eluate as a result of any gas bubbles remaining from regeneration. Therefore, a further 85 mL of eluate was pumped at 28 mL/h through the bed, and the last 5 mL were collected for analysis. The Cs^+ concentration in this last sample satisfied the target ratio to that in the LAW feed. The column was then further rinsed with DI water.

Two ~1-mm-diameter bubbles were also observed during waste processing in Cycle 14, again approximately a third of the way up from the base of the resin bed. The bed was not disturbed during this cycle, and the eluate to simulated LAW feed Cs^+ concentration ratio target in the last 5 mL of eluate was achieved, as shown in Table 3.21.

Table 3.21. Elution Performance Data for Cycles 11 Through 14

Cycle	Eluate to Simulated LAW Feed Cs^+ Concentration Ratio in Last 5 mL of Eluate
11	Not measured (sample inadvertently discarded)
12	0.007
13	0.04 / 0.01
14	0.0001

Figure 3.16 through Figure 3.19 present the breakthrough curves for these cycles. Note that smaller volumes of simulated LAW was processed at the lower flow rates to maintain the processing period of 50 hours. The column-distribution coefficients were estimated by extrapolating the profiles, assuming linearity, to 50% breakthrough, although this was not possible for Cycle 12 since breakthrough remained <0.05%. Table 3.22 tabulates the estimated column-distribution coefficients.

Table 3.22. Estimated Column-Distribution Coefficients for Cycles 11 Through 14

Cycle	Simulated LAW Batch #	Concentrations			Flow Rate (BV/h)	Column Distribution Coefficient
		Cs^+ (mM)	K^+ (M)	OH^- (M)		
11	5	0.235	0.008	2.20	5.9	120
12	5	0.066	0.008	2.20	1.4	(1)
13	5	0.235	0.008	2.20	2.9	128
14	6	0.235	0.008	1.20	1.4	100
1. Determination precluded since no significant breakthrough observed.						

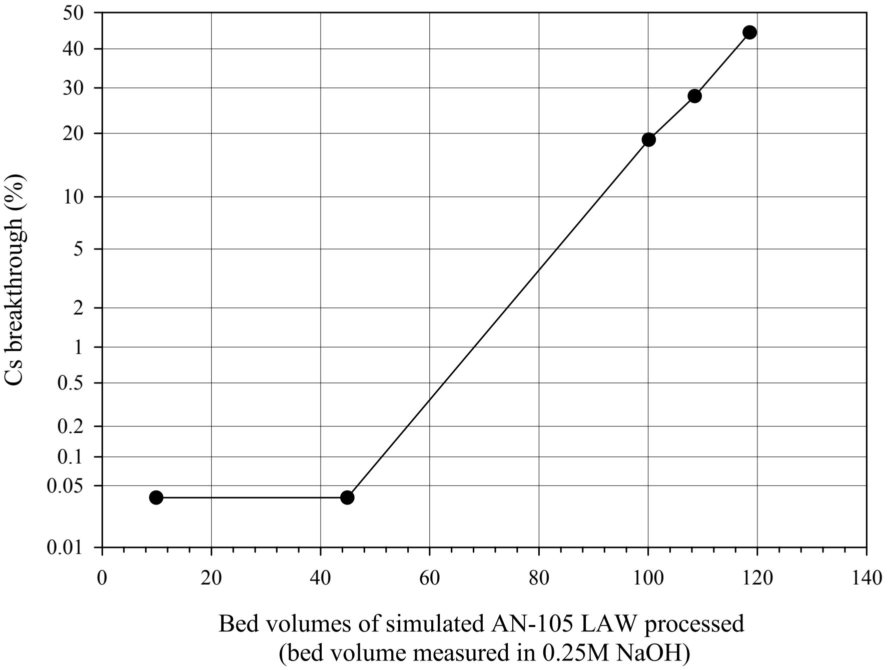

Figure 3.16. Cycle 11 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 5.9 BV/h, ~5 M Na, 0.235 mM Cs^+, 8 mM K^+, 2.2 M OH^-, 0.1 M NO_3^-, 1.46 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.6 mL in simulated AN-105 LAW)

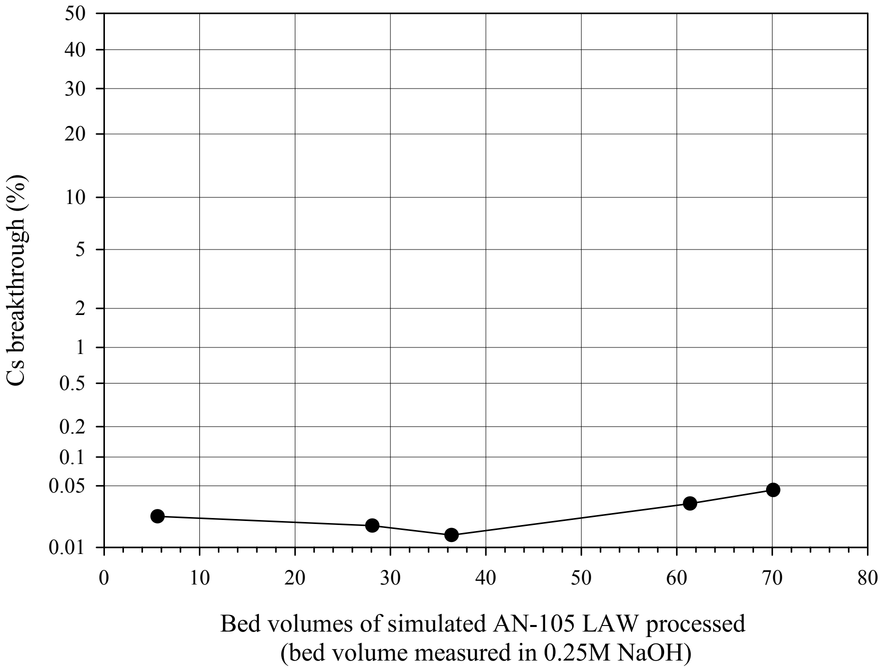

Figure 3.17. **Cycle 12 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 1.4 BV/h, ~5 M Na, 0.066 mM Cs^+, 8 mM K^+, 2.2 M OH^-, 0.1 M NO_3^-, 1.46 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)**

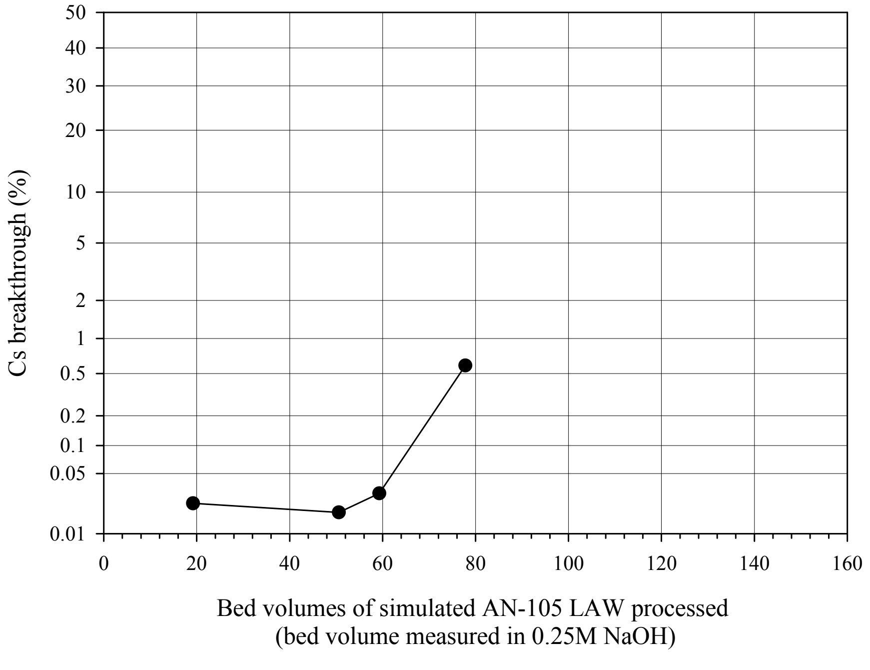

Figure 3.18. **Cycle 13 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.9 BV/h, ~5 M Na, 0.235 mM Cs^+, 8 mM K^+, 2.2 M OH^-, 0.1 M NO_3^-, 1.46 M NO_2^-, ambient conditions, BV = 10.4 mL in 0.25 M NaOH and 8.7 mL in simulated AN-105 LAW)**

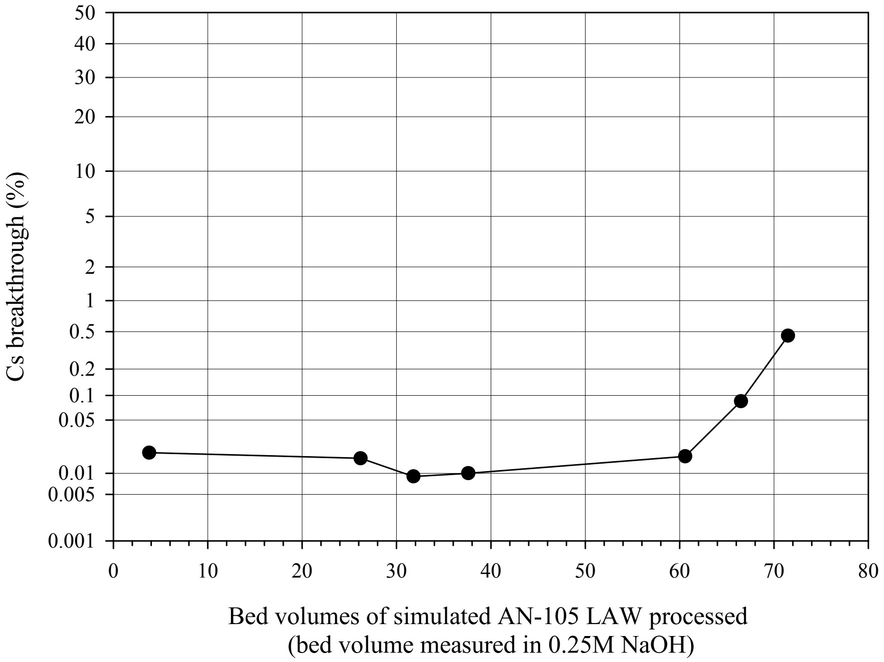

Figure 3.19. Cycle 14 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 1.4 BV/h, ~5 M Na, 0.235 mM Cs^+, 8 mM K^+, 1.2 M OH^-, 0.5 M NO_3^-, 2.46 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)

Half of the elution operation of Cycle 14 was performed at a nominal 2 BV/h, instead of the baseline 1 BV/h, to investigate the impact of flow rate on the elution profile. Only the profile at 2 BV/h is illustrated in Figure 3.20 and shows a peak at ~4 BVs of eluate. It also shows that the target eluate to LAW feed Cs^+ concentration ratio of 0.01 was achieved at 12.4 BV. The remaining half of the processing period was performed at a flow rate 1 BV/h to maintain the normal elution period of 15 h and is not illustrated because no samples were taken.

Dissolved-oxygen-concentration data were collected in Cycles 13 and 14, and the profiles are presented in Figure 3.21 and Figure 3.22, respectively. Note that the probe on the feed line failed during Cycle 14, and the profile was obtained by assuming that all reagents were saturated in oxygen. Comparison of the feed-concentration profiles in Cycles 13 and 14 shows this assumption to be good. Oxygen was removed from all reagents on passage through the ion exchange column in both cycles. There is a spike in the exit concentration during regeneration in both cycles just before commencing the LAW feed, which is possibly associated with the discharge of gas bubbles observed in the bed. The oxygen consumption rate appears to decrease in Cycle 13, but it increases in Cycle 14 during elution.

Table 3.23 presents the oxygen-consumption rates and the total quantities removed for each operation of Cycles 13 and 14. As Cycle 1, the oxygen-consumption rate generally increased with increasing dissolved-oxygen-feed concentration, and the greatest proportion (53%) of the total quantity of oxygen removed during all operations occurred during elution. Consumption rates of 0.42 μmol/h and 1.2 μmol/h were observed for the eluant flow rates of 2 BV/h and 1 BV/h, respectively.

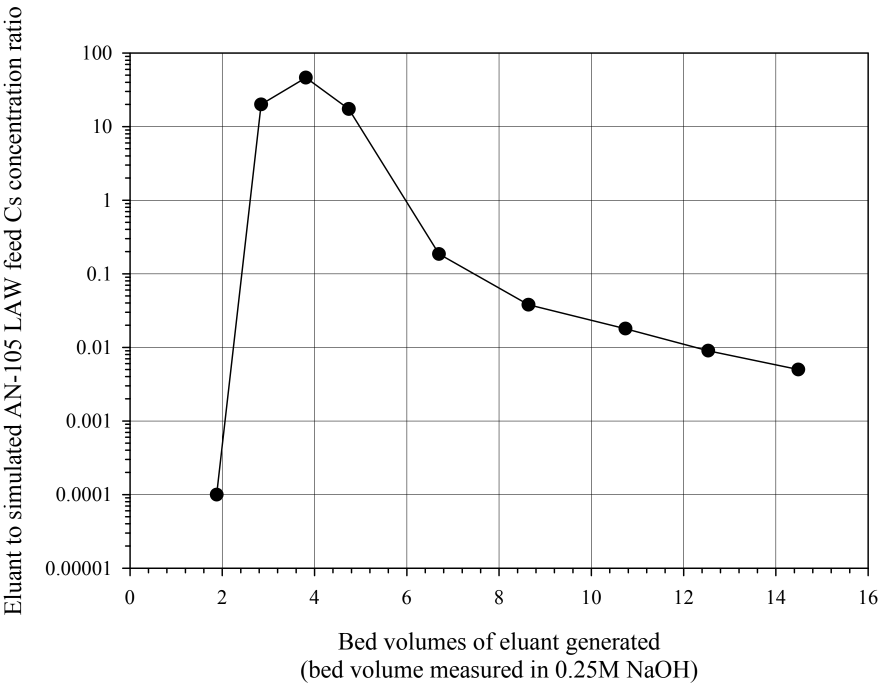

Figure 3.20. Cycle 14 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 1.9 BV/h, ambient conditions, BV = 10.3 mL in 0.25 M NaOH)

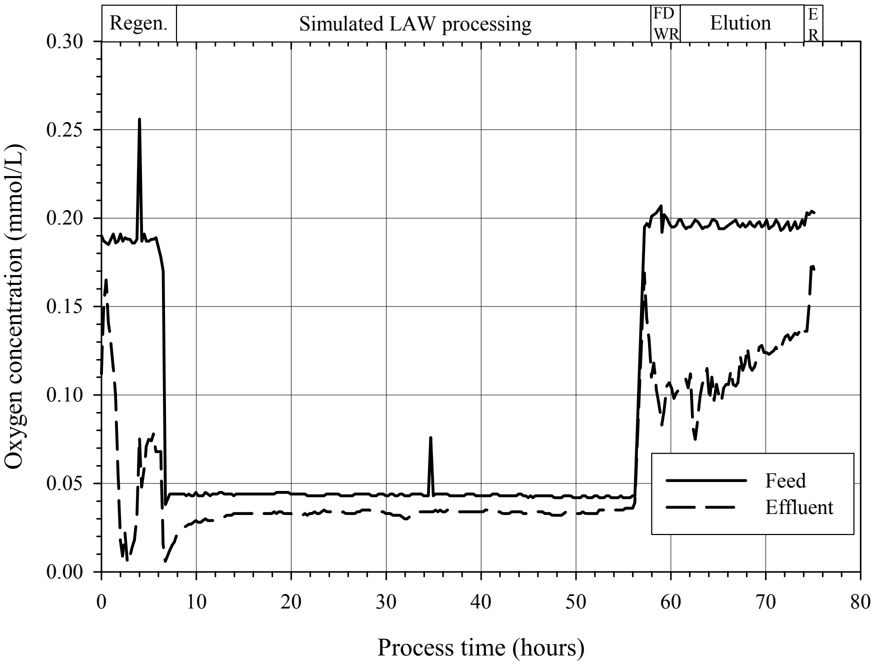

Figure 3.21. Cycle 13 Dissolved-Oxygen-Concentration Profiles (Regen – regeneration, FD – 0.1 M NaOH feed displacement, WR – water rinse, ER – elution rinse)

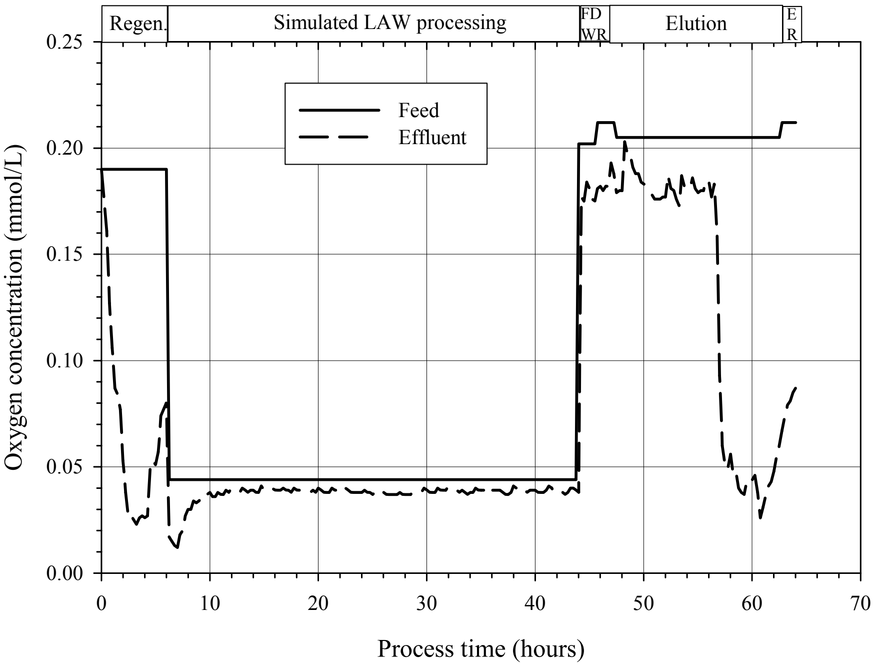

Figure 3.22. Cycle 14 Dissolved-Oxygen-Concentration Profiles (Regen – regeneration, FD – 0.1 M NaOH feed displacement, WR – water rinse, ER – elution rinse)

Table 3.23. Dissolved-Oxygen-Consumption Characterization in Cycles 13 and 14

	Oxygen-Consumption Rate (μmol/h)		**Total Oxygen Removed (μmol)**		**Fraction of Total Oxygen Removed (%)**	
Cycle	13	14	13	14	13	14
Operation						
Regeneration	1.2	1.2	7.2	7.2	8.5	21
LAW Processing	0.36	0.12	18	6.0	21	18
0.1 M NaOH Rinse	1.1	0.71	1.7	1.1	2.0	3.3
Water Rinse	3.0	0.84	4.5	1.3	5.3	3.9
Elution	2.5	0.42 / 1.2	38	12	45	36
Water Rinse	1.3	4.0	2.0	6.0	2.4	18

3.3.11 Operational Details for Cycle 15

Table 3.24 presents the operational details for Cycle 15. The system was essentially operated as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2, except that the cycle had to be temporarily suspended just before terminating the LAW feed. The column was left idle for approximately 40 h in simulated LAW before operations were recommenced. The effluent from regeneration was collected in two fractions. The first fraction was again discolored a deep red-brown while the second fraction was clear but contained fine resin material.

Table 3.24. Cycle 15 Operational Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	10.6	58	5.5	9.7	0.9
Waste processing	Batch 4 simulated AN-105 LAW	8.9	1474	139	30	2.8
Feed displacement	0.1 M NaOH	10.1	44	4.2	30	2.8
Rinse	DI water	10.4	46	4.3	31	2.9
Elution	0.5 M HNO_3	7.1	198	19	10	1.0
Rinse	DI water	7.1	45	4.3	30	2.8

The simulated AN-105 LAW feed contained Cs^+ at a concentration identical to that used in Cycle 10 at 1.22E-4 M. The breakthrough profile presented in Figure 3.23 shows that resin breakthrough performance had deteriorated over that observed in Cycle 10. For example, 1% breakthrough was observed after ~72 BVs and ~46 BVs of feed had been processed in Cycles 10 and 15, respectively. The suspension to operations occurred between the last and penultimate data points and was probably too late in the operation to have a significant impact on the profile. The breakthrough profile has a bow to it on the probability scale, indicating deterioration in the ion exchange kinetics, and provides a column-distribution coefficient of 108.

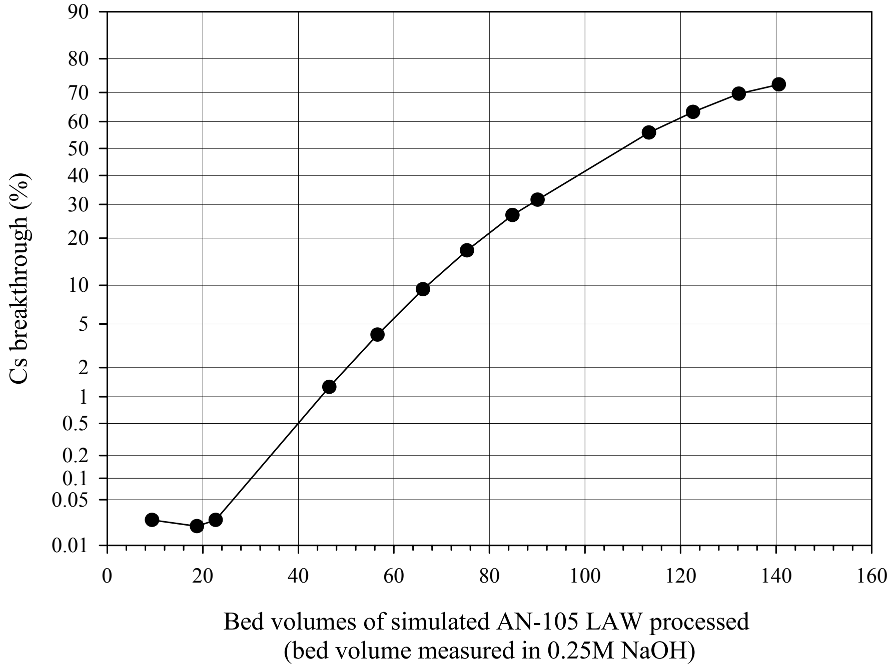

Figure 3.23. Cycle 15 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.8 BV/h, 4.91 M Na, 0.122 mM Cs^+, 7.9 mM K^+, 1.72 M OH^-, 1.34 M NO_3^-, 1.24 M NO_2^-, ambient conditions, BV = 10.6 mL in 0.25 M NaOH and 8.9 mL in simulated AN-105 LAW)

Figure 3.24 presents the elution profile. The Cs^+ concentration in the eluate peaked when ~4 BVs of eluate had been generated and was 1% of that in the simulated LAW feed after ~13.2 BVs of eluate had been generated.

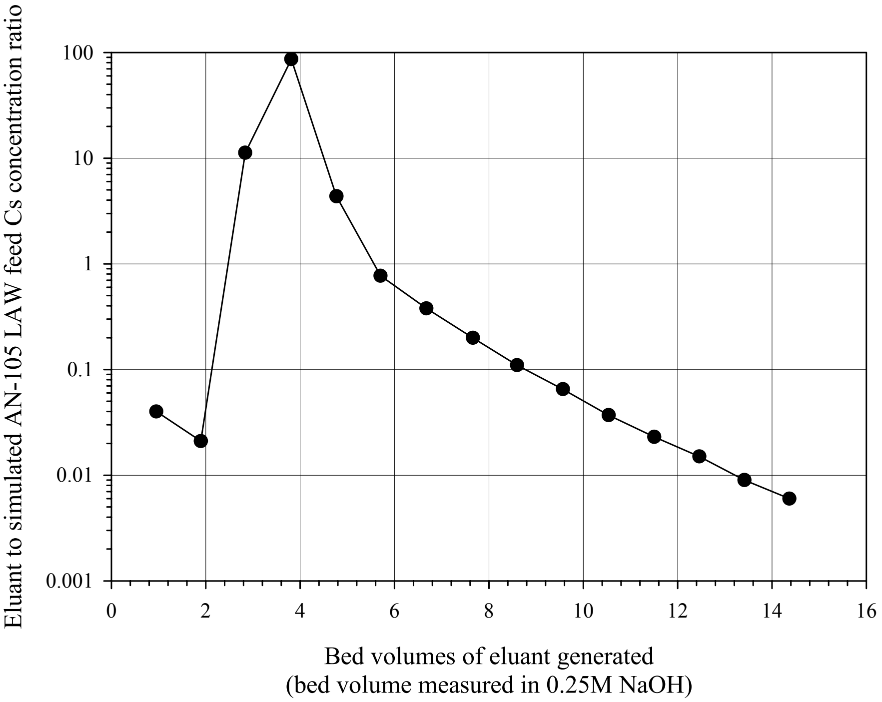

Figure 3.24. Cycle 15 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 1.0 BV/h, ambient conditions, BV = 10.6 mL in 0.25 M NaOH)

The activity balance for Cycle 15 is presented in Table 3.25 and shows that approximately 83% of the influent Cs was separated onto the resin and recovered by elution. The balance is considered good with ~94% recovery of the feed activity accounted for in the effluents.

Table 3.25. Activity Balance for Cycle 15

Process Stream	Total Count Rate (CPM)	Fraction of Feed (%)
Simulated LAW Feed	9.38E5	100
Simulated LAW effluent	7.73E4	8.2
Feed displacement effluent	1.63E4	1.7
Rinse effluent	1.05E3	0.1
Elution effluent	7.83E5	84
Rinse effluent	3.39E1	0.0
Total recovery of feed ^{137}Cs in effluents	8.68E5	94

Figure 3.25 presents the dissolved-oxygen-concentration profiles. The oxygen detection system was shut off when operations were suspended such that its calibration was upset. The data are therefore limited to the regeneration and LAW processing operations.

Table 3.26 presents the oxygen-consumption rate and the total quantity removed for the regeneration and LAW processing operations of Cycle 15. A spike in the effluent concentration during regeneration is again observed just before commencing LAW processing. Also, as observed before, the oxygen-consumption rate increased with increasing dissolved-oxygen-feed concentration.

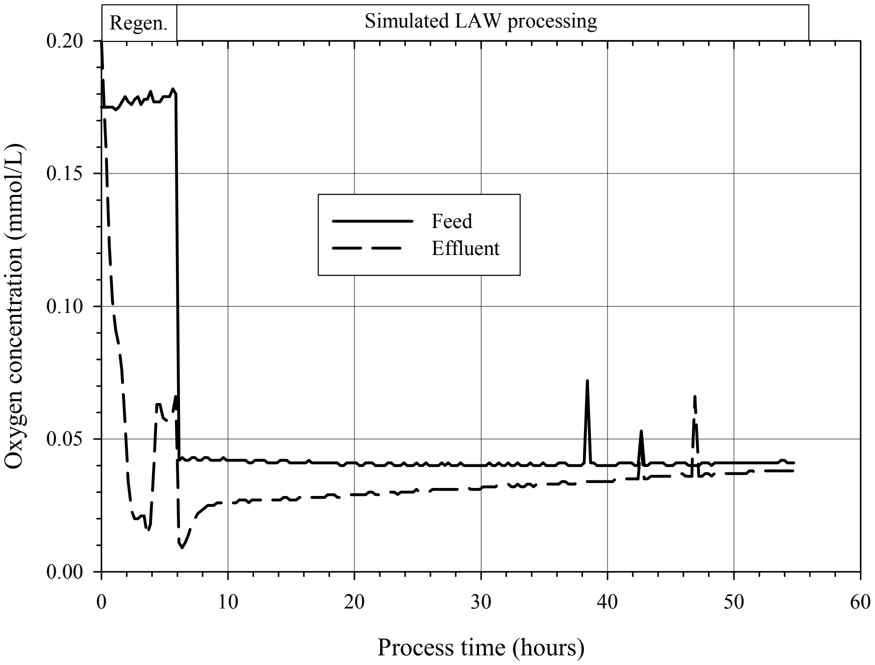

Figure 3.25. Cycle 15 Dissolved-Oxygen-Concentration Profiles (Regen – regeneration)

Table 3.26. Dissolved-Oxygen-Consumption Characterization in Cycle 15

Operation	Oxygen-Consumption Rate (μmol/h)	Total Oxygen Removed (μmol)
Regeneration	1.1	6.6
LAW processing	0.28	14

Table 3.27 presents the results of the composite simulated LAW effluent and eluate chemical analysis. There were 6.77 meq of metal in the eluate. Only Ba and Fe appear to have been significantly separated onto the resin and eluted in addition to Cs. Note again that Fe was presumably a contaminant of the reagents used to prepare the simulated LAW and the high concentration of B in the eluate probably arises from glassware contamination.

Table 3.27. Chemical Analysis of Composite Simulated LAW Effluent and Eluate from Cycle 15

		Concentration (mg/L)$^{(1,9)}$			Total mass (mg)			Fraction
Analyte	**Analysis Method**	**LAW Feed**	**LAW Effluent**	**Eluate**	**LAW Feed**	**LAW Effluent**	**Eluate**	**Separated (%)$^{(3)}$**
Ag	ICP-AES	<0.63	<0.63	0.286	<0.93	<0.92	0.057	>6
Al	ICP-AES	16,300	16,000	36.4	24,000	23,600	7.21	0.03
As	ICP-AES	62.9	*62*	<0.25	92.7	*91*	<0.05	<0.05
B	ICP-AES	29.2	36.6	6.44	43.0	53.9	1.28	11
Ba	ICP-AES	*0.56*	*0.49*	0.799	*0.83*	0.72	0.158	28
Ca	ICP-AES	<0.63	<6.3	2.99	<9.3	<9.2	0.592	>6
Cd	ICP-AES	*0.91*	*0.82*	0.827	*1.34*	*1.2*	0.164	9
Cl	IC	5,500	5,800	<13	8,100	8,600	<2.6	<0.03
Cr	ICP-AES	1,490	1,460	7.10	2,200	2,150	1.41	0.06
Cs	ICP-MS	16.3$^{(4)}$	2.72$^{(5)}$	100$^{(5)}$	24.0	4.0	20.0	83
F	IC	1,000	1,000	<13	1,500	1,500	<2.6	<0.2
Fe	ICP-AES	*1.6*	*1.5*	3.64	*2.4*	*2.2*	0.721	30
K	ICP-AES	3,190	3,080	42.9	4,700	4,540	8.49	0.2
Mg	ICP-AES	<2.5	<2.5	*0.22*	<3.7	<3.7	*0.04*	>1.0
Mo	ICP-AES	40.9	40.0	*0.084*	60.3	59.0	*0.017*	0.03
Na	ICP-AES	115,000$^{(8)}$	113,000$^{8)}$	993$^{8)}$	169,000	167,000	197	$^{(6)}$
P	ICP-AES	127	125	*0.36*	190	184	*0.07*	0.04
Pb	ICP-AES	56.3	55.4	35.8	83.0	81.6	7.09	8
Se	ICP-AES	*52*	*50*	<0.25	*77*	*74*	<0.05	<0.07
Si	ICP-AES	*120*	*110*	8.83	*180*	*160*	1.75	0.8
U	ICP-AES	<50	<50	34.1	<74	<74	6.75	>9
Zn	ICP-AES	*4.8*	*4.7*	*0.15*	*7.1*	*6.9*	*0.03*	0.4
C_2O_4	IC	<500	<500	<25	<700	<700	<5.0	<0.71
NO_2^-	IC	57,900	57,800	<25	85,300	85,400	<5.0	<0.01
NO_3^-	IC	83,500	83,800	27,600	123,000	124,000	5,480	$^{(7)}$
PO_4^-	IC	1,300	1,300	<25	1,900	1,900	<5.0	<0.7
SO_4^-	IC	2,600	2,600	87	3,800	3,800	17	0.4
TIC	HP	1,570	1,680	<10	2,300	2,480	<2.0	<0.01
Total Carbon	Furnace	2,790	2,640	46	4,100	3,880	9.1	0.22
TOC	HP	1,140	1,120	<40	1,680	1,660	<7.9	<0.47
	Furnace	45$^{(10)}$	43$^{(10)}$	<20	66	63	<4.0	<6.1
	Furnace Total Carbon – HP TIC$^{(2)}$	1,220	960	<46, >36$^{(11)}$	1,790	1,410	<9.1, >7.1$^{(11)}$	<0.51, >0.40$^{(11)}$

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus, the best TOC result may be the difference between these measurements.
3. Assumed to be the fraction of the feed constituent appearing in the eluate..
4. Nominal value.
5. Value is the product of the nominal feed concentration and fraction of ^{137}Cs determined in the solution by GEA.
6. Not applicable because resin initially in the Na form; no net separation of Na from the LAW feed.
7. Not applicable because eluate is 0.5 M HNO_3.
8. Relative % difference of 4.0% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
9. Reported results satisfy the WTP project QC criteria unless otherwise noted.
10. TOC recoveries from the caustic matrix spike lower than the QC acceptance criterion makes this result doubtful. See also Note 2.
11. Upper and lower bounds given since the HP TIC result was below detection.

3.3.12 Operational Details for Cycles 16 through 19

Table 3.28 presents the operational details for Cycles 16 through 19. All effluents from regeneration were again discolored a deep red-brown. Cycles 16 and 17 processed simulated LAW from Batch 6 while Batch 7 simulated LAW was used in Cycles 18 and 19. The cycles were performed essentially as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2. However, the Cycle 16 LAW feed flow rate was reduced to 10 mL/h approximately 5 h before termination to assure that the requisite 50 h of LAW processing were completed with the available feed.

From Cycle 17, 20 BVs of eluate were pumped through the bed for consistency with the fully sampled cycles when taking account of the samples for VOA and SVOA. As shown in Table 3.29, the eluate to LAW feed Cs^+ concentration ratio in the last 5 mL of eluate was 0.03, significantly exceeding the target of 0.01. Therefore, another 63 mL of eluate was pumped through the bed at 27 mL/h followed by a further DI water rinse. The final sample from the second elution provided a Cs^+ concentration that satisfied the target of 0.01 times that in the LAW feed. Table 3.29 also shows that the target was not achieved by a small margin in Cycle 18, but this was attributed to contamination from the bulk effluent, and a second elution was not performed.

Figure 3.26 through Figure 3.29 illustrate the breakthrough profiles. Breakthrough profiles are generally linear on the probability scale, and breakthroughs of between ~1% and ~99% were measured. The bow to the Cycle-15 breakthrough profile therefore appears to be due to a temporary deterioration in the ion exchange kinetics. The column-distribution coefficients were estimated for all cycles except 19 in which breakthrough exceeded 90% before the first sample had been taken. Table 3.30 tabulates the estimated column-distribution coefficients, which vary between 190 and 44 for Cycles 16 and 18, respectively.

Table 3.28. Operational Details for Cycles 16 Through 19

Cycle	Measurement	Unit	Regeneration (0.25 M NaOH)	Simulated AN-105 Processing	Feed Displacement (0.1 M NaOH)	Rinse (DI Water)	Elution (0.5 M HNO_3)	Rinse (DI Water)
16	Bed volume	mL	10.3	8.5	Not measured	Not measured	6.7	6.0
	Reagent volume	mL	58	1500	44	45	221	143
		BV	5.6	146	4.3	4.4	21	14
	Reagent flow rate	mL/h	9.7	63 / 10	29	30	10 / 27	32
		BV/h	0.94	6.1 / 0.97	2.9	2.9	1.0 / 2.6	3.1
17	Bed volume	mL	10.3	8.3	Not measured	Not measured	Not measured	6.0
	Reagent volume	mL	62	1459	41	47	199	76
		BV	6.0	142	4.0	4.6	19	7.4
	Reagent flow rate	mL/h	10	30	28	32	10	51
		BV/h	1.0	2.9	2.7	3.1	0.97	4.9
18	Bed volume	mL	10.3	8.0	9.7	9.7	5.7	Not measured
	Reagent volume	mL	59	716	44	45	202	45
		BV	5.8	69	4.3	4.4	20	4.3
	Reagent flow rate	mL/h	9.9	15	30	30	10	30
		BV/h	0.96	1.5	2.9	2.9	0.98	2.9
19	Bed volume	mL	10.3	7.8	Not measured	Not measured	Not measured	6.0
	Reagent volume	mL	61	3058	45	51	203	46
		BV	5.9	297	4.4	5.0	20	4.4
	Reagent flow rate	mL/h	10	64	30	30	10	30
		BV/h	0.98	6.2	2.9	3.0	0.98	2.9

Table 3.29. Elution Performance Data for Cycles 11 Through 14

Cycle	Eluate to Simulated LAW Feed Cs^+ Concentration Ratio in Last 5 mL of Eluate
16	0.03 / 0.01
17	0.005
18	0.016
19	0.007

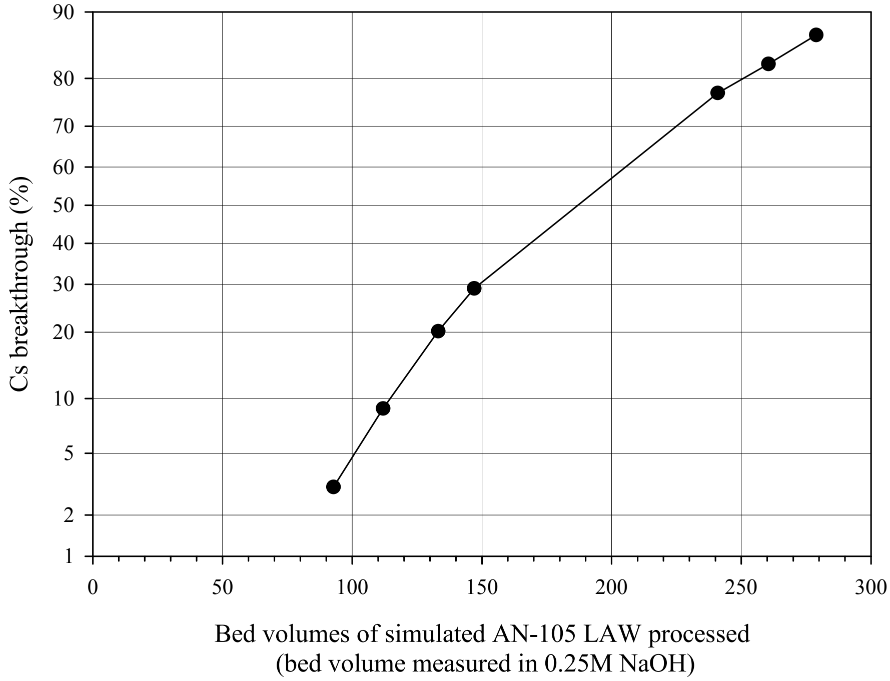

Figure 3.26. **Cycle 16 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 6.1 BV/h, ~5 M Na, 0.066 mM Cs^+, 8 mM K^+, 1.2 M OH^-, 0.5 M NO_3^-, 2.46 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.5 mL in simulated AN-105 LAW)**

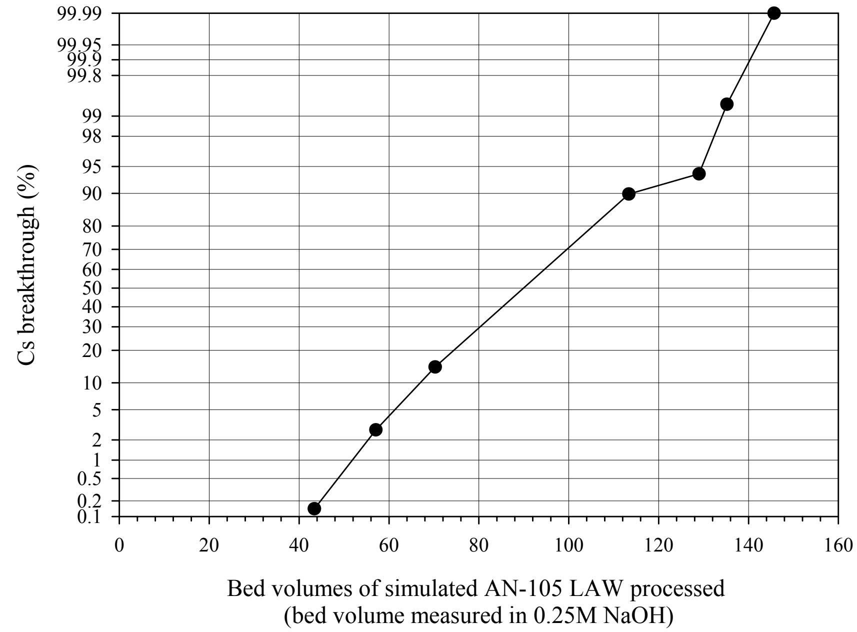

Figure 3.27. Cycle 17 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.9 BV/h, ~5 M Na, 0.235 mM Cs^+, 8 mM K^+, 1.2 M OH^-, 0.5 M NO_3^-, 2.46 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.3 mL in simulated AN-105 LAW)

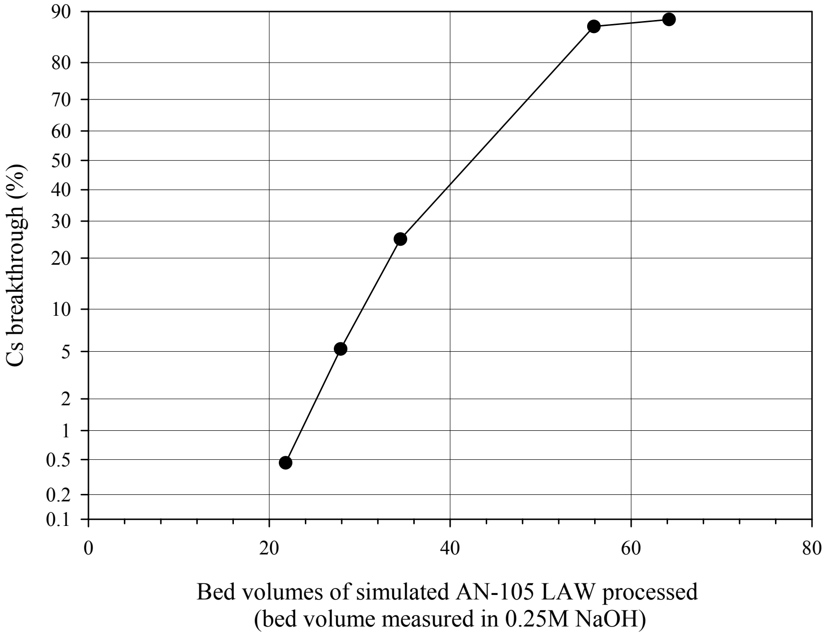

Figure 3.28. Cycle 18 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 1.5 BV/h, ~5 M Na, 0.235 mM Cs^+, 0.8 M K^+, 2.2 M OH^-, 1.36 M NO_3^-, 0.58 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.0 mL in simulated AN-105 LAW)

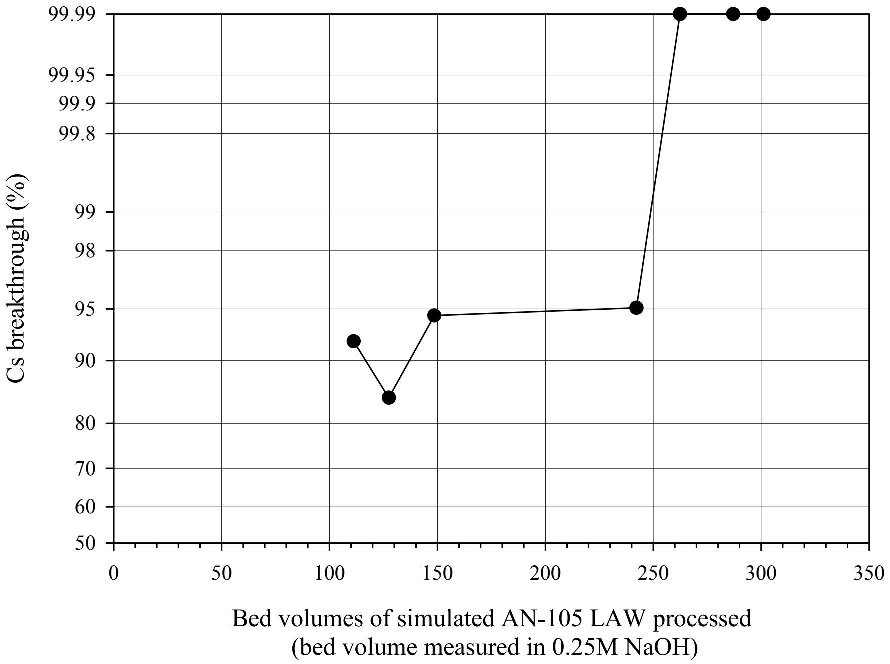

Figure 3.29. Cycle 19 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 6.2 BV/h, ~5 M Na, 0.066 mM Cs^+, 0.8 M K^+, 2.2 M OH^-, 1.36 M NO_3^-, 0.58 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 7.8 mL in simulated AN-105 LAW)

Table 3.30. Estimated Column-Distribution Coefficients for Cycles 16 Through 19

Cycle	Simulated LAW batch #	Concentrations			Flow rate (BV/h)	Column-Distribution Coefficient
		Cs^+ (mM)	K^+ (M)	OH^- (M)		
16	6	0.066	0.008	1.20	6.1	190
17	6	0.235	0.008	1.20	2.9	90
18	7	0.235	0.80	2.20	1.5	44
19	7	0.066	0.80	2.20	6.2	Not measured

3.3.13 Operational Details for Cycle 20

Table 3.31 presents the operational details for Cycle 20. The system was operated as expected based on the schedule outlined in Sections 3.3.1 and 3.3.2. The effluent from regeneration was again collected in two fractions, both of which were discolored a deep red-brown. No resin fines were observed.

Table 3.31. Cycle 20 Operational Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	10.3	61	5.9	10	1
Waste processing	Batch 4 simulated AN-105 LAW	8.3	1489	145	30	2.9
Feed displacement	0.1 M NaOH	10.1	44	4.3	30	2.9
Rinse	DI water	10.6	45	4.4	30	2.9
Elution	0.5 M HNO_3	6.7	208	20	10	1.0
Rinse	DI water	5.8	45	4.4	30	2.9

The simulated AN-105 LAW feed contained Cs^+ at a concentration identical to that used in Cycle 15 at 1.22E-4 M. The breakthrough profile presented in Figure 3.30 shows that resin breakthrough performance had deteriorated over that observed in Cycle 15. For example, the column-distribution coefficient (50% breakthrough) was 108 and 96 in Cycles 15 and 20, respectively. However, initial breakthrough at 1% occurred after processing approximately the same volume of simulated LAW (~50 BVs) in Cycles 15 and 20.

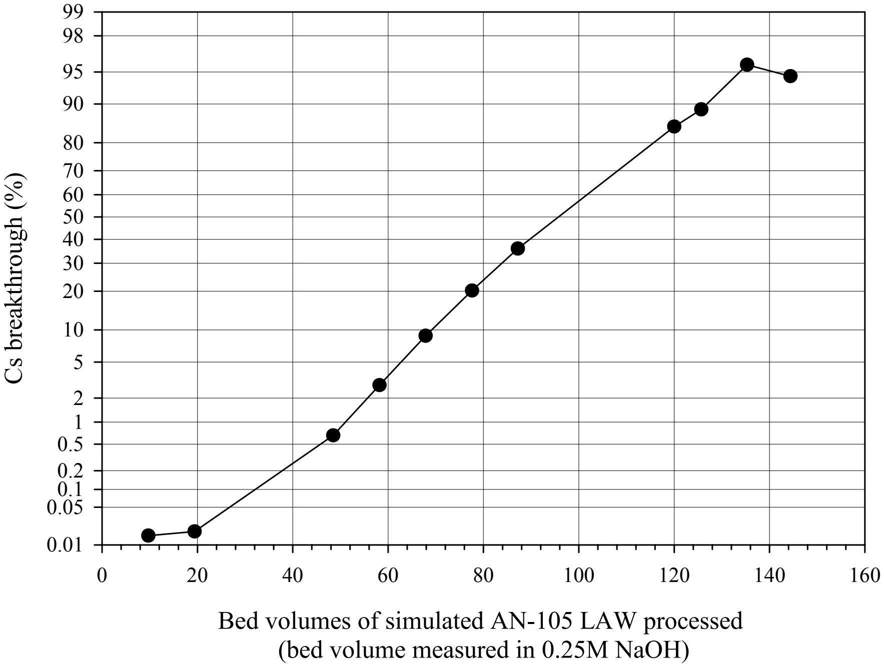

Figure 3.30. Cycle 20 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.9 BV/h, 5.00 M Na, 0.122 mM Cs^+, 8.1 mM K^+, 1.72 M OH^-, 1.35 M NO_3^-, 1.26 M NO_2^-, ambient conditions, BV = 10.3 mL in 0.25 M NaOH and 8.3 mL in simulated AN-105 LAW)

Figure 3.31 presents the elution profile. The Cs^+ concentration in the eluate peaked when ~4 BVs of eluate had been generated and was 1% of that in the simulated LAW feed after ~10.4 BVs of eluate had been generated. The first three eluate fractions were light tan in color, but no precipitation was observed.

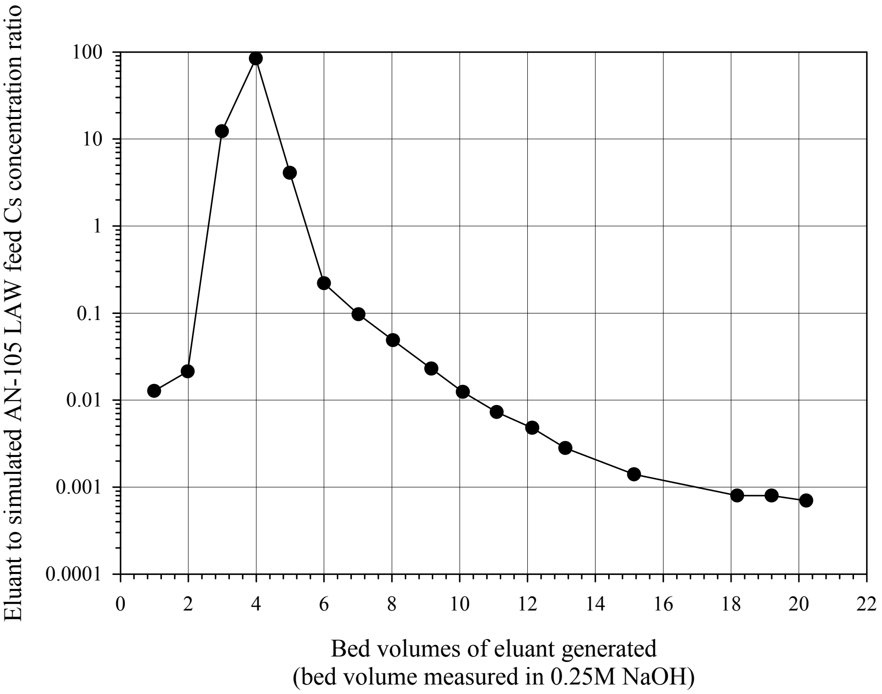

Figure 3.31. Cycle 20 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 1.0 BV/h, ambient conditions, BV = 10.3 mL in 0.25 M NaOH)

The activity balance for Cycle 20 is presented in Table 3.32 and shows that approximately 80% of the influent Cs was separated onto the resin and recovered by elution. The balance is considered reasonable with 17% more of the activity fed to the system accounted for in the effluents.

Table 3.32. Activity Balance for Cycle 20

Process Stream	Total Count Rate (CPM)	Fraction of feed (%)
Simulated LAW Feed	9.00E5	100
Simulated LAW effluent	3.08E5	34
Feed displacement effluent	1.60E4	1.8
Rinse effluent	1.19E3	0.1
Elution effluent	7.24E5	80
Rinse effluent	1.13E1	0.0
Total recovery of feed ^{137}Cs in effluents	9.93E5	117

Table 3.33 presents the results of the composite simulated LAW effluent and eluate chemical analysis. There were 7.86 meq of metal in the eluate. As observed in previous cycles, the eluate contained significant fractions of the feed Ag, Ba, Fe and Ca. Note again that Fe was presumably a contaminant of the reagents used to prepare the simulated LAW.

Table 3.33. Chemical Analysis of Composite Simulated LAW Effluent and Eluate from Cycle 20

		Concentration (mg/L)(1,9)			Total mass (mg)			Percentage
Analyte	Analysis Method	LAW Feed	LAW Effluent	Eluate	LAW Feed	LAW Effluent	Eluate	Separated (%)(3)
Ag	ICP-AES	<0.63	<0.63	1.48	<0.93	<0.93	0.308	>33
Al	ICP-AES	16,300	16,400	19.7	24,300	24,400	4.10	0.02
As	ICP-AES	62.9	*62*	<0.25	93.7	*92*	<0.05	<0.05
B	ICP-AES	29.2	37.2	6.62	43.5	55.4	1.38	3
Ba	ICP-AES	*0.56*	*0.53*	0.681	*0.83*	0.79	0.142	17
Ca	ICP-AES	<0.63	<0.63	2.52	<0.93	<0.93	0.524	>56
Cd	ICP-AES	*0.91*	*1.0*	0.793	*1.4*	*1.5*	0.165	12
Cl	IC	5,500	4,600	32	8,200	6,800	6.7	0.08
Cr	ICP-AES	1,490	1,480	3.54	2,220	2,200	0.736	0.03
Cs	ICP-MS	16.3(4)	3.23(5)	93.0 (5)	24.2	4.8	19.4	80
F	IC	1,000	700	<13	1,500	1,000	<2.7	<0.2
Fe	ICP-AES	*1.6*	*1.4*	2.87	*2.4*	*2.1*	0.597	25
K	ICP-AES	3,190	3,260	38.9	4,750	4,850	8.09	0.17
Mg	ICP-AES	<2.5	*3.6*	*0.21*	<3.7	*5.4*	*0.04*	>1
Mo	ICP-AES	40.9	40.1	<0.05	60.9	59.7	<0.01	<0.02
Na	ICP-AES	115,000(8)	115,000(8)	844(8)	171,000	171,000	176	(6)
P	ICP-AES	127	131	*0.20*	189	195	*0.04*	<0.01
Pb	ICP-AES	56.3	54.8	29.9	83.8	81.6	6.22	7
Se	ICP-AES	*52*	*49*	<0.25	*77*	*73*	<0.05	<0.06
Si	ICP-AES	*120*	127	7.95	*180*	189	1.65	0.9
U	ICP-AES	<50	<50	29.5	<74	<74	6.14	>8.0
Zn	ICP-AES	*4.8*	17.7(7)	*0.15*	*7.1*	26.4	*0.03*	0.4
C_2O_4	IC	<500	300	<25	<700	450	<5.2	<0.74
NO_2^-	IC	57,900	57,500	<25	86,100	85,500	<5.2	<0.01
NO_3^-	IC	83,500	83,400	23,100	124,000	124,000	4,810	(12)
PO_4^-	IC	1,300	1,400	<25	1,900	2,100	<5.2	<0.3
SO_4^-	IC	2,600	1,100	<25	3,900	1,600	<5.2	<0.1
TIC	HP	1,570	1,670	<10	2,330	2,480	<2.1	<0.09
Total Carbon	Furnace	2,790	2,640	25	4,150	3,930	5.2	0.13
TOC	HP	1,140	1,120	<40	1,700	1,670	<8.3	<0.49
	Furnace	45(10)	200(10)	<19	67	300	<4.0	<6.0
	Furnace Total Carbon – HP TIC(2)	1,220	970	<25, >15(11)	1,810	1,440	<5.2, >3.1(11)	<0.29, >0.17(11)

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus, the best TOC result may be the difference between these measurements.
3. Assumed to be the fraction of the feed constituent appearing in the eluate.
4. Nominal value.
5. Value is the product of the nominal feed concentration and fraction of ^{137}Cs determined in the solution by GEA.
6. Not applicable since resin initially in the sodium form.
7. Observed Zn concentration in the blank did not satisfy QC acceptance criteria, and this Zn concentration consequently is likely up to 75% over-estimated
8. Relative % difference of 4.0% between duplicates did not satisfy the QC acceptance criterion of 3.5%. No significant impact on results expected.
9. Reported results satisfy the WTP project QC criteria unless otherwise noted.
10. TOC recoveries from the caustic matrix spike lower than the QC acceptance criterion makes this result doubtful. See also Note 2.
11. Upper and lower bounds given since the HP TIC result was below detection.
12. Not applicable since eluate is 0.5M HNO_3.

3.3.14 Operational Details for Cycles 21 through 24

Table 3.34 provides the operational details for Cycles 21 through 24. All effluents from regeneration were again discolored a deep red-brown. Cycle 21 processed simulated LAW from Batch 7 except for the final 11 h and 20 min when the effluent was recycled in order to maintain the desired LAW processing time of 50 h. Simulated LAW from Batch 8 was processed in Cycles 22 through 24.

Significant gas generation was observed in Cycle 23 after 3 h of the regeneration operation. Three bubbles approximately 5 mm in diameter were first observed (Figure 3.32), which coalesced to fill the entire cross-section of the column about a third of the way up from the base (Figure 3.33). The 0.25 M NaOH solution continued to flow down the column wall with no significant increase in pressure drop. The bed re-settled about a half hour after the bubbles were first observed by a combination of resin particles from the top becoming entrained into the 0.25 M NaOH and the bubble working its way up through the bed. Another bubble again filled the column cross-section approximately ¼ of the way up 15 min later, and a significant volume of gas was observed in the effluent line. The bed had re-settled by the same mechanism noted before 10 min later. Another bubble was observed to be forming approximately 5 mm from the base of the bed 5 min after the bed had last re-settled. This bubble again grew to fill the column cross-section, and the bed re-settled 15 min after it was first observed. No further gas-generation events were observed, but the bed was fluidized since its high volume of 11 mL appeared to indicate the hold-up of gas within it. The BV had reduced to 10.6 mL after it had been fluidized.

Occurrence of this phenomenon in prior cycles cannot be discounted since the tests were not continuously watched, and the bed appeared normal once re-settled. Indeed, the gas bubbles observed in Cycles 13 and 14 may be indicative of this phenomenon occurring.

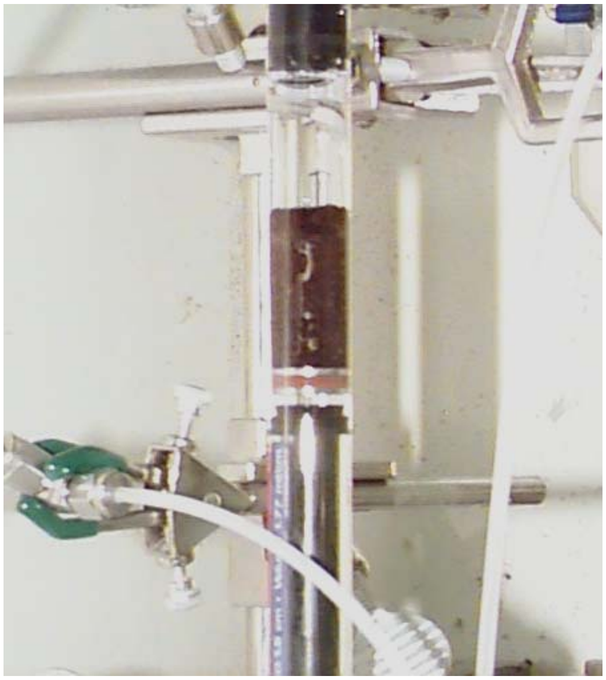

Figure 3.32. Initial Generation of Gas Bubbles in Cycle 23 Regeneration (after processing 3 BVs of 0.25M NaOH)

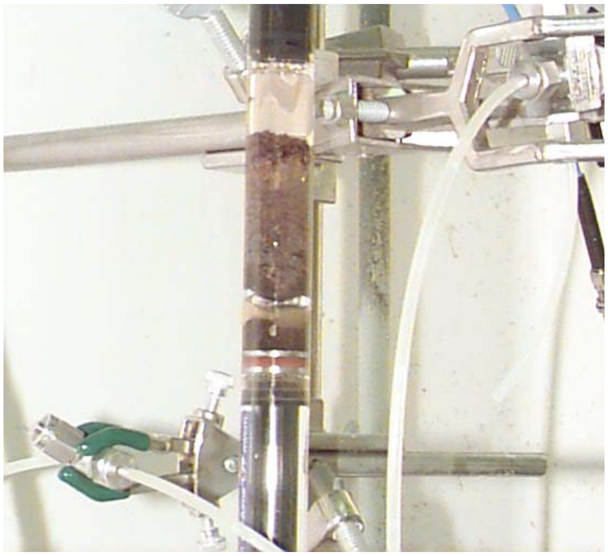

Figure 3.33. Final Coalescence of Gas Bubbles to Separate the Bed in Cycle 23 Regeneration (after processing 3 BVs of 0.25M NaOH)

Gas generation was also observed after 3 h in the regeneration operation of Cycle 24. Bubbles were formed approximately ¼ of the way up from the base of the bed but did not coalesce. Bubbles were also observed in the effluent line. No further bubbles were observed an hour after they were first observed.

Figure 3.34, Figure 3.35, and Figure 3.36, respectively, provide the breakthrough profiles for Cycles 21, 23, and 24. The profile for Cycle 22 is not shown since breakthrough was >90% when the first sample was taken after ~110 BVs of simulated LAW had been processed. The profiles are approximately linear on the probability scale, and column-distribution coefficients of ~50 are provided in Table 3.35.

Table 3.34. Operational Details for Cycles 21 Through 24

Cycle	Measurement	Unit	Regeneration (0.25 M NaOH)	Simulated AN-105 Processing	Feed Displacement (0.1 M NaOH)	Rinse (DI water)	Elution (0.5 M HNO_3)	Rinse (DI water)
21	Bed volume	mL	10.4	8.3	Not measured	Not measured	5.7	5.8
	Reagent volume	mL	60	1477	45	44	199	45
		BV	5.8	142	4.4	4.3	19	4.3
	Reagent flow rate	mL/h	10	30	31	29	10	30
		BV/h	1.0	2.9	3.0	2.8	0.96	2.9
22	Bed volume	mL	10.6	8.1	Not measured	Not measured	Not measured	6.0
	Reagent volume	mL	59	3095	~46(1)	~44(1)	199	46
		BV	5.5	292	~4.4(1)	~4.2(1)	19	4.3
	Reagent flow rate	mL/h	9.8	63	~31(1)	~30(1)	10	30
		BV/h	0.92	5.9	~2.9(1)	~2.8(1)	0.94	2.9
23	Bed volume	mL	10.6	8.5	Not measured	Not measured	Not measured	6.0
	Reagent volume	mL	~60(1)	735	45	44	200	50
		BV	~5.6(1)	69	4.2	4.2	19	4.7
	Reagent flow rate	mL/h	~1.0(1)	15	30	29	10	33
		BV/h	~0.94(1)	1.4	2.8	2.8	0.94	3.1
24	Bed volume	mL	10.1	8.0	Not measured	Not measured	5.5	5.5
	Reagent volume	mL	59	1108	45	45	197	44
		BV	5.9	110	4.5	4.4	20	4.4
	Reagent flow rate	mL/h	9.9	30 / 15	31	30	9.9	29
		BV/h	0.98	2.9 / 1.5	3.0	3.0	0.98	2.9

1. Values approximate since effluent bottle inadvertently not weighed before starting operation. Used the weight previously recorded for the same bottle.

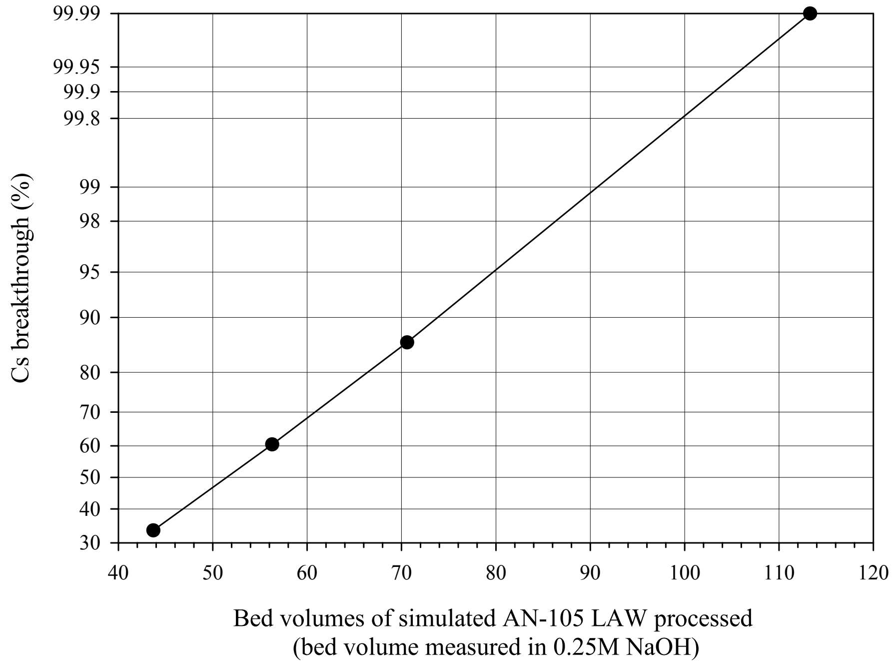

Figure 3.34. **Cycle 21 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.9 BV/h, ~5 M Na, 0.066 mM Cs^+, 0.8 M K^+, 2.2 M OH^-, 1.36 M NO_3^-, 0.58 M NO_2^-, ambient conditions, BV = 10.4 mL in 0.25 M NaOH and 8.3 mL in simulated AN-105 LAW)**

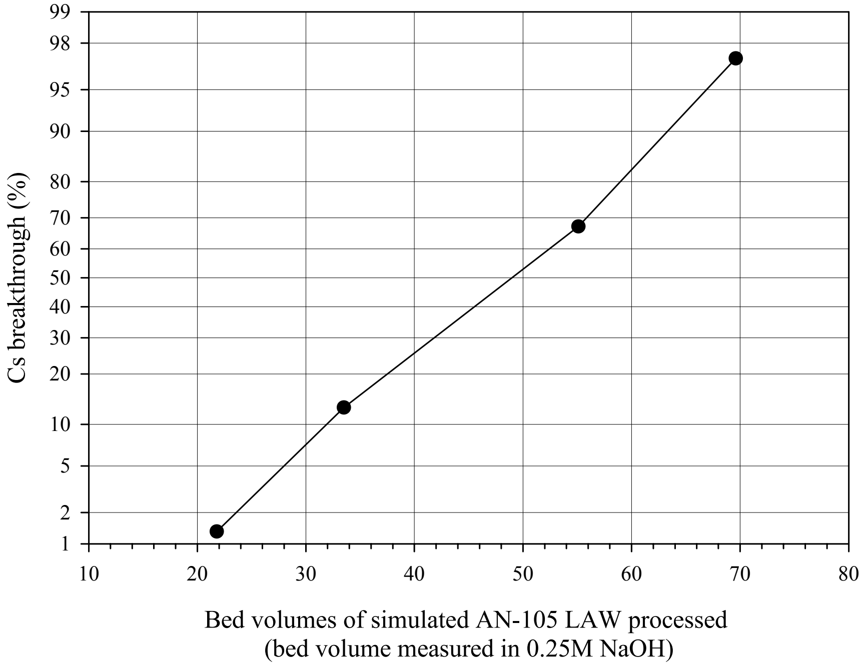

Figure 3.35. **Cycle 23 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 1.4 BV/h, ~5 Mop Na, 0.066 mM Cs^+, 0.8 M K^+, 1.2 M OH^-, 1.36 M NO_3^-, 1.6 M NO_2^-, ambient conditions, BV = 10.6 mL in 0.25 M NaOH and 8.5 mL in simulated AN-105 LAW)**

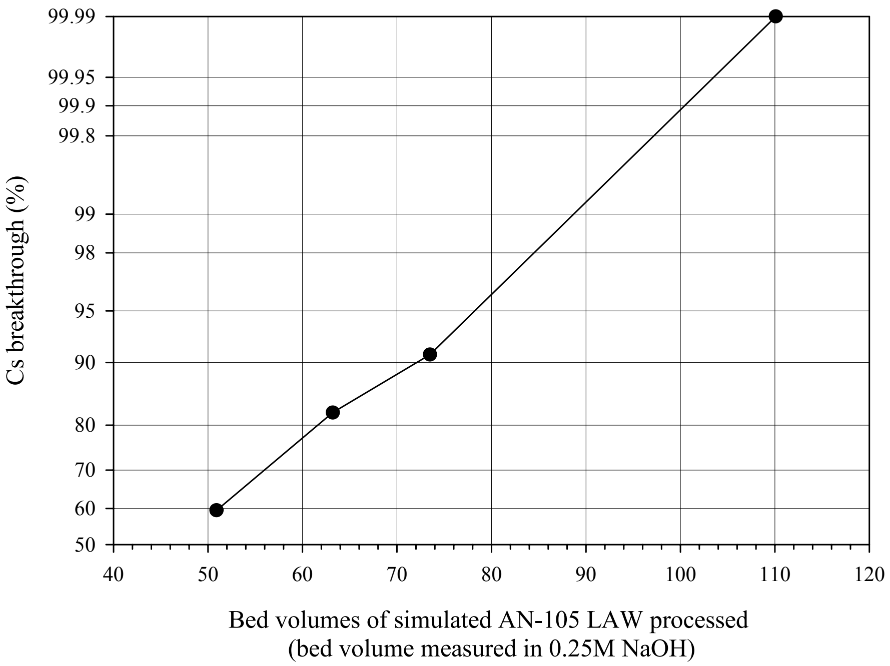

Figure 3.36. Cycle 24 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 2.9 BV/h, ~5 M Na, 0.066 mM Cs^+, 0.8 M K^+, 1.2 M OH^-, 1.36 M NO_3^-, 1.6 M NO_2^-, ambient conditions, BV = 10.1 mL in 0.25 M NaOH and 8.0 mL in simulated AN-105 LAW)

Table 3.35. Estimated Column-Distribution Coefficients for Cycles 21 Through 24

Cycle	Simulated LAW Batch #	Concentrations			Flow Rate (BV/h)	Column Distribution Coefficient
		Cs^+ (mM)	K^+ (M)	OH^- (M)		
21	7	0.066	0.80	2.20	2.9	54
22	8	0.235	0.80	1.20	5.9	<107(1)
23	8	0.066	0.80	1.20	1.4	48
24	8	0.066	0.80	1.20	2.9	46

Note: 1. This value is the number of BVs processed when the first sample was taken, which returned a breakthrough of >90%. The column distribution coefficient (given at 50% breakthrough) is therefore less than this value.

Table 3.36 shows that the target eluate to simulated LAW feed Cs concentration ratio of 0.01 was apparently not achieved in Cycles 21 and 23. This was attributed to contamination from the bulk effluent (the effluent line was transferred from the bulk-effluent collection bottle to the sample vial) since results from fully sampled cycles shows that the target was achieved in no more than 13.5 BVs.

Table 3.36. Elution Performance in Cycles 21 Through 24

Cycle	Eluate to Simulated LAW Feed Cs Concentration Ratio in Last 5 mL of Eluate
21	0.020
22	0.005
23	0.022
24	0.008

The oxygen-concentration profiles are illustrated in Figure 3.37 and Figure 3.38 for Cycles 21 and 22, respectively. There is a spike in the effluent concentration just before commencing LAW processing, and the effluent concentration increases during elution, as observed previously.

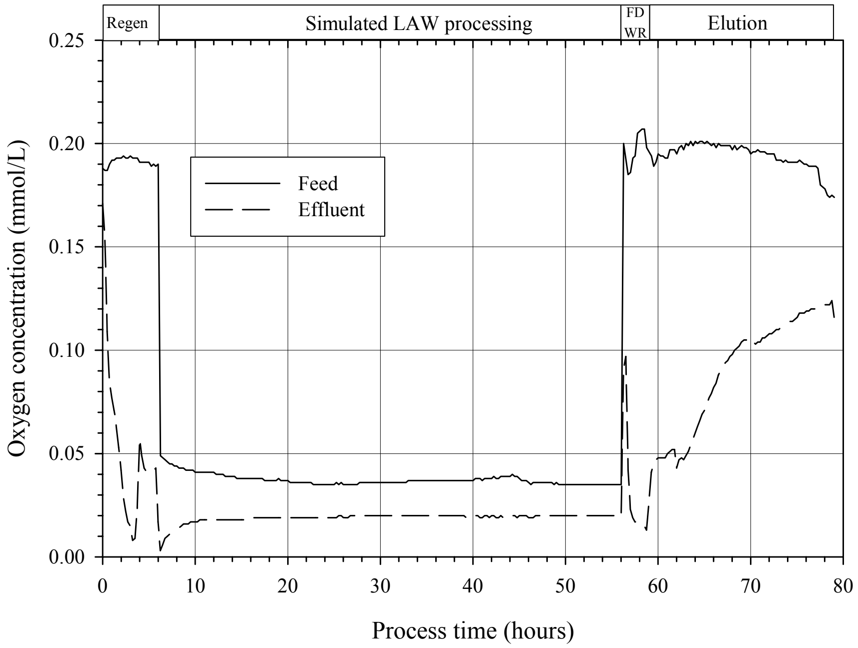

Figure 3.37. Oxygen-Concentration Profile for Cycle 21 (Regen – regeneration, FD – 0.1 M NaOH feed displacement, WR – water rinse, ER – elution rinse)

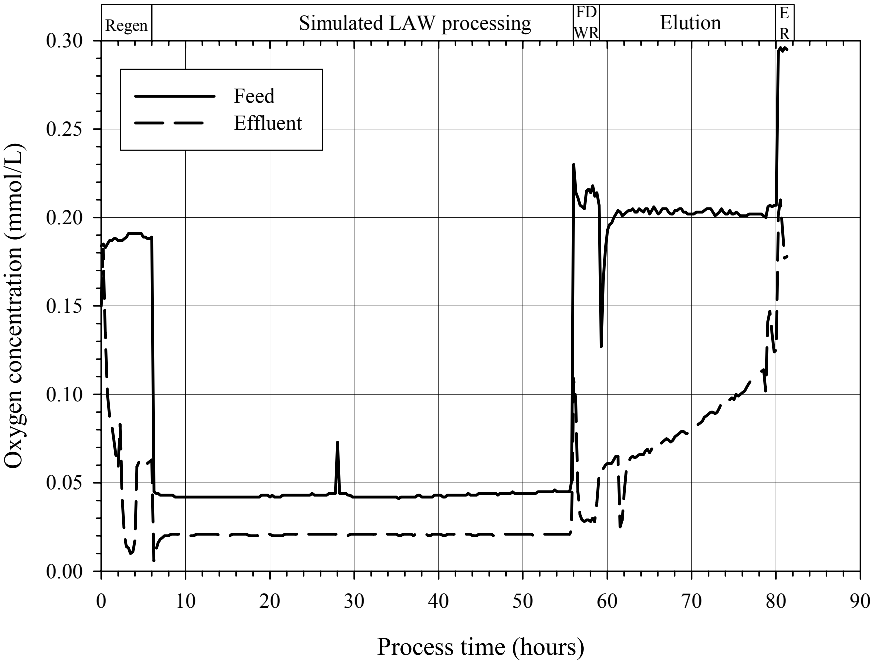

Figure 3.38. Oxygen-Concentration Profile for Cycle 22 (Regen – regeneration, FD – 0.1 M NaOH feed displacement, WR – water rinse, ER – elution rinse)

Table 3.37 presents the oxygen-consumption rates and the total quantities removed for each operation of Cycles 21 and 22. As in previous cycles, the oxygen-consumption rate generally increased with increasing dissolved-oxygen-feed concentration. The proportions of total oxygen removed was approximately equal in the elution and LAW processing operations of Cycle 21, in contrast to earlier cycles. Indeed, the greatest proportion of the total quantity of oxygen removed during all operations occurred when processing the simulated LAW in Cycle 22.

Table 3.37. Dissolved-Oxygen-Consumption Characterization in Cycles 21 and 22

	Oxygen-Consumption Rate (μmol/h)		Total Oxygen Removed (μmol)		Fraction of Total Oxygen Removed (%)	
Cycle / Operation	21	22	21	22	21	22
Regeneration	1.4	1.3	8.4	7.8	9.8	6.3
LAW processing	0.56	1.4	28	70	33	56
0.1 M NaOH rinse	4.3	5.6	6.5	8.4	7.6	6.8
Water rinse	7.6	6.6	11	9.9	13	7.8
Elution	1.5	1.2	30	24	35	19
Water rinse	1.4	2.7	2.1	4.1	2.4	3.3

3.3.15 Operational Details for Cycle 25

Table 3.38 presents the operational details for Cycle 25. The system was operated without deviating from the plan except that an extra 4 BVs of eluate were generated to investigate the effect of extended elution on the elution profile. The effluent from regeneration was again collected in two fractions, both of which were discolored a deep red-brown. Fine resin material was observed in the second fraction.

Table 3.38. Cycle 25 Operational Details

		Measured Resin Bed Volume	Total Volume of Reagent		Flow Rate of Reagent	
Operation	**Reagent**	**mL**	**mL**	**BV**	**mL/h**	**BV/h**
Regeneration	0.25 M NaOH	9.9	64	6.4	11	1.1
Waste processing	Batch 8 simulated AN-105 LAW	8.0	1474	149	30	3.0
Feed displacement	0.1 M NaOH	9.2	42	4.3	28	2.8
Rinse	DI water	10.1	45	4.5	30	3.0
Elution	0.5 M HNO_3	5.8	241	24	10	1.0
Rinse	DI water	5.5	45	4.5	30	3.0

The simulated AN-105 LAW feed contained Cs^+ at a concentration identical to that used in Cycle 20 at 1.22E-4 M. The breakthrough profile presented in Figure 3.39 shows that resin-breakthrough performance had deteriorated over that observed in Cycle 20. For example, the column-distribution coefficient (50% breakthrough) was 96 and 82 in Cycles 20 and 25, respectively. However, initial breakthrough at 1% occurred after processing approximately the same volume of simulated LAW (~50 BVs) in Cycles 15, 20, and 25.

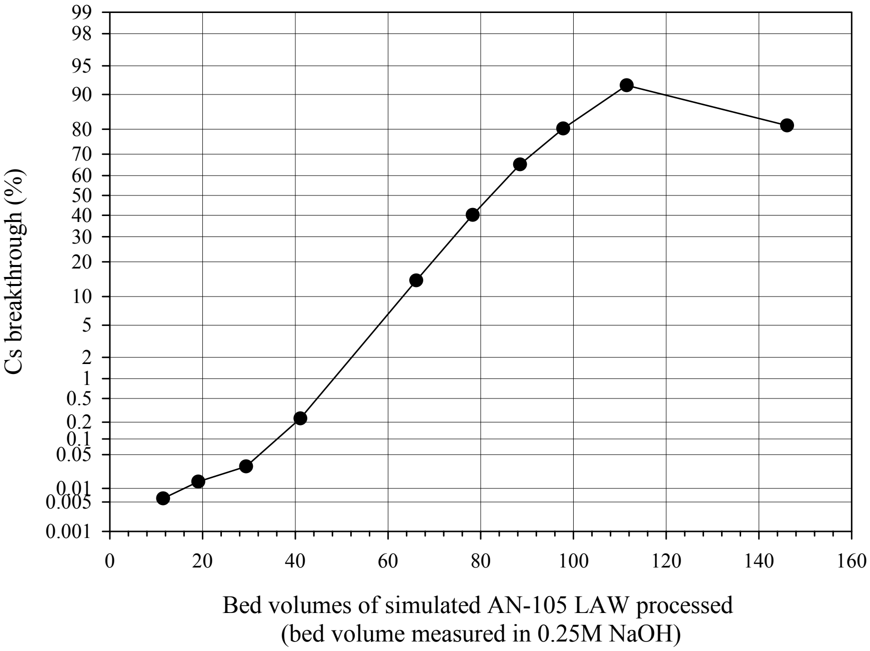

Figure 3.39. Cycle 25 Cesium Breakthrough Performance (SL-644 resin batch SMC-010319SMC-IV-73, 3.0 BV/h, 5.00 M Na, 0.122 mM Cs^+, 8.6 mM K^+, 1.72 M OH^-, 1.36 M NO_3^-, 1.26 M NO_2^-, ambient conditions, BV = 9.9 mL in 0.25 M NaOH and 8.0 mL in simulated AN-105 LAW)

Figure 3.40 presents the elution profile. The Cs^+ concentration in the eluate peaked when ~4 BVs of eluate had been generated and was 1% of that in the simulated LAW feed after ~10.4 BVs of eluate had been generated. The rate of reduction in the eluate Cs^+ concentration decreased significantly after ~15 BVs of eluate had been generated.

The activity balance for Cycle 25 is presented in Table 3.39 and shows that approximately 73% of the influent Cs was separated onto the resin and recovered by elution. The balance shows that 83% of the activity fed to the system was accounted for in the effluents.

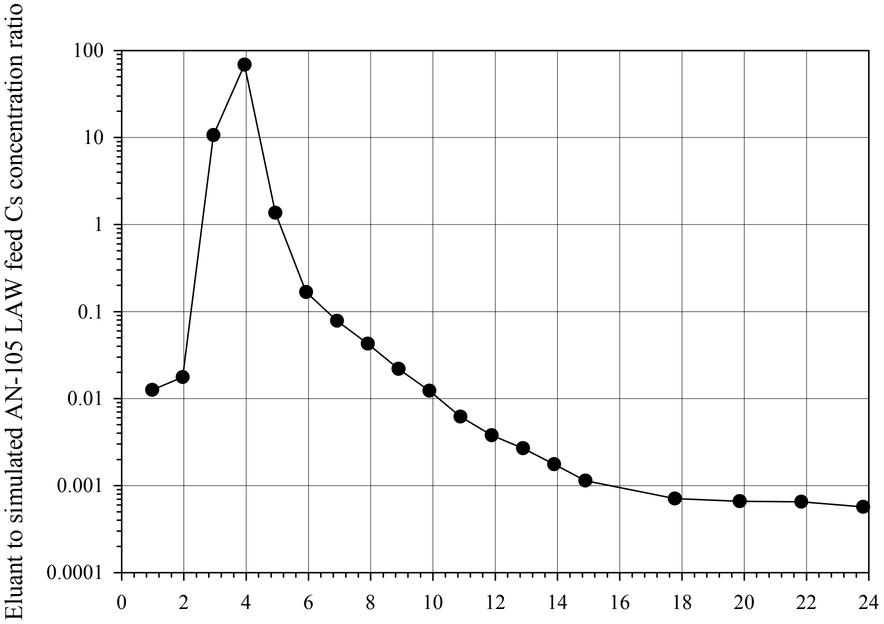

Figure 3.40. Cycle 25 Elution Profile (SL-644 resin batch SMC-010319SMC-IV-73, 1.0 BV/h, ambient conditions, BV = 9.9 mL in 0.25 M NaOH)

Table 3.39. Activity Balance for Cycle 25

Process Stream	Total Count Rate (CPM)	Fraction of Feed (%)
Simulated LAW Feed	8.62E5	100
Simulated LAW effluent	6.51E4	7.6
Feed displacement effluent	2.02E4	2.3
Rinse effluent	1.35E3	0.2
Elution effluent	6.26E5	73
Rinse effluent	1.43E1	0.0
Total recovery of feed ^{137}Cs in effluents	7.13E5	83

Figure 3.41 illustrates the oxygen-concentration profiles for Cycle 25. In common with previous cycles, there is a spike in the effluent concentration just before commencing simulated LAW processing. The concentration of dissolved oxygen in the eluate also increases during elution as previously noted. Table 3.40 tabulates the oxygen-consumption rates and quantities for each operation. The greatest consumption rates occur for those operations having the greatest influent oxygen concentration as observed for previous cycles. The elution and LAW processing operations equally accounted for the greatest proportion (~35%) of the total oxygen removed in the cycle.

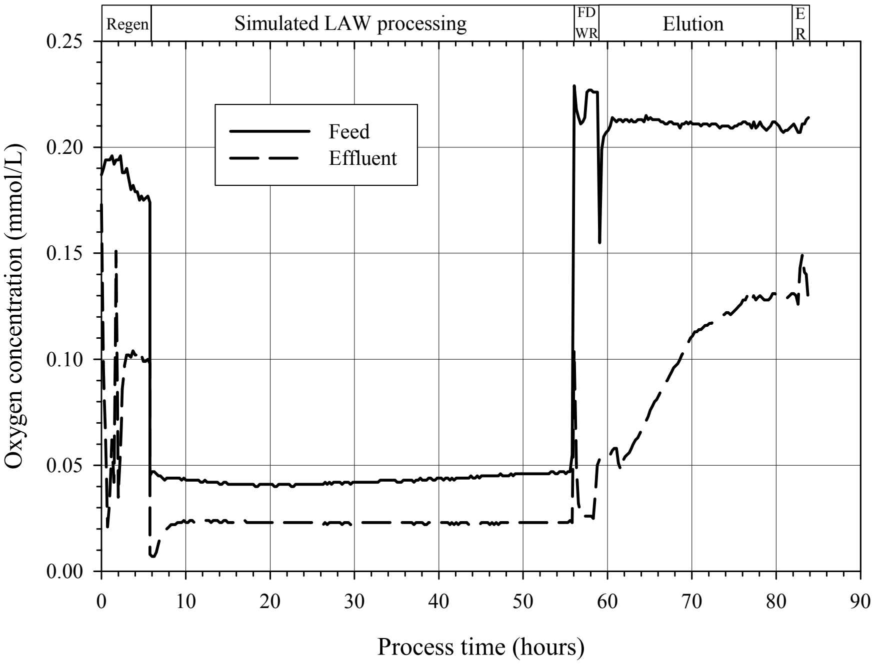

Figure 3.41. Oxygen-Concentration Profile for Cycle 25 (Regen – regeneration, FD – 0.1 M NaOH feed displacement, WR – water rinse, ER – elution rinse)

Table 3.40. Dissolved-Oxygen-Consumption Characterization in Cycle 25

Operation	Oxygen-Consumption Rate (μmol/h)	Total Oxygen Removed (μmol)	Fraction of Total Oxygen Removed (%)
Regeneration	1.4	8.4	10
LAW processing	0.56	28	32
0.1 M NaOH rinse	4.3	6.5	7.5
Water rinse	7.6	11	13
Elution	1.5	30	35
Water rinse	1.4	2.1	2.4

Table 3.41 presents the results of the composite simulated LAW effluent and eluate chemical analysis. There were 7.92 meq of metal in the eluate. As in previous cycles, significant fractions of feed Ag, Ba, Ca and Fe were observed in the eluate. Note again that Fe was presumably a contaminant of the reagents used to prepare the simulated LAW.

Table 3.41. Chemical Analysis of Composite Simulated LAW Effluent and Eluate from Cycle 25

Analyte	Analysis Method	Concentration (mg/L)(1,7)			Total mass (mg)			Fraction Separated (%)(3)
		LAW Feed	LAW Effluent	Eluate	LAW Feed	LAW Effluent	Eluate	
Ag	ICP-AES	<0.625	<0.625	2.23	<0.921	<0.921	0.449	>49
Al	ICP-AES	16,200	15,600	21.2	23,900	23,000	4.27	0.02
As	ICP-AES	*61*	*59*	<0.25	*89*	*87*	<0.05	<0.06
B	ICP-AES	20.3(8)	25.5(8)	5.70(8)	29.9	37.6	1.15	4
Ba	ICP-AES	*0.55*	*0.50*	1.07	*0.81*	*0.74*	0.215	27
Ca	ICP-AES	<0.625	<0.625	2.56	<0.921	<0.921	0.516	>56
Cd	ICP-AES	*1.4*	*1.1*	0.903	*2.1*	*1.6*	0.182	9
Cl	IC	4,600	4,500	50	6,800	6,700	10	0.1
Cr	ICP-AES	1,450	1,400	3.86	2,140	2,060	0.777	0.04
Cs	ICP-MS	15.8	4.26	84.6(4)	23.3	6.30	17.0(4)	73
F	IC	800	800	<25	1,000	1,000	<5.0	<0.5
Fe	ICP-AES	*1.8*	*1.3*	2.39	*2.7*	*1.9*	0.481	18
K	ICP-AES	3,350	3,250	49.2	4,940	4,790	9.91	0.2
Mg	ICP-AES	*3.3*	*3.4*	*0.13*	*4.9*	*5.0*	*0.03*	0.6
Mo	ICP-AES	39.3(8)	37.9(8)	*<0.05(8)*	57.9	55.9	<0.01	<0.02
Na	ICP-AES	115,000	111,000	876	170,000	170,000	176	(5)
P	ICP-AES	93.2	90.0	*0.14*	137	133	*0.03*	0.02
Pb	ICP-AES	56.5	49.1	27.0	83.3	72.4	5.44	7
Se	ICP-AES	*48*	*46*	<0.25	*71*	*68*	<0.05	<0.07
Si	ICP-AES	*89(8)*	*110(8)*	8.99(8)	*131*	162	1.81	1
U	ICP-AES	<50	<50	*6.7*	<74	<74	*1.3*	>2
Zn	ICP-AES	17.7(6)	17.1	*0.14(6)*	26.1	25,2	*0.03*	0.1
C_2O_4	IC	300	200	<50	400	300	<10	<2.6
NO_2^-	IC	57,900	57,300	<50	85,600	84,700	<10	<0.1
NO_3^-	IC	84,200	83,000	26,400	124,000	123,000	5,300	(10)
PO_4^-	IC	1,300	1,400	<50	1,900	2,100	<10	<0.6
SO_4^-	IC	1,100	1,100	<50	1,600	1,600	<10	<0.6
TIC	HP	1,490	1,580	<3	2,200	2,340	<4	<0.18
Total Carbon	Furnace	2,720	2,720	<130	4,020	4,020	<26	<0.65
TOC	HP	1,240	1,220	<9	1,830	1,800	<13	<0.71
	Furnace	<170(9)	<170(9)	<130(9)	<250	<250	<26	<10
	Furnace Total Carbon – HP TIC(2)	1,230	1,140	<130	1,820	1,690	<26	<1.4

1. ICP-AES results in normal type have errors likely <15%, but those in italics are within ten times their detection limit with errors likely exceeding 15%.
2. The furnace method typically produces the best total carbon results while the best TIC results are obtained from the HP method. Thus the best TOC result may be the difference between these measurements.
3. Assumed to be the fraction of the feed constituent appearing in the eluate..
4. Value is the product of the feed concentration and fraction of ^{137}Cs determined in the eluate by GEA.
5. Not applicable since resin eluted initially in the Na form; no net separation of Na from the LAW feed.
6. Observed Zn concentration in the blank did not satisfy QC acceptance criteria, and this Zn concentration consequently is likely up to 75% over-estimated.
7. Reported results satisfy the WTP project QC criteria unless otherwise noted.
8. B, Mo, and Si achieved recoveries of 72%, 73%, and 59%, respectively, from the matrix spike sample and so did not satisfy the QC acceptance criterion of >75%. No significant impact on results expected.
9. TOC recoveries from the matrix spike lower than the QC acceptance criterion makes this result doubtful. See also Note 2.
10. Not applicable since eluate is 0.5M HNO_3.

3.3.16 Spent Resin Analysis

The resin was removed from the column after completion of this cycle for further analysis by weighing, scanning electron microscopy (SEM), modified TCLP and digestion followed by ICP-MS to determine the concentration of residual Cs^+. The spent and dry acid form resin was found to weigh 1.268 g.

The concentrations of the TC metals in the leachates from the spent-resin leach are compared with their regulatory levels in Table 3.42. The results indicate that the spent resin would not exhibit any toxicity characteristics if a formal TCLP were performed since all of the leachate concentrations are below the regulatory levels. Note that the concentration of Ag is significantly higher in the leachate than other metals and is approximately half its regulatory level. Fine Ag precipitates, e.g. silver chloride, that were either not separated in pre-filtration of the simulated LAW or precipitated afterward might have been filtered by the ion exchange bed and then remained undissolved during elution. Indeed, the consistency between the Ag concentrations in the eluates and TCLP leachate appears to indicate that a solubility limit is being attained.

Table 3.42. Results of modified TCLP on Spent SL-644 Resin

Metal	Regulatory Level (mg/L)	Concentration in TCLP Leachate (mg/L)(1,2)
Ag	5.0	2.77
As	5.0	<0.39
Ba	100.0	<0.02
Cd	1.0	<0.02
Cr	5.0	*0.094*
Hg	0.2	0.00942
Pb	5.0	<0.16
Se	1.0	<0.39

1. Results in italics are within ten times their detection limit with errors likely exceeding 15%.
2. Reported results satisfy the WTP project QC criteria.

The resin was air-dried at room temperature and digested in 16M HNO_3 at 200°C. ICP-MS analysis of the digestate returned a Cs^+ concentration of 1.64 μg/g (0.36 μg/mL) of resin in the acid-form. The TCLP leachate was also analyzed by ICP-MS and returned a Cs^+ concentration equivalent to 0.07 μg/g (0.015 μg/mL) of resin. Therefore, the total Cs^+ remaining on the resin after the cycle 25 elution was 1.71 μg/g (0.375 μg/mL). All WTP project QC criteria were satisfied for these analyses.

4.0 Results Analysis

This section describes physical changes in resin, such as mass loss, expansion and contraction, and appearance. It also describes 1) the impact of chemical degradation on process performance and 2) the consumption of dissolved oxygen. Column and equilibrium performance are compared, results from parametric study are analyzed, and the toxicity characteristics of spent resin are described.

4.1 Physical Changes in Resin

4.1.1 Resin Mass Loss

The acid form resin was removed from the column following the elution rinse of Cycle 25, dried and found to weigh 1.268 g. The initial dry mass of as-received resin was 4.101 g, equivalent to 2.297 g in the acid form, such that an equivalent of 1.029 g of dry acid-form resin was lost from the system. Some resin (0.135 g) was lost on the filter following the initial wash with 1 M NaOH. This would represent 0.108 g of dry acid-form resin, assuming the physical properties in Table 2.6 and the resin was in the Na form such that 2.189 g of acid-form resin was loaded into the column. In addition, 0.325 g of Na-form resin, equivalent to 0.260 g of dry acid-form resin, was removed from the column in Cycle 4. Therefore, the column theoretically contained the equivalent of 1.929 g of dry acid form resin after Cycle 4. An equivalent mass of approximately 0.009 g of dry acid-form resin was removed in Cycle 13. Therefore, the total mass unaccounted for is 0.652 g of dry acid-form resin, or 34% of the total remaining after Cycle 4. Figure 4.1 illustrates the foregoing by comparing the expected bed mass based on the initial resin mass and subsequent extractions with the mass measured after the resin was removed after Cycle 25.

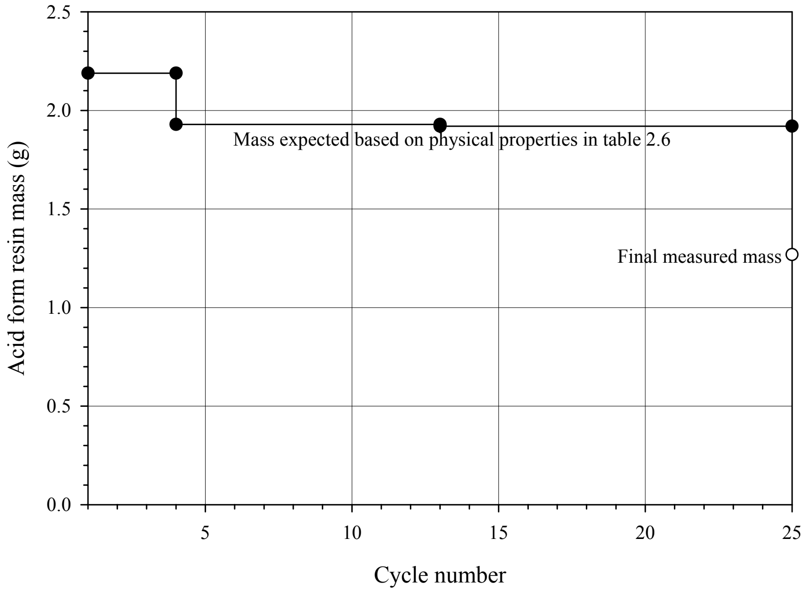

Figure 4.1. Comparison of Expected and Measured Acid-form Bed Masses

The regeneration effluents of every cycle were observed to be colored deep red-brown, presumably due to resin dissolution, and some contained resin fines that settled after ~1 day. An acid-form resin mass-loss rate of 0.031 g/cycle (1.5% per cycle) would account for the total mass lost, and this rate is equivalent to 0.041 g/cycle of Na-form resin. Assuming the dry bed density of 0.22 g/mL in 0.25 M NaOH, this mass rate is equivalent to 0.19 mL of wet resin per cycle.

4.1.2 Resin Expansion and Contraction

Figure 4.2 illustrates the changes in resin contraction and expansion as the different reagents are processed for each operation. The BV in 0.25 M NaOH is plotted with respect to the baseline BV of 10 mL. Bed volumes in other reagents are plotted with respect to the BV in 0.25 M NaOH measured during the regeneration operation of the same cycle.

The BV appears to contract to progressively smaller volumes in the LAW and in nitric acid and water when in the acid form, relative to its volume in 0.25 M NaOH in the same cycle, for each successive cycle up to Cycle 15. The degree of contraction appears to become steady after Cycle 17. The relative volume in the Na form in 0.1 M NaOH and DI water essentially remained constant for all cycles.

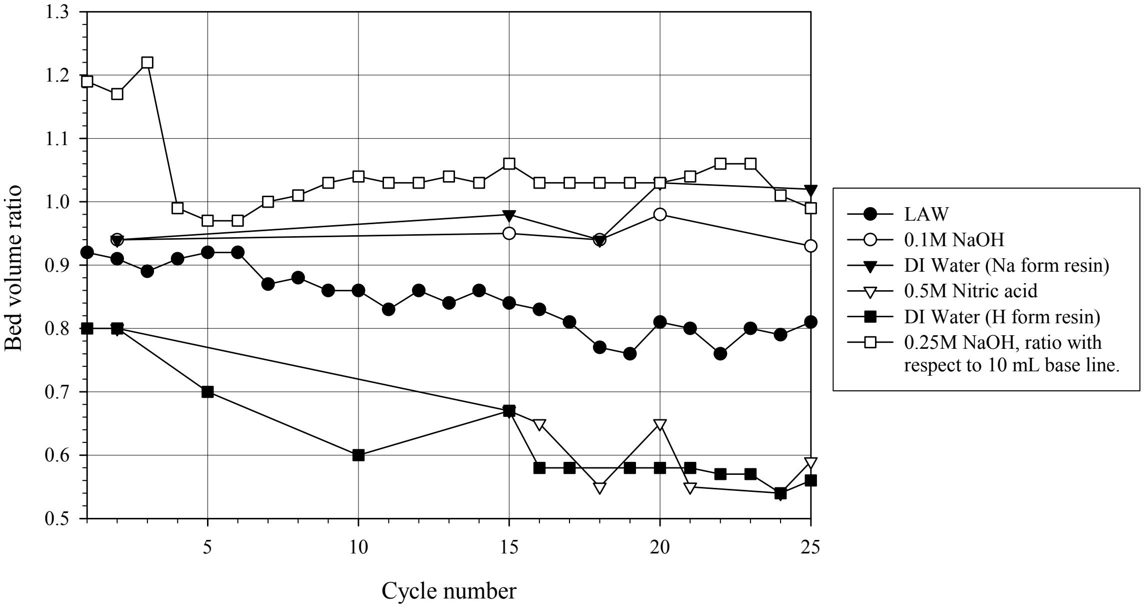

Figure 4.2. Bed Volume Variability During the Chemical Degradation Test (BV expressed as its ratio to that in 0.25 M NaOH except as noted in legend)

Note that the bed appears to be expanding more during regeneration in each cycle to maintain its volume since approximately 34% of the acid-form resin mass was lost from the column during operations. As illustrated in Figure 4.3, the dry-bed density of the resin in 0.25 M NaOH therefore decreased from 0.234 g/mL in cycle 1 to 0.160 g/mL in Cycle 25. Indeed, the 34% loss in resin mass is nearly consistent with the reduction in BV of the acid-form resin (38%), and the acid-form dry-bed density reduced by only 6% from 0.233 g/mL to 0.219 g/mL.

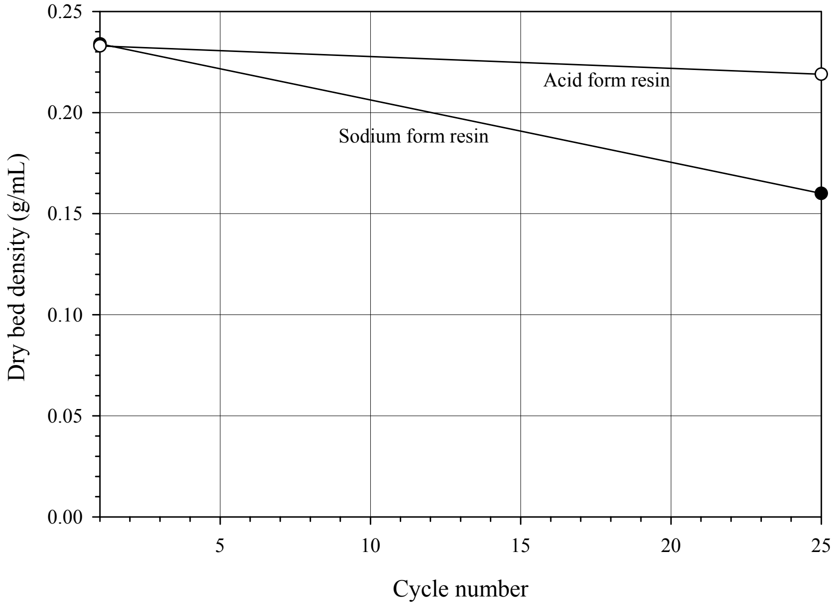

Figure 4.3. Comparison of the Reduction in Dry Bed Density for the Resin in the Acid and Sodium Forms (Resin batch 010319SMC-IV-73)

4.1.3 Resin Appearance

Individual resin particles were examined by SEM. As shown in Figure 4.4, at low magnification (x135), the used and unused resins appear similar with little evidence of erosion of edges.

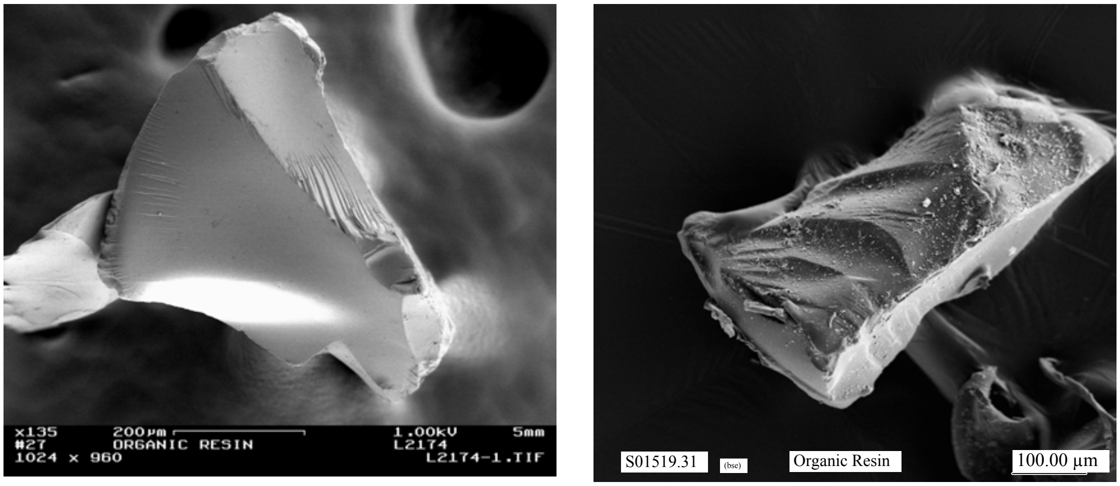

Figure 4.4. Low Magnification (x135) SEM Images of Unused (left) and Used (right) SL-644 from Batch 010319SMC-IV-73 in the Acid Form

However, at higher magnification (x2000), significant changes in the resin surface are apparent as illustrated in Figure 4.5 and the used acid-form resin appears more porous and fragmented, consistent with the reduction in bed density noted in Section 4.1.2.

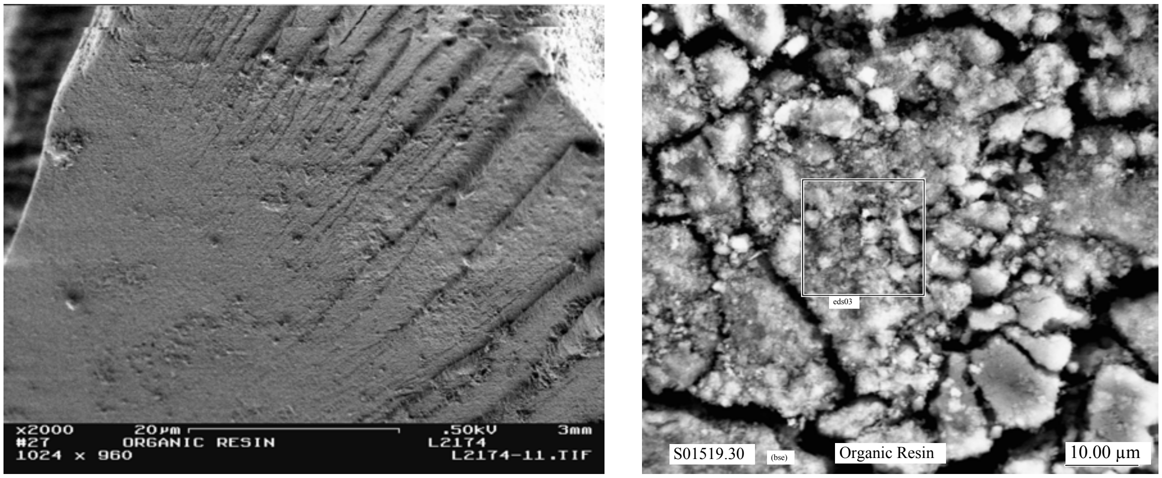

Figure 4.5. High Magnification (x2000) SEM Images of Unused (left) and Used (right) SL-644 from Batch 010319SMC-IV-73 in the Acid Form

4.2 Impact of Chemical Degradation on Process Performance

4.2.1 Breakthrough Performance

Chemical degradation would be expected to impact the Cs breakthrough performance of the resin. Figure 4.6 shows the deterioration in breakthrough performance by comparing the breakthrough profiles from Cycles 1, 5, 10, 15, 20, and 25 in which simulated LAW composition and flow rate were consistent. Note that the result from the first cycle appears to be anomalous and was probably associated with a temporary and deleterious resin characteristic that was alleviated in the subsequent elution. The bow to the Cycle-15 profile is readily apparent when compared to the profiles from other cycles.

One measure of breakthrough performance is the column-distribution coefficient. Figure 4.7 plots the column-distribution coefficients with respect to both the BV in 0.25 M NaOH and LAW, because the latter decreases with increasing cycle number, for the fully sampled cycles (5, 10, 15, 20, and 25). The figure shows that the column-distribution coefficients, λ, can be correlated with cycle number by a linear relationship. The correlation presented in Figure 4.7 for the data measured with respect to the BV in 0.25 M NaOH is

$$\lambda = 182 - 4.25n \tag{8}$$

Here, n is the cycle number. The correlation was derived by correlating the column distribution coefficients obtained from all cycles with cycle number, flow rate, and potassium and cesium concentrations (see Section 4.5.1 for more details) and then setting the latter three parameters to the values used in Cycles 5, 10, 15, 20, and 25. Only the plotted data were used to derive the correlation for the results determined with respect to the BV in simulated LAW. The difference in the intercepts between the two correlations is due to the difference in BV between the reagents. The reduction in BV in simulated LAW contrasting with the constancy of that measured in 0.25 M NaOH accounts for the difference between the gradients of the correlations.

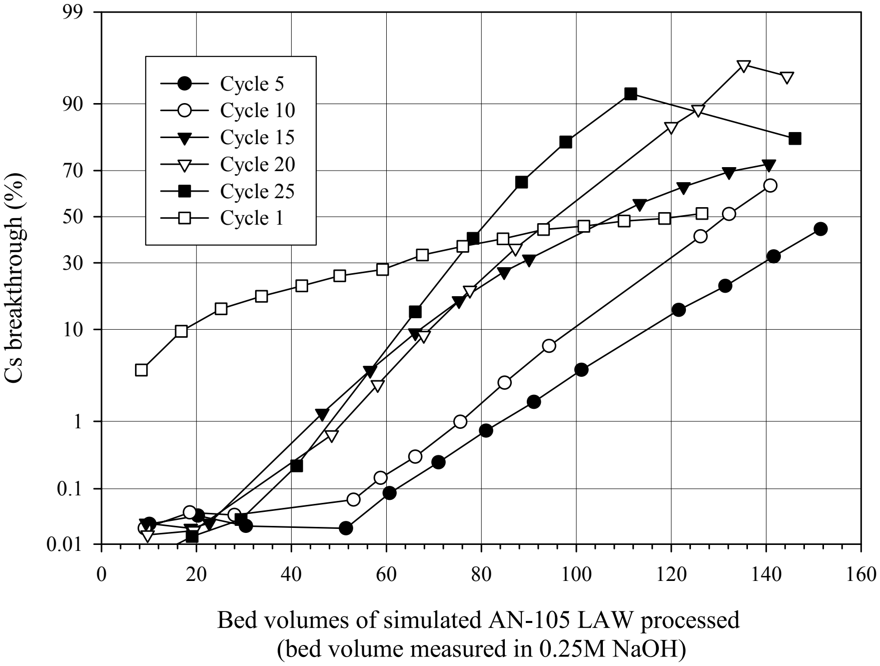

Figure 4.6. **Comparison of Breakthrough Profiles from Cycles 1, 5, 10, 15, 20, and 25 (SL-644 resin batch SMC-010319SMC-IV-73, nominal 3.0 BV/h, ~5 M Na, 1.22E-04 M Cs^+, 9.51E-2 M K^+, 1.72 M OH^-, 1.23 M NO_3^-, 1.21 M NO_2^-, ambient conditions, BV = nominal 10 mL in 0.25 M NaOH and nominal 8.0 mL in simulated AN-105 LAW)**

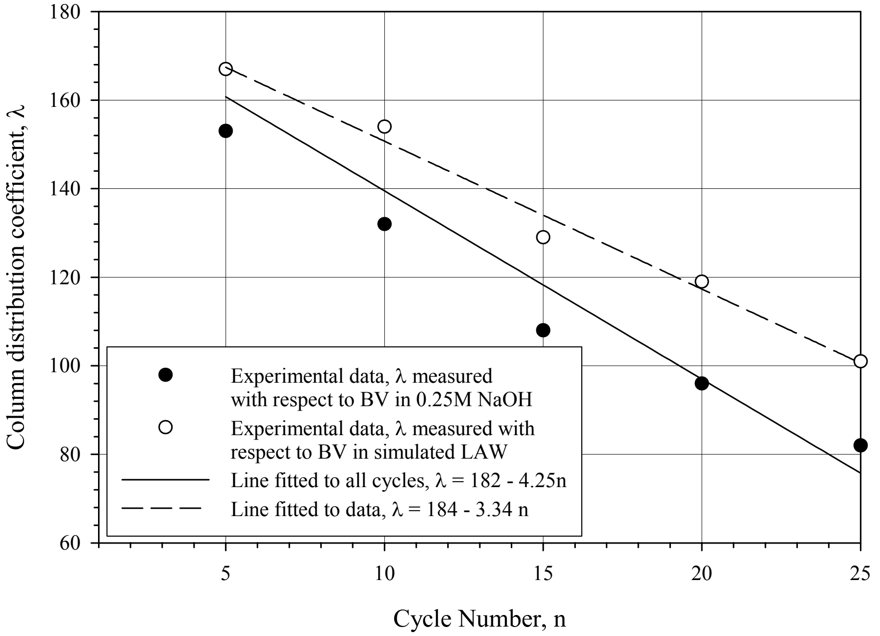

Figure 4.7. Correlation of the Column Distribution Coefficient with Cycle Number

The resin appears to lose approximately 60% of its effective capacity for Cs^+ over the course of 25 cycles, based on the volume of the bed in 0.25 M NaOH because the column-distribution coefficient is related to the resin capacity. Note that ~34% of the resin mass was also lost over this period. Therefore, ~34% of the capacity loss is associated with resin loss and ~26% with presumably chemical alteration of the ion exchange sites. Reference is made to "effective" capacity to include changes in the selectivity, accessibility, and concentration of ion exchange sites.

Changes in the capacity of the resin were also interpreted from the compositions of the eluates. Note that this method provides an underestimate of the capacity because it does not account for the metals irreversibly separated onto the resin. Figure 4.8 shows that the total metal capacity appears to remain approximately constant at ~1 meq/mL until Cycle 20 when it increases to attain a value of ~1.4 meq/mL in Cycle 25. These results suggest that the number of effective ion exchange sites increased probably due to improving accessibility as indicated by the reduction in bed density.

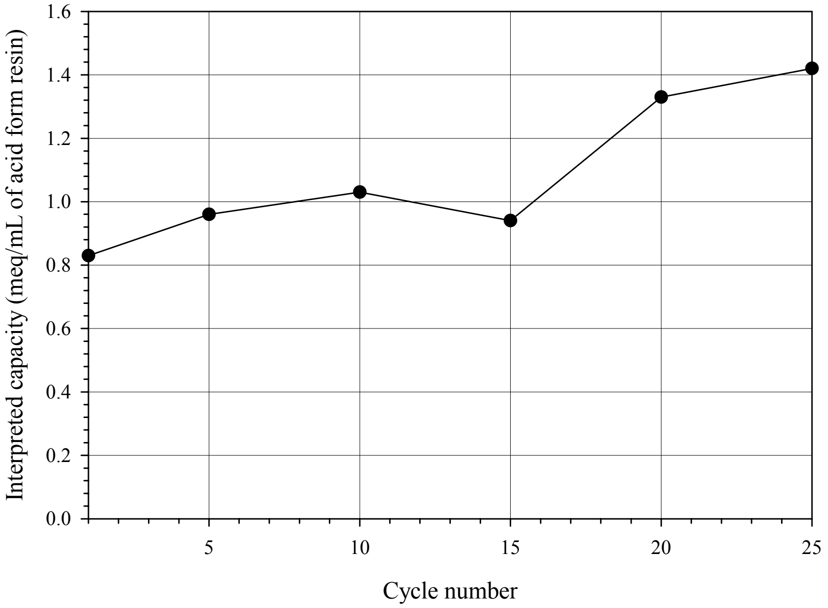

Figure 4.8. Changes in Resin Capacity Interpreted from Eluate Compositions

The results from this work were compared to those from Brown et al. (1995). Brown et al. (1995) measured the rate of reduction in the batch-equilibrium coefficient after immersing SL-644 resin in 1 M NaOH and simulated neutralized current acid waste (NCAW). The immersion was conducted in a closed vessel with an atmosphere of pure oxygen that was maintained at constant pressure. The volume of oxygen added to maintain pressure was therefore equivalent to that consumed by the resin.

The batch distribution coefficients decreased logarithmically from 1880 mL/g to 125 mL/g and 25 mL/g after 16 hours immersed in the NCAW and 1M NaOH, respectively, at an equilibrium Na^+ to Cs^+ molar ratio of 10^5. The total quantities of oxygen consumed from NCAW and 1M NaOH were 5.26 $\mu molg^{-1}$ and 6.64 $\mu molg^{-1}$, respectively. The average rates of reduction in the batch distribution coefficients were, therefore, 330 mL/mmol and 280 mL/mmol of consumed oxygen in NCAW and 1M NaOH, respectively. In comparison, the batch distribution coefficient for the resin studied here reduced from 770 mL/g (derived by dividing the initial column distribution coefficient of ~180 by the initial bed density of 0.234) to 510 mL/g (derived by dividing the cycle 25 column distribution coefficient of ~80 by the final bed density of 0.16) over the 25 cycles. The total quantity of oxygen consumed was estimated at 1.2 μmolg^{-1} from the average consumption rates derived in section 4.3 to give an average batch distribution coefficient reduction rate of 210 mL/mmol of consumed oxygen. The rate of reduction in the batch equilibrium coefficient derived for the resin studied here compares well with that from the work of Brown et al. (1995) considering the potential differences in resin batch characteristics and test methods. Indeed, the somewhat lower rate observed here was probably because the resin had already degraded by exposure to air, noting Brown et al. (1995) observed a logarithmic reduction in the batch equilibrium coefficient.

A change in ion exchange kinetics is also apparent from Figure 4.6. The onset of breakthrough occurred at ~50 BVs for Cycles 5 and 10 and then at ~20 BVs for Cycles 15, 20, and 25. The result from Cycle 1 is not considered since it appears to be anomalous as described earlier. This observation suggests that both the capacity and kinetic performance of the resin is impacted by chemical degradation. The kinetic performance of the resin appears to improve since the breakthrough profiles become increasingly steeper while 50% breakthrough is attained at progressively smaller volumes of processed LAW. The kinetic-performance improvement may be related to a reduction in the particle size from attrition and/or better accessibility to ion exchange sites as indicated by the reduction in bed density and increasing porosity of the resin noted in Section 4.1.

4.2.2 Elution Performance

Figure 4.9 shows that the eluate Cs$^+$ concentration peaked after ~4 BVs of eluate had been generated for every cycle. However, the volume of eluate attaining a concentration of Cs$^+$ in the eluate of 1% of that in the simulated LAW feed decreases with increasing cycle number. The one anomaly is Cycle 15, which required the greatest volume and which might have been associated with the anomalous breakthrough performance described in Section 3.3.11.

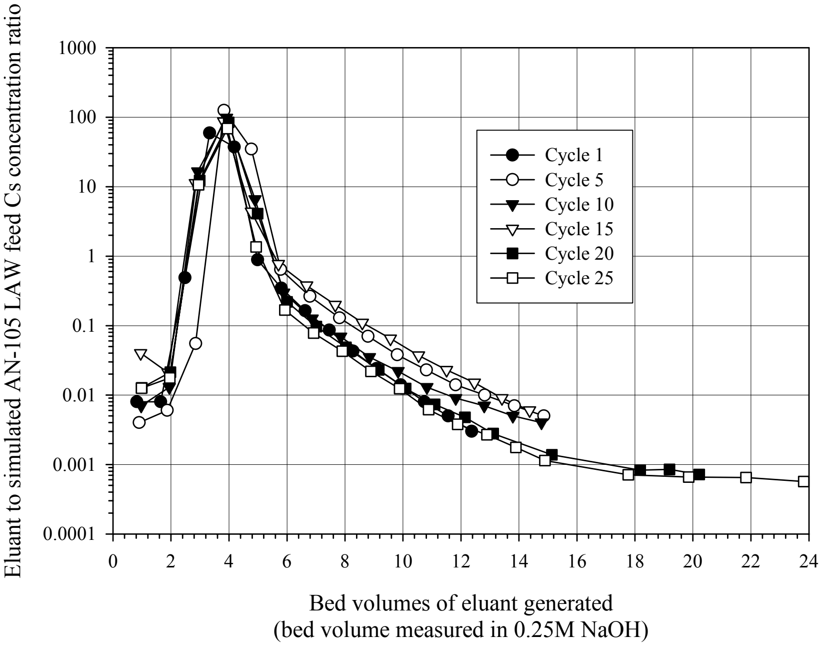

Figure 4.9. Comparison of Elution Profiles from Cycles 1, 5, 10, 15, 20, and 25 (SL-644 resin batch SMC-010319SMC-IV-73, nominal 1.0 BV/h, ambient conditions, BV = nominal 10 mL in 0.25 M NaOH)

Elution performance would be expected to depend on the quantity of Cs separated onto the resin with which the decreasing volume of eluate with increasing cycle number is consistent since the breakthrough performance progressively deteriorated. Figure 4.10 illustrates this effect and also shows that the volume of eluate required to achieve an eluate-to-LAW feed Cs^+ concentration ratio of 1% is a linear function of the quantity of Cs on the resin. The anomalous result from Cycle 15 is more clearly demonstrated and was not considered in fitting the correlation.

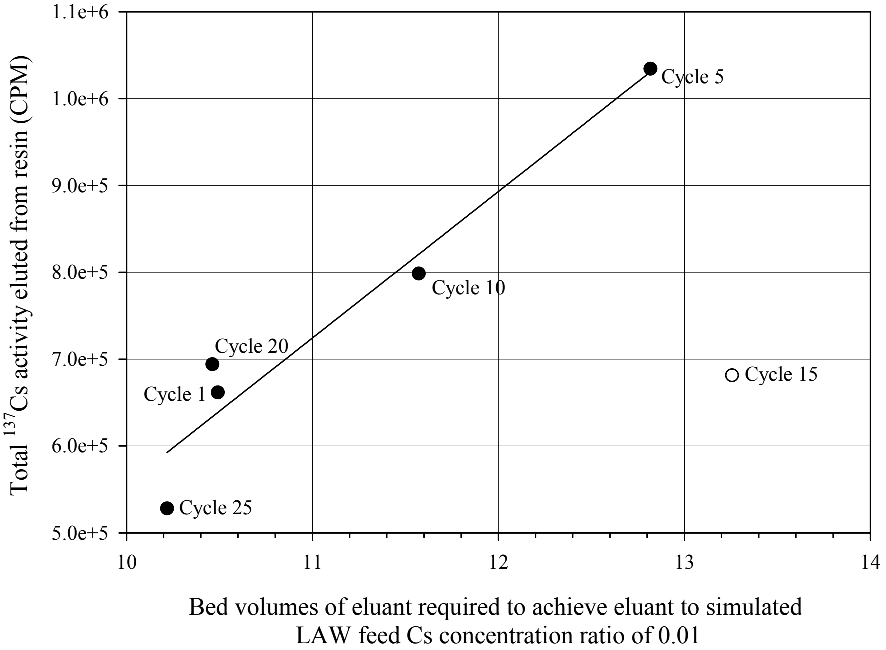

Figure 4.10. Elution Performance Correlated with the Quantity of Separated Cesium (SL-644 resin batch SMC-010319SMC-IV-73, nominal 1.0 BV/h, ambient conditions, BV = nominal 10 mL in 0.25 M NaOH)

The elution performance was also analyzed considering the cumulative fraction of eluted Cs. The cumulative fraction of eluted Cs is illustrated in Figure 4.11 on a probability scale as a function of the generated volume of eluate. Elution data presented in this way appear to enable a more straightforward comparison between cycles. In general, the elution profiles are essentially coincident when presented this way except for Cycles 5 and 15. The Cycle-5 profile appears sharper though delayed. The reason for the Cycle 5 behavior is not known but may be related to the greater quantity of Cs separated onto the resin compared to other cycles. The anomalous behavior in Cycle 15 is also apparent here, as noted before.

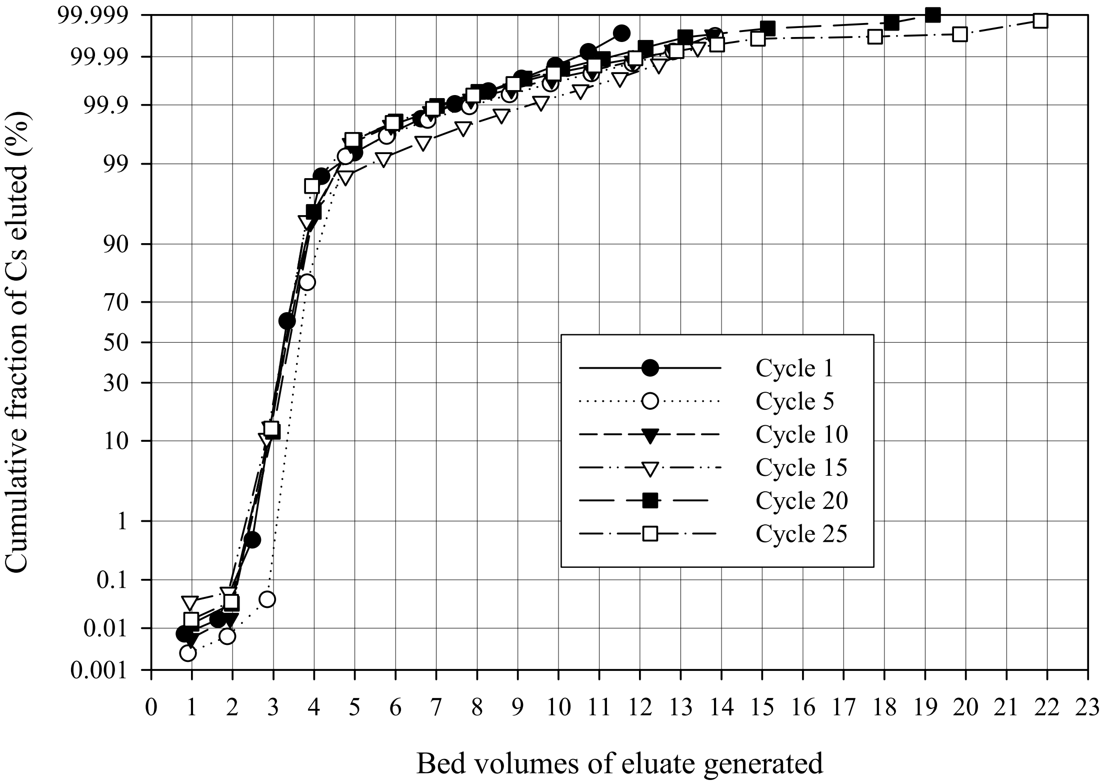

Figure 4.11. Cumulative Cs Eluted as a Function of Generated Eluate Volume (SL-644 resin batch SMC-010319SMC-IV-73, nominal 1.0 BV/h, ambient conditions, BV = nominal 10 mL in 0.25 M NaOH)

There appears to be no impact on elution performance of chemical degradation on the basis of these analyses. However, note that these analyses consider the volume of eluate in terms of the BV measured in the preceding regeneration operation. Section 4.1.2 describes how the BV during elution decreased with each cycle even though the BV during regeneration remained nearly constant and that this appeared to be associated with resin loss from the bed. Therefore, elution performance indeed deteriorates when the eluate volume is considered in terms of the BV measured during elution. Unfortunately, the data gathered on the BV during elution was limited to Cycles 2, 15, 20, and 25. However, based on the available data, Figure 4.12 clearly shows that a greater volume of eluant is required to elute the same quantity of Cs with each successive cycle. For example, 99.9% of the total Cs that eluted in Cycles 2 and 25 was eluted in 9.5 BVs and 12 BVs, respectively.

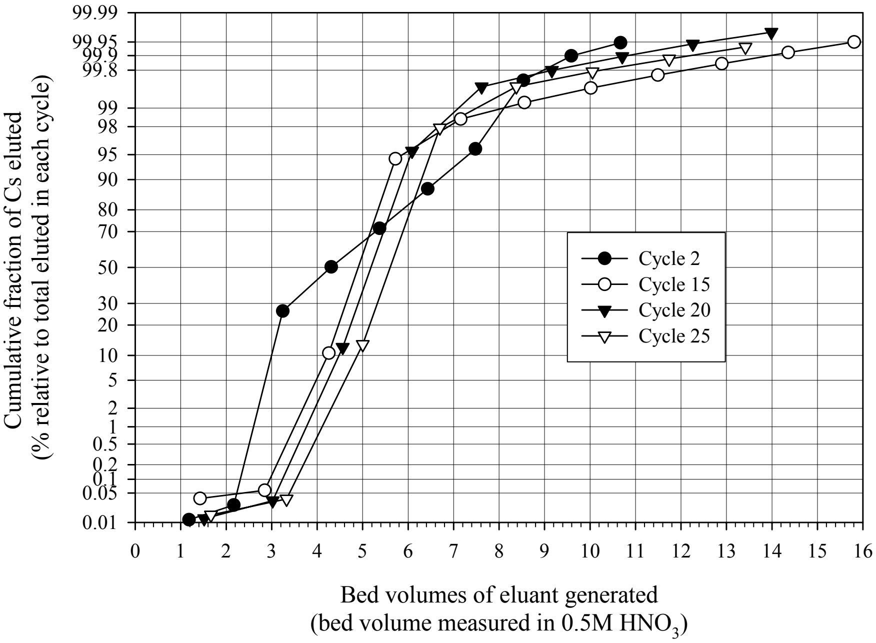

Figure 4.12. Cumulative Fraction of Cs Eluted as a Function of Generated Eluate Volume (SL-644 resin batch SMC-010319SMC-IV-73, nominal 1.0 BV/h, ambient conditions)

4.3 Resin Consumption of Dissolved Oxygen

4.3.1 General Observations

Table 4.1 compares the oxygen-consumption rates for each operation of every cycle where measured. The rates are expressed in terms of the mass of air-dried acid-form resin expected to exist for each cycle based on a linear relationship between resin mass and cycle number. In general, the rate of consumption increases with increasing oxygen solubility—increasing in the order LAW, 0.25 M NaOH, 0.1 M NaOH, 0.5 M HNO_3, and water—as expected since mass transfer and chemical reaction rates would increase with increasing feed concentration. This observation is consistent with the work of Brown et al. (1995) who observed that the rate of oxygen consumption from a simulated neutralized current acid waste LAW was lower than from 1 M NaOH.

4.3.2 Regeneration

The effluent oxygen-concentration profiles consistently indicate an initial sharp increase in the rate of oxygen consumption that is probably associated with conversion of the resin from the acid to the Na form. This hypothesis is consistent with observations from previous work, e.g. Steimke et al. (2001), that indicate that the SL-644 resin is more prone to oxidation in the Na form. It is also consistent with the observed rates derived for the elution and elution-rinse operations below. The reason for the spike in the effluent oxygen concentration just before LAW processing is unknown.

In general, the oxygen-consumption rate was consistent for Cycles 13 and higher, an average of 0.84 $\mu molg^{-1}h^{-1}$ with a standard deviation of 0.11 $\mu molg^{-1}h^{-1}$ (±13%). However, the consumption rate in the first cycle was 1.8 times higher than the average of the remaining cycles.

Table 4.1. Comparison of Oxygen-Uptake Rates (Rate recorded per unit mass of resin in acid form)

	Regeneration		LAW Processing		Feed Displacement		Water Rinse		Elution		Elution Rinse	
Cycle	Flow rate (BV/h)	O_2 consumption rate ($\mu molg^{-1}h^{-1}$)	Flow rate (BV/h)	O_2 consumption rate ($\mu molg^{-1}h^{-1}$)	Flow rate (BV/h)	O_2 consumption rate ($\mu molg^{-1}h^{-1}$)	Flow rate (BV/h)	O_2 consumption rate ($\mu molg^{-1}h^{-1}$)	Flow rate (BV/h)	O_2 consumption rate ($\mu molg^{-1}h^{-1}$) (1)	Flow rate (BV/h)	O_2 consumption rate ($\mu molg^{-1}h^{-1}$)
1	0.8	1.5	2.5	0.30	2.9	1.2	2.5	1.9	0.8	1.4 / 1.9	2.4	1.9
13	1.0	0.76	2.9	0.22	3.3	0.68	2.9	1.9	1.0	1.7 / 1.3	2.9	0.82
14	1.0	0.78	1.4	0.075	3.3	0.45	2.9	0.52	1.9 / 1.0	0.27 / 0.78	3.1	2.5
15	0.9	0.73	2.8	0.18	3.2	Not measured	2.9	Not measured	1.0	Not measured	2.8	Not measured
21	1.0	1.0	2.9	0.40	3.4	3.1	2.8	5.5	1.0	0.96 / 0.58	2.9	1.0
22	0.9	0.93	5.9	1.0	3.3	4.1	2.8	4.9	0.9	0.99 / 0.79	2.9	2.0
25	1.1	0.85	3.0	0.48	3.4	4.7	3.0	5.5	1.0	1.2 / 0.70	3.0	2.3

Note. 1. The first rate refers to the first 10 hours of elution while the second rate refers to the remaining elution period.

4.3.3 LAW Processing

The oxygen-consumption rate appears to be dependent on the flow rate. For example, the average consumption rate was 0.32 μmolg^{-1}h^{-1} with a standard deviation of 0.12 μmolg^{-1}h^{-1} (±39%) for those operations in which the flow rate was between 2.5 and 3.0 BV/h. The consumption rate appeared to disproportionately increase by a factor of between 3 and 5 when the flow rate was doubled. The reason for this observation is not known at present.

4.3.4 Feed Displacement

The average consumption rate is 2.4 μmolg^{-1}h^{-1}, but the standard deviation of 1.8 μmolg^{-1}h^{-1} indicates the significant scatter in the data. This is possibly associated with simulated LAW back-mixing and the short processing time leading to fewer measurements for this operation compared to regeneration or LAW processing.

4.3.5 Water Rinse

As for feed displacement, the relatively short processing time and feed displacement solution back-mixing appear to introduce significant scatter, giving a standard deviation of 3.4 μmolg^{-1}h^{-1} for a mean of 2.2 μmolg^{-1}h^{-1}.

4.3.6 Elution

Analysis for this operation is complicated by the fact that the consumption rates varied during the processing time. Table 4.1 gives the rates for the first 10 h and then the remaining period of elution.

The consumption rates in Cycles 1 and 13 during the period from 10 hours until termination were significantly higher than in the other cycles. In addition, in contrast to other cycles where the flow rate remained constant, the consumption rate increased during the course of elution in Cycle 1. The higher rates in Cycle 1 may be associated with changes in the resin that led to the improvement in breakthrough performance. The similarity between the initial rates of Cycles 1 and 13 may indicate a continuation of a trend from Cycle 1. Indeed, the consumption rate during the latter part of elution may decrease with each cycle (the lack of data limits conclusiveness) up to approximately Cycle 15 when a constant rate was apparent. This trend would then correlate with that in the degree of resin contraction observed during the elution operation of successive cycles (Section 4.1.2).

The consumption rates during the later parts of Cycles 14, 21, 22, and 25 were consistent, giving an average of 0.71 μmolg^{-1}h^{-1} and a standard deviation of 0.10 μmolg^{-1}h^{-1}. For the first halves of Cycles 21, 22, and 25, consumption rates were also consistent, providing an average rate of 1.1 μmolg^{-1}h^{-1} with a standard deviation of 0.13 μmolg^{-1}h^{-1}. Eluate was generated at double the baseline rate during the first half of Cycle 14 elution, and an oxygen consumption rate significantly lower than elsewhere at 0.27 μmolg^{-1}h^{-1} was observed, presumably due to the shorter residence time of solution in the bed.

The rate of oxygen consumption probably decreased during elution as a result of conversion of the resin from the Na to acid form. The acid form has appeared to be more stable to oxidation than the Na form. However, stability may be more associated with the relative BV since the trend in the rate of consumption appears to correlate with that in the degree of contraction.

4.3.7 Elution Rinse

For this operation, the mean consumption rate was 1.8 μmolg^{-1}h^{-1} with a standard deviation of 0.69 μmolg^{-1}h^{-1}. The short processing time and eluate back-mixing would again introduce greater uncertainty. The reason for the difference in the rates between the water rinse and elution rinse is probably associated with the form of the resin (the higher rate associated with the Na form) consistent with the observations recorded for the regeneration and elution operations.

4.4 Comparison of Column and Equilibrium Performance

Cycles 4 and 6 through 9 processed simulated LAW with progressively increasing concentrations of Cs, and column-distribution coefficients were determined. The rate of deterioration of the column-distribution coefficient with cycle number was assumed the same as derived in Section 4.5.1 to derive the column-distribution coefficients at the same Na^+-to-Cs^+ molar ratios expected with fresh resin. The column-distribution coefficients were also converted into acid-form resin BV terms based on the average Na (in 0.25 M NaOH) to H (in DI water) form resin BV ratios from Cycles 5 and 10, as provided in Section 4.1.2. The normalized column distribution coefficients (based on the acid form bed volume) are plotted in Figure 4.13 as a function of the Na^+-to-Cs^+ molar ratio and are correlated by the equation

$$\lambda = 96 Ln\left(\frac{[Na]}{[Cs]}\right) - 744 \tag{9}$$

This correlation is the same form as that used to correlate the batch-contact data, also plotted in Figure 4.13. The ratio of the column-distribution coefficients to the batch-contact data is approximately 0.15 g/mL, which is theoretically the dry-bed density of the acid-form resin. However, the acid-form dry-bed density remained nearly constant at 0.20 g/mL as presented in Section 4.1.2. Also plotted in Figure 4.13 is the curve derived from dividing the column-distribution coefficient correlation by the experimentally determined acid-form resin-bed density. The latter curve provides an excellent fit to the batch-equilibrium data at the four lowest $[Na^+]$ to $[Cs^+]$ ratios. This observation appears to suggest that batch-equilibrium data can be used to predict column performance but only over limited $[Na^+]$-to-$[Cs^+]$ ratio ranges.

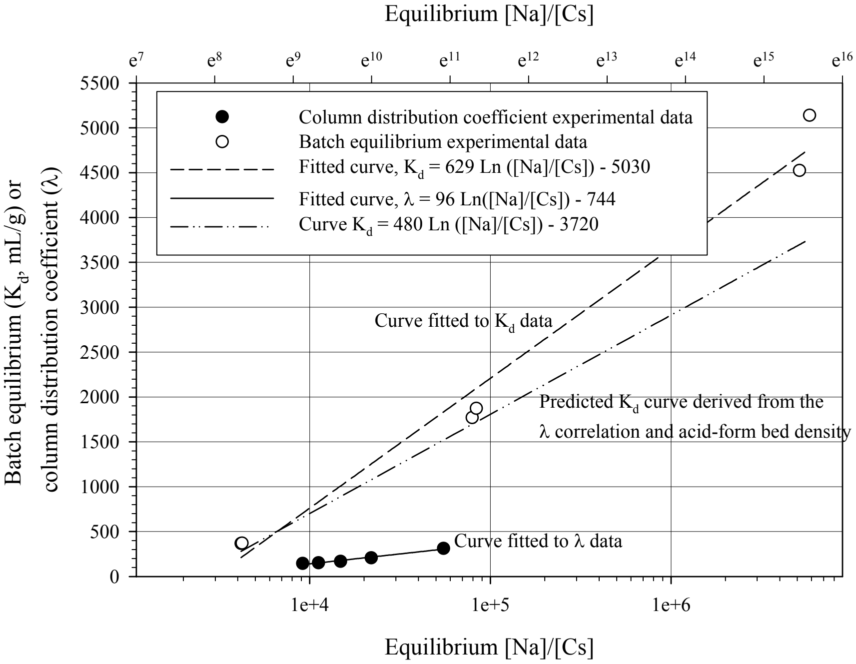

Figure 4.13. Comparison of Column and Batch-Equilibrium Performance (SL-644 resin batch SMC-010319SMC-IV-73, nominal 3.0 BV/h, ~5 M Na, 95.1 mM K^+, 1.72 M OH^-, 1.23 M NO_3^-, 1.21 M NO_2^-, ambient conditions, BV = nominal 10 mL in 0.25 M NaOH and nominal 8.0 mL in simulated AN-105 LAW)

4.5 Analysis of Results from Parametric Study

4.5.1 Breakthrough Performance

While baseline simulated LAW flow rate and concentrations of cesium, potassium, and hydroxide were used in Cycles 1, 5, 10, 15, 20, and 25, the remaining cycles processed simulated LAW over a range of values. The purpose of doing so was to investigate the relative effects of these parameters on breakthrough performance, as measured by the column-distribution coefficient. The values of each parameter and the resulting column-distribution coefficient were entered into a statistical software package to facilitate analysis. The detailed results of the statistical analysis are provided in Appendix B.

Cycle number, Cs^+ concentration and flow rate were recognized as significant in affecting the column distribution coefficient when each parameter was considered independently with a linear impact on the column distribution coefficient. The Cs^+ concentration and cycle number were identified as the most significant parameters followed by flow rate. The K^+ and OH^- concentrations were not significant over the ranges investigated. The column distribution coefficient decreases as the significant parameters are increased. Increasing Cs^+ and K^+ concentrations are known to decrease the batch-distribution coefficient of the resin for Cs^+ and thereby decrease the column distribution coefficient. The column distribution coefficient decreases with increasing flow rate presumably due to a significant diffusional resistance in the mass-transfer films surrounding the resin particles.

The data were fitted to the correlation,

$$\lambda = 226 - 4.79n - 2.04[Cs] - 13.9F \tag{10}$$

where

λ = column-distribution coefficient

n = cycle number (applicable range is 1 to 25)

$[Cs^+]$ = Cs^+ concentration, ppm (applicable range is 7 ppm to 60 ppm)

F = simulated LAW flow rate, BV/h (applicable range is 1.5 BV/h to 6 BV/h, except when $[Cs^+]$>25 ppm, when F = 3 BV/h.

The root mean square error of this correlation is 28.1 so that the 95% confidence interval is approximately 56 for the column distribution coefficient.

Further analysis was performed considering binary parameter interactions and quadratic dependencies. All parameters, except the OH^- concentration, were found to have a significant impact on the column distribution coefficient. The data were fitted to the correlation,

$$\begin{aligned}\lambda = 350 - 4.25n - 6.58[Cs] + 0.058[Cs]^2 + 105[K] - 215[K]^2 \\ - 58.4F + 8.06F^2 + 6.56[Cs][K] - 21.0[K]F\end{aligned} \tag{11}$$

The applicable range for $[K^+]$ in equation 10 is 0.008 M to 0.8 M, except when $[Cs^+]$ > 25 ppm, when $[K^+]$ = 0.095M. The root mean square error of this correlation is 9.4 so that the 95% confidence interval is approximately 19 for the column distribution coefficient. The good statistical fit of the data appears to show that all significant parameters are accounted for in the correlation and that LAW channeling was not a significant issue after Cycle 1. Note that the correlation appears to predict that the column-distribution coefficient decreases and then increases as each parameter increases, which is not physically reasonable. For example, for a K^+ concentration of 0.8 M, the column-distribution coefficient increases for Cs^+ concentrations above 23 ppm. The limitations of the test must then be considered. In the example, no simulated LAW was ever processed with Cs^+ and K^+ concentrations simultaneously exceeding 25 ppm and 0.095 M, respectively.

A physical basis for the form of the correlation, at least for the analytes, was investigated by considering the mathematical description of the Langmuir adsorption mechanism,

$$[M]_r = \frac{K[M]_s}{1 + K[M]_s} \tag{12}$$

where $[M]_r$ and $[M]_s$ are the metal concentrations in the resin and solution, respectively, and K^+ is a constant. The column-distribution coefficient is proportional to the batch-distribution coefficient, which is the ratio of metal concentrations in the resin and solution. Therefore,

$$\lambda \propto K_d = \frac{[M]_r}{[M]_s} = \frac{K}{1 + K[M]_s} \tag{13}$$

The Taylor expansion of this relationship about a metal concentration M up to the quadratic term, is

$$\frac{K}{1+K[M]_s} \approx \frac{1}{1+M} - \frac{[M]_s - M}{(1+M)^2} + \frac{([M]_s - M)^2}{2(1+M)^3} \tag{14}$$

Hence, the quadratic form of the correlation has a physical basis in the Langmuir-type adsorption mechanism.

4.5.2 Elution Performance

The elution operation of Cycle 14 was performed at a flow rate of 1.9 BV/h, or double the baseline rate, to investigate the impact of eluant flow rate on elution performance. Figure 4.14 compares the elution profiles in terms of the cumulative quantity of Cs eluted for Cycles 10, 14, and 15. The comparison appears to show that the eluant flow rate does not significantly impact the elution performance since the profiles are nearly coincident.

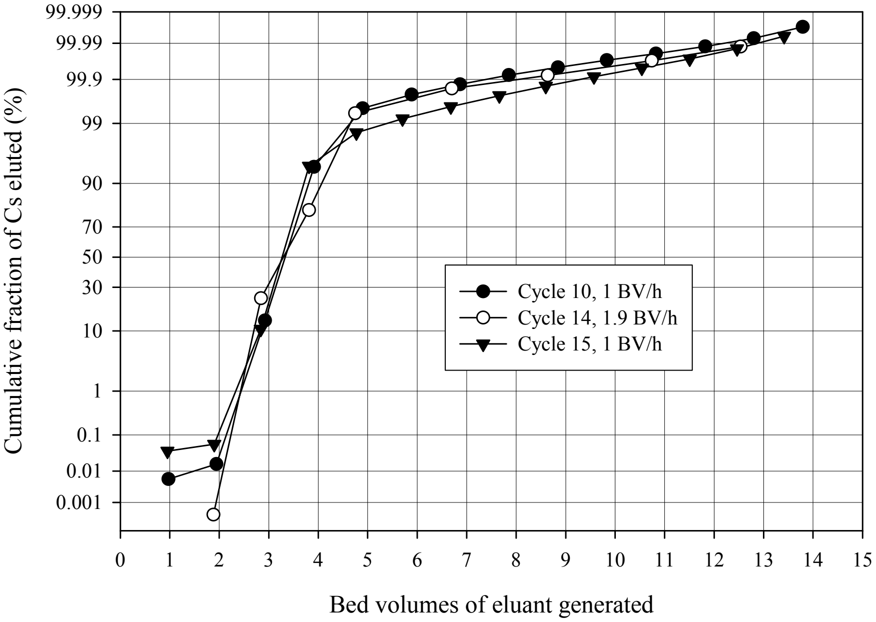

Figure 4.14. Comparison of Elution Profiles at 1 BV/h and 1.9 BV/h (SL-644 resin batch SMC-010319SMC-IV-73, ambient conditions, BV = nominal 10 mL in 0.25 M NaOH)

5.0 Implications for WTP Design and Operations

This section discusses the implications for the WTP design and operations of test observations and results.

5.1 Implications for WTP Design and Operations from Test Observations

Top of resin bed observed to pull away from column walls during LAW processing and elution.
This observation appears to indicate that adhesive forces, manifested during resin shrinkage, causing the bed to pull away from the column walls are stronger than the gravitational forces that would tend to make the bed settle to fill the entire column cross-section. The bottom of the bed fills the column cross-section, presumably because the weight of the resin is sufficient to overcome the adhesive forces. The phenomenon causes the feed LAW or eluant to channel through the bed and reduce its breakthrough or elution performance to some extent. If it were limited to the bed only pulling away from the walls, then the impact may be negligible in the full-scale column because the cross-section is much larger and the resin weight would act to preclude the phenomenon. However, fracturing throughout the cross-section of the bed may occur in a large column, and this would exacerbate the channeling problem.

Gas generation during regeneration observed after Cycle 15, causing gas bubbles to form within the bed and entrainment in the effluent.
This observation suggests that the WTP design should accommodate the entrainment of gas bubbles in the effluent from the column and their subsequent separation. Gas bubbles retained in the bed during LAW processing and elution would also reduce the breakthrough and elution performance of the bed. Fluidization of the bed following regeneration may be required to remove bubbles and maintain the performance of the bed. The composition of the gas is not known. Consideration should be given to accommodating the generation and entrainment of gas bubbles in the WTP column design and associated safety analyses, especially considering the more intense phenomenon observed in Cycle 22.

5.2 Implications for WTP Design and Operations from Test Results

5.2.1 Resin Life and Disposal

Average resin mass loss rate of ~1.36% per cycle.
This phenomenon might allow fresh resin to be periodically added to a partially full column providing it is sized to accommodate the reduction in the density of the used, Na-form resin.

The resin consumes less oxygen in the contracted, acid-form at constant dissolved oxygen concentration.
This observation suggests that the length of time the resin spends in the expanded, sodium-form and exposed to oxygen should be minimized. This is applicable to both resin storage before it is used and during normal and off-normal operations. The resin should be in the acid form immersed in water containing no dissolved oxygen to minimize degradation when it is not being used.

Column-distribution coefficient reduces by ~4 BVs (2.3%) every cycle.
This result was used to derive a preliminary value for the service life of the resin and investigate means to extend it. Interpretation of the result for a 3-column system is difficult because a bed in the WTP would process LAW in the polishing, lag, and then lead positions before it is eluted and regenerated. The correlation between column distribution coefficient and cycle n (Equation 8) can be re-written taking account of different initial column distribution coefficients and variable LAW processing

periods using the oxygen consumption rates derived in Section 4.3 assuming degradation is solely due to resin oxidation. The generalized correlation between the column distribution coefficient and cycle number is then

$$\lambda = \frac{40.5\lambda_0 - 110.5n}{40.5 + 0.411n} \tag{15}$$

Here, λ_0 is the initial column distribution coefficient. On this basis of Equation 15, the ion exchange bed would require replacement after completing the 14th cycle, consistent with the design assumption of Olson (2001). The following assumptions were used in deriving this result.

- Olson (2001) defines a cycle for the bed in the polishing, lag, and lead positions, normally processing 100 BVs of Envelope A or C LAW at 3 BV/h in each position and undergoing elution and regeneration after processing LAW in the lead position. The normal volume of LAW processed of 100 BVs is assumed to be an average over the 14 cycles with Cycle 7 processing 100 BVs of LAW assuming sufficient lag storage is available. The first cycle processes 126 BVs of LAW.
- The entire elution operation normally requires approximately 25 h to complete, consistent with Olson (2001). This discussion assumes that a bed is replaced when the loading duration becomes less than the elution duration to maintain overall throughput. Therefore, the bed is replaced when the column-distribution coefficient reduces to 75, given a LAW processing rate of 3 BV/h.
- The lead bed is eluted upon 50% breakthrough from the lead column although in actual operation, elution of the lead column would be initiated when breakthrough from the polishing column becomes too high to maintain product acceptability.
- Reaction with dissolved oxygen is the only significant resin degradation mechanism. Therefore, no degradation is assumed when the resin is in the lag or polishing positions since the resin in the lead position consumes most of the dissolved oxygen. In addition, insignificant dissolution of oxygen from the pressurized air sweep of the column head spaces into the column feeds is assumed. No degradation arising from irradiation is included.

Removing the oxygen dissolved in the elution and regeneration reagents could be accomplished by sparging the feedstocks with nitrogen and would extend the resin life. For example, if oxygen is completely removed from the elution and regeneration reagents and assuming that degradation is split between the operations according to the relative amounts of consumed oxygen described in Section 4.3, then the resin would require replacement after the 68th design cycle, and the resin service life would be extended by approximately 5 times. Here, 100 BVs of LAW are processed in the 26th cycle for the initial column distribution coefficient of 126 and the average number of BVs processed is 94.

Furthermore, approximately half of the deterioration in breakthrough performance is due to resin loss. If fresh resin were added to the column to maintain the acid-form BV then the bed-replacement frequency would be halved, assuming that the mechanisms leading to resin capacity and mass losses are independent. The density of the resin reduces in the Na form and so the column would need to be sized to accommodate the additional volume of Na-form resin.

The analysis presented above is conservative since the column-distribution coefficient is typically higher than 100. For example, Fiskum et al. (2002) and Kurath et al. (2001a) measured column distributions of 300 and 143, processing simulated and actual AW-101 LAW, respectively. Note that Fiskum et al. (2002) used resin from the same batch used for this work but shortly after receipt. If the initial column distribution coefficient of 300 were assumed, then resin replacement would be required after the 64th cycle.

The modified TCLP on the spent resin indicates that it may not exhibit toxicity characteristics.
This result suggests that the spent resin could be planned for disposal as a non-toxic waste. However, this result may not be appropriate for regulatory purposes or submissions since the TCLP had to be modified from the standard EPA SW-846 method to account for the small sample size.

Residual 1.71 μg/g Cs^+ found on the spent resin.
Chemical analysis of the leached acid-form resin and TCLP leachate showed the spent resin to have contained Cs^+ at a concentration of 1.71 μg/g of resin air-dried at room temperature (0.375 g/m^3) after it had been eluted with 24 BVs of 0.5M nitric acid in Cycle 25. If the resin had been processing waste containing Cs of which a quarter was ^{137}Cs, the ratio found by Kurath et al. (2000a) and Kurath et al. (2000b) for Envelope A AW-101 and Envelope C AN-107 LAW samples, then the residual activity would have been 37 μCi/g (8.1 Ci/m^3). Hassan et al. (2001) found that the fraction of ^{137}Cs in the total Cs in an Envelope B AZ-102 LAW sample was a third. If this value were assumed, then the residual ^{137}Cs activity would have been 50 μCi/g (11 Ci/m^3). These concentrations are factors of >1.4 higher than that of the 26 μCi/g[1] found by Kurath and Wagner (2000) on resin previously used to process actual AW-101 and AN-107 LAW samples. In addition, these values are between the Hanford Site Solid Waste Acceptance Criteria (McDowell 2002) category 1 and 3 limits (5.5×10^{-3} Ci/m^3 and 12,000 Ci/m^3, respectively) indicating that the spent resin from WTP would have to be dispositioned in high integrity containers.

5.2.2 Breakthrough Performance

Column breakthrough performance diminishes with increasing LAW flow rate and Cs^+ and K^+ concentrations.
The most important compositional factors impacting breakthrough performance are the Cs^+ and K^+ concentrations; there appears to be no impact of hydroxide concentration over the range studied (1.2M to 2.2M). Increasing concentrations of Cs^+ and K^+ decrease the column-distribution coefficient. Increasing the LAW flow rate also decreases the column distribution coefficient, indicating that film diffusion is significant in the ion exchange process.

5.2.3 Elution Performance

Approximately 1% increase in the volume of eluate required every cycle per unit acid-form BV.
This observation would have become an issue if the resin had not dissolved since the volume of eluant, based on the Na-form BV, required in the tests did not increase to recover the same proportion of Cs on the resin. However, the volume of eluant, based on the acid-form BV, required to achieve the same recovery of Cs increased by 10% after 10 test cycles since the acid-form BV decreased as a result of resin dissolution. Thus, the volume of eluant would increase by ~1% every cycle if the resin remains stable.

On the basis of this work, however, there is no impact on the elution volume from chemical degradation because resin loss compensates for the deterioration in performance (i.e., the elution volume remains constant if the first cycle BV is used as a basis for determining elution requirements).

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[1] The value of 21.3 μCi/g of resin dried at 95°C and including interstitial solution actually reported by Kurath and Wagner (2000) is based on the sodium form resin from a different batch than used here. The value of 26 μCi/g is based on the expected equivalent acid form resin derived by applying the sodium to acid form resin mass conversion factor determined for the resin batch used for this study and presented in Section 2.3.

No significant impact of doubling the eluant flow rate from 1 BV/h to 2 BV/h on eluant volume.

The eluant flow rate can apparently be doubled without any deleterious impact on the volume of eluate generated. This observation may be important if the volume of eluant needs to increase to achieve greater Cs recoveries than currently accommodated in the design without having to double the elution time.

6.0 Conclusions

The following conclusions from this work are categorized for clarity.

Physical Characteristics of Resin

- Resin dissolved at an average rate of 1.36 wt% per cycle so that the total mass loss from the bed after 25 cycles was 34%. The acid-form resin BV also decreased at the same rate. However, the fully expanded BV in 0.25 M NaOH remained constant such that the bed density also decreased by 34%. These observations were confirmed by SEM examination of the acid form resin that showed the used resin to have apparently higher porosity than the fresh material. Some resin at least appeared to dissolve in the 0.25 M NaOH regeneration solution since the regeneration effluents of every cycle were observed to be colored deep red-brown, presumably due to resin dissolution, and some contained resin fines that settled after ~1 day.

Process Performance of Resin

- The tested resin appeared to lose approximately 60% of its effective capacity for Cs^+ over the course of 25 cycles, based on the volume of the bed in 0.25 M NaOH. Note that ~34% of the resin mass was also lost over this period, and the 26% reduction in effective capacity accounting for the mass loss is presumably due to chemical alteration of the ion exchange sites.
- Breakthrough performance deteriorated with increasing concentrations of Cs^+ (0.066 mM to 0.938 mM) and K (0.008M to 0.8M), consistent with a Langmuir-type adsorption model. There was no significant impact of OH^- over the range studied of 1.2M to 2.2M.
- Breakthrough performance improved with decreasing flow rate over the range 6 BV/h to 1.5 BV/h, indicating that the ion exchange process is controlled by diffusion in the mass-transfer film around the resin particles.
- Greater volumes of eluant are required to elute the same volume of acid-form resin with each successive cycle, although this phenomenon was totally masked by the resin mass loss (i.e., the elution volume remained constant on the basis of the first cycle BV).
- Doubling the eluant flow rate from 1 BV/h to 2 BV/h appeared to have no significant impact on the required volume of eluant.
- Column distribution coefficients may be accurately predicted from the product of the bed density and batch-equilibrium coefficient. However, batch-contact data must be interpolated with care since the relationship between the batch contact coefficient and Cs^+ concentration appears to be more complicated than the exponential form commonly assumed.

Design and Operation Implications for the WTP

- Preliminary analysis of the results presented in this report show that the ion exchange bed would require replacement after completing the 14th cycle to maintain plant throughput, assuming that degradation is due only to reaction with dissolved oxygen and insignificant dissolution of oxygen from the pressurized air sweep of the column head spaces into the column feeds. In the WTP system description for Cs ion exchange, Olson (2001) assumes replacement after every 10th cycle and so is consistent with the preliminary value derived from this work.
- Resin life can be improved by minimizing the time the resin spends in the expanded, sodium form and its exposure to dissolved oxygen. For example, assuming degradation is due only to reaction

with dissolved oxygen, an approximately five-fold improvement in the resin life may be realized by removing the dissolved oxygen in the elution and regeneration reagents. This could be accomplished by sparging the feedstocks with nitrogen.

- The modified TCLP indicates that the spent resin would not exhibit toxicity characteristics if a formal TCLP were performed. However, this result may not be appropriate for regulatory purposes or submissions since the TCLP had to be modified from the standard EPA SW-846 method to account for the small sample size.
- Chemical analysis of the leached acid-form resin and TCLP leachate showed the spent resin to have contained Cs^+ at a concentration of 1.71 μg/g of resin air-dried at room temperature (0.375 g/m^3) after eluting with 24 BVs of 0.5M nitric acid in Cycle 25. If the resin had been processing waste containing Cs of which a quarter was ^{137}Cs, which is typical for Envelope A or C LAW, then the residual activity would have been 37 μCi/g (8.1 Ci/m^3). If a value typical of Envelope B LAW of a third were assumed, then the residual ^{137}Cs activity would have been 50 μCi/g (11 Ci/m^3). These concentrations are factors of >1.4 higher than that of the 26 μCi/g[1] found by Kurath and Wagner (2000) on resin previously used to process actual AW-101 and AN-107 LAW samples. In addition, these values are between the Hanford Site Solid Waste Acceptance Criteria (McDowell 2002) category 1 and 3 limits (5.5×10^{-3} Ci/m^3 and 12,000 Ci/m^3, respectively) indicating that the spent resin from WTP would have to be dispositioned in high integrity containers.

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[1] The value of 21.3 μCi/g of resin dried at 95°C and including interstitial liquid actually reported by Kurath and Wagner (2000) is based on the sodium form resin from a different batch than used here. The value of 26 μCi/g is based on the expected equivalent acid form resin derived by applying the sodium to acid form resin mass conversion factor determined for the resin batch used for this study and presented in Section 2.3.

7.0 References

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Appendix A: Cycle Process Data Sheets

CYCLE 1 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	322.3	g				
Feed density	1.250	g/mL				
Bed height in 0.25M NaOH	6.7	cm				
Bed volume in 0.25M NaOH	11.9	mL				
Cesium concentration	1.22E-04	M				
Potassium concentration	9.51E-02	M				
Hydroxide concentration	1.72	M				
Nitrate concentration	1.23	M				
Nitrite concentration	1.21	M				
Flow rate	30.0	mL/h, or	2.5	BV/h		
SVOA sample mass	29.426	g, taken from	8/22/01 11:50	to	8/22/01 12:35	
VOA sample mass	30.397	g, taken from	8/22/01 14:12	to	8/22/01 14:58	

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/g)
Feed 1	17721	300	6.350	558
Feed 2	17548	300	6.343	553
Feed 3	18005	300	6.350	567
Feed 4	17636	300	6.340	556
Feed 5	18209	300	6.350	574
Average	17824	300	6.347	562

Effluent bottle mass (g)	Mass of feed in bottle & VOA/SVOA (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/g)	C/C0 (%)	Total activity in sample (CPM)
440.7	118.4	8/20/01 13:00	8/20/01 16:20	28.4	99.7	8.4	2818	1200	6.174	4.939	23	4.1	141
559.3	237.0	8/20/01 16:30	8/20/01 19:40	30.0	199.5	16.8	6756	1200	6.254	5.003	54	9.6	338
677.6	355.3	8/20/01 19:50	8/20/01 23:00	29.9	299.1	25.2	10088	1200	6.103	4.882	83	14.7	504
797.6	475.3	8/20/01 23:10	8/21/01 2:20	30.3	400.1	33.7	12888	1200	6.314	5.051	102	18.2	644
916.2	593.9	8/21/01 2:30	8/21/01 5:40	30.0	500.3	42.2	11940	900	6.598	5.278	121	21.5	796
1029.4	707.1	8/21/01 5:50	8/21/01 8:50	30.2	595.9	50.2	13344	900	6.347	5.078	140	25.0	890
1155.1	832.8	8/21/01 9:00	8/21/01 12:21	30.0	701.8	59.2	15223	900	6.635	5.308	153	27.2	1015
1273.8	951.5	8/21/01 12:31	8/21/01 15:40	30.1	801.8	67.6	17531	900	6.307	5.046	185	33.0	1169
1393.4	1071.1	8/21/01 15:50	8/21/01 19:00	30.2	902.5	76.1	19579	900	6.345	5.076	206	36.6	1305
1513.3	1191.0	8/21/01 19:10	8/21/01 22:20	30.3	1003.5	84.6	21133	900	6.299	5.039	224	39.8	1409
1633.3	1311.0	8/21/01 22:30	8/22/01 1:40	30.3	1104.1	93.1	21100	900	5.702	4.562	247	43.9	1407
1752.9	1430.6	8/22/01 1:50	8/22/01 5:00	30.2	1204.8	101.6	24182	900	6.315	5.052	255	45.5	1612
1872.0	1549.7	8/22/01 5:10	8/22/01 8:20	30.1	1305.2	110.1	17087	600	6.370	5.096	268	47.8	1709
1991.7	1669.4	8/22/01 8:30	8/22/01 11:40	30.2	1406.2	118.6	18038	600	6.564	5.251	275	48.9	1804
2043.1	1780.6	8/22/01 11:50	8/22/01 14:58	28.4	1500.5	126.5	19175	600	6.662	5.330	288	51.2	1918

CYCLE 1 - ELUTION

0.5M HNO3 density 1.015 g/mL

Eluant mass processed (g)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
9.847	9.847	9.700	0.818	515	1200	4.988	4.913	5	51	51	0.00733	0.0093
9.919	19.766	19.470	1.642	572	1200	5.048	4.972	6	56	107	0.01542	0.0102
10.111	29.877	29.430	2.482	34400	1200	5.111	5.034	342	3403	3510	0.50579	0.6083
10.277	40.154	39.553	3.335	84789	1200	0.104	0.102	41384	418931	422441	60.87886	73.6786
10.184	50.338	49.584	4.181	54815	1200	0.107	0.105	26004	260858	683299	98.47166	46.2968
9.705	60.043	59.144	4.987	61988	1200	5.099	5.023	617	5899	689198	99.32180	1.0986
9.829	69.872	68.826	5.804	24259	1200	5.103	5.027	241	2336	691534	99.65849	0.4296
9.918	79.790	78.595	6.627	11456	1200	5.094	5.018	114	1115	692649	99.81921	0.2032
9.965	89.755	88.411	7.455	6047	1200	5.102	5.026	60	591	693240	99.90431	0.1071
9.883	99.638	98.146	8.276	3035	1200	5.108	5.032	30	294	693533	99.94662	0.0537
9.852	109.490	107.851	9.094	1720	1200	5.121	5.044	17	165	693699	99.97047	0.0304
9.938	119.428	117.640	9.920	1475	1800	5.107	5.031	10	96	693795	99.98426	0.0174
9.794	129.222	127.287	10.733	861	1800	5.110	5.033	6	55	693850	99.99218	0.0102
9.888	139.110	137.027	11.555	534	1800	5.103	5.027	4	34	693884	99.99715	0.0063
9.856	148.966	146.736	12.373	306	1800	5.091	5.015	2	20	693904	100.00000	0.0036

CYCLE 1 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	24.4	g				
Final mass of effluent bottle	80.4	g				
Mass of feed processed	56	g, or	55.5	mL		
Start date and time	8/20/01 7:00					
Finish date and time	8/20/01 13:00					
Average flow rate	9.2	mL/h or	0.8	BV/h		
Bed height	6.7	cm				
Bed volume	11.9	mL				

Feed

Starting mass of effluent bottle	322.3	g				
Final mass of effluent bottle	2043.1	g				
Mass of all samples	154.812	g				
Mass of feed processed	1875.612	g or	1500	mL		
Average flow rate	30.0	mL/h or	2.5	BV/h		
Activity concentration in feed	561.7	CPM/mL				
Total activity processed	8.43E+05	CPM				
Bed height	6.2	cm				
Bed volume	11.0	mL				

Simulated LAW Effluent

Total acitvity in samples	16659.6	CPM				
Estimated activity in VOA/SVOA samples	13463.2	CPM				
Activity in effluent bottle sample	15633	counts in	900	seconds of mass	6.253	g
Effluent bottle activity concentration	166.7	CPM/g				
Total mass of effluent in bottle	1721	g				
Total activity in bulk effluent	286809.2	CPM				
Total activity in simulated LAW effluent	3.17E+05	CPM, or	37.6%	of feed		

Feed Displacement

Tare mass of effluent bottle	24.8	g				
Final mass of effluent bottle	76.6	g				
Mass of feed processed	51.8	g or	43.9	mL		
Start date and time	8/22/01 15:13					
Finish date and time	8/22/01 16:43					
Average flow rate	29.3	mL/h or	2.5	BV/h		
Activity in bulk composite sample	19366	counts in	900	seconds of mass	5.896	g
Composite bulk activity concentration	219.0	CPM/g				
Total activity in effluent	1.13E+04	CPM, or	1.3%	of feed		
Bed height	NM	cm				

Water Rinse

Tare mass of effluent bottle	24.4	g				
Final mass of effluent bottle	68.8	g				
Mass of feed processed	44.4	g, or	43.7	mL		
Start date and time	8/22/01 16:45					
Finish date and time	8/22/01 18:15					
Average flow rate	29.1	mL/h or	2.5	BV/h		
Activity in bulk composite sample	1046	counts in	1800	seconds of mass	5.085	g
Composite bulk activity concentration	6.9	CPM/g				
Total activity in effluent	3.04E+02	CPM, or	0.0%	of feed		
Bed height	NM	cm				

Elution

Total mass of eluant processed	149	g				
Average flow rate	9.93	mL/h, or	0.8	BV/h		
Activity in bulk composite sample	52188	counts in	300	seconds of mass	4.771	g
Composite bulk activity concentration	2187.7	CPM/g				
Total activity in effluent	6.94E+05	CPM, or	82.3%	of feed based on eluant fraction analysis		
Total activity in effluent	3.26E+05	CPM, or	38.7%	of feed based on composite analysis		

Note: likely error in records leads to underestimate of eluant activity based on composite analysis.

Bed height	NM	cm

Water Rinse

Tare mass of effluent bottle	19.7	g				
Final mass of effluent bottle	101.1	g				
Mass of feed processed	81.4	g, or	80.6	mL		
Start date and time	8/23/01 14:00					
Finish date and time	8/23/01 16:50					
Average flow rate	28.5	mL/h or	2.4	BV/h		
Activity in bulk composite sample	74	counts in	1200	seconds of mass	5.048	g
Composite bulk activity concentration	0.7	CPM/g				
Total activity in effluent	5.97E+01	CPM, or	0.0%	of feed		
Bed height	5.4	cm				
Bed volume	9.6	mL				

Total

Total activity in all effluents	1022542.8	CPM
Total activity in feed	842790.1	CPM
Activity recovery, as fraction of feed	121.3%	

CYCLE 2 - SIMULATED AW-101 LAW PROCESSING

Effluent bottle tare	322.2	g		
Feed density	1.236	g/mL		
Bed height in 0.25M NaOH	6.6	cm		
Bed volume in 0.25M NaOH	11.7	mL		
Cs-133 concentration	6.40E-05	M		
Potassium concentration	4.30E-01	M		
Hydroxide concentration	4.32	M		
Nitrate concentration	1.52	M		
Nitrite concentration	0.79	M		
Flow rate	30.9	mL/h or	2.6	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	15201	300	6.398	587
Feed 2	15955	300	6.536	603
Feed 3	15298	300	6.398	591
Feed 4	15714	300	6.398	607
Feed 5	16104	300	6.536	609
Average				600

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
441.7	119.5	8/27/01 14:00	8/27/01 17:20	29.0	102.0	8.7	32	3000	6.599	5.339	0.120	0.02
562.8	240.6	8/27/01 17:30	8/27/01 20:40	30.9	205.2	17.6	51	3000	6.471	5.235	0.195	0.03
683.7	361.5	8/27/01 20:50	8/28/01 0:00	30.9	307.9	26.4	339	3000	5.936	4.803	1.412	0.24
805.0	482.8	8/28/01 0:10	8/28/01 3:20	31.0	411.3	35.2	1087	3000	6.593	5.334	4.076	0.68
925.8	603.6	8/28/01 3:30	8/28/01 6:40	30.9	514.3	44.0	2377	3000	6.424	5.197	9.147	1.53
1046.4	724.2	8/28/01 6:50	8/28/01 10:00	30.8	617.1	52.8	4884	3000	6.498	5.257	18.580	3.10
1167.2	845.0	8/28/01 10:10	8/28/01 13:20	30.9	720.1	61.6	8487	3000	6.517	5.273	32.193	5.37
1288.0	965.8	8/28/01 13:30	8/28/01 16:40	30.9	823.0	70.5	8278	1800	6.431	5.203	53.033	8.84
1408.8	1086.6	8/28/01 16:50	8/28/01 20:00	30.9	925.9	79.3	12168	1800	6.361	5.146	78.812	13.14
1529.9	1207.7	8/28/01 20:10	8/28/01 23:20	30.9	1029.2	88.1	11365	1200	6.612	5.350	106.225	17.72
1651.0	1328.8	8/28/01 23:30	8/29/01 2:40	30.9	1132.4	96.9	14402	1200	6.386	5.167	139.374	23.24
1771.9	1449.7	8/29/01 2:50	8/29/01 6:00	30.9	1235.4	105.8	13499	900	6.392	5.172	174.017	29.02
1892.4	1570.2	8/29/01 6:10	8/29/01 9:20	30.8	1338.2	114.6	17738	900	6.570	5.316	222.467	37.10
2013.5	1691.3	8/29/01 9:30	8/29/01 12:40	30.9	1441.4	123.4	14416	600	6.435	5.206	276.895	46.18
2134.7	1812.5	8/29/01 12:50	8/29/01 16:00	31.0	1544.7	132.2	16940	600	6.486	5.248	322.816	53.84
2255.4	1933.2	8/29/01 16:10	8/29/01 19:20	30.8	1647.5	141.0	9419	300	6.436	5.207	361.774	60.33
2376.0	2053.8	8/29/01 19:30	8/29/01 22:40	30.8	1755.3	150.3	10632	300	6.467	5.232	406.406	67.78
2400.8	2078.6	8/29/01 22:50	8/29/01 23:30	30.1	1775.3	152.0	8909	300	6.116	4.948	360.089	60.05

CYCLE 2 - ELUTION

0.5M HNO_3 density 1.015 g/mL

Eluant mass processed (g)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
11.245	11.245	11.077	0.948	896	1200	5.055	4.979	8.997	99.659	99.659	0.01162	0.0150
9.409	20.654	20.345	1.742	1355	1200	5.064	4.988	13.582	125.881	225.540	0.02630	0.0227
10.241	30.895	30.432	2.605	42535	1200	0.097	0.096	22258.522	224536.564	224762.104	26.21199	37.1217
10.149	41.044	40.429	3.461	81280	1200	0.200	0.197	20628.864	206227.680	430989.784	50.26247	34.4038
10.100	51.144	50.378	4.312	27440	900	0.101	0.099	18387.517	182933.333	613923.117	71.59634	30.6658
10.087	61.231	60.314	5.163	20556	900	0.102	0.100	13639.511	135521.812	749444.929	87.40103	22.7473
10.010	71.241	70.174	6.007	36647	300	1.019	1.004	7302.068	71999.307	821444.236	95.79766	12.1780
10.058	81.299	80.082	6.855	16743	300	1.011	0.996	3362.511	33313.767	854758.004	99.68275	5.6078
10.036	91.335	89.967	7.701	12504	800	5.080	5.004	187.412	1852.709	856610.713	99.89881	0.3126
10.273	101.608	100.087	8.568	4147	1200	5.079	5.003	41.446	419.395	857030.107	99.94772	0.0691
9.870	111.478	109.809	9.400	1999	1200	5.050	4.974	20.093	195.348	857225.455	99.97051	0.0335
10.272	121.750	119.927	10.266	1143	1200	5.034	4.959	11.525	116.616	857342.071	99.98411	0.0192
10.105	131.855	129.881	11.118	640	1200	5.078	5.002	6.397	63.679	857405.750	99.99153	0.0107
10.263	142.118	139.990	11.983	651	1800	5.087	5.011	4.331	43.780	857449.529	99.99664	0.0072
10.185	152.303	150.023	12.842	433	1800	5.098	5.022	2.874	28.836	857478.365	100.00000	0.0048

CYCLE 2 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	25.4	g				
Final mass of effluent bottle	91.6	g				
Mass of feed processed	66.2	g, or	65.6	mL		
Start date and time	8/27/01 7:00					
Finish date and time	8/27/01 13:00					
Average flow rate	10.9	mL/h or	0.9	BV/h		
Bed height	6.6	cm				
Bed volume	11.7	mL				

Feed

Starting mass of effluent bottle	322.2	g				
Final mass of effluent bottle	2400.8	g				
Mass of all samples	115.73					
Mass of feed processed	2194.33	g or	1775	mL		
Average flow rate	30.7	mL/h or	2.6	BV/h		
Acivity concentration in feed	485.1	CPM/g				
Total activity processed	1.06E+06	CPM				
Bed height	6.0	cm				
Bed volume	10.6	mL				

Simulated LAW Effluent

Total acitvity in samples	13325.1	CPM				
Activity in effluent bottle sample	5028	counts in	600	seconds of mass	6.27	g
Effluent bottle activity concentration	80.2	CPM/g				
Total mass of effluent in bottle	2078.6	g				
Total activity in bulk effluent	166685.8	CPM				
Total activity in simulated LAW effluent	1.80E+05	CPM, or	16.9%	of feed		

Feed Displacement

Tare mass of effluent bottle	25.5	g				
Final mass of effluent bottle	74.9	g				
Mass of feed processed	49.4	g or	41.9	mL		
Start date and time	8/29/01 23:42					
Finish date and time	8/30/01 1:12					
Average flow rate	27.9	mL/h or	2.4	BV/h		
Activity in bulk composite sample	6684	counts in	1200	seconds of mass	5.899	g
Composite bulk activity concentration	56.7	CPM/g				
Total activity in effluent	2.80E+03	CPM, or	0.3%	of feed		
Bed height	6.2	cm				
Bed volume	11.0	mL				

Water Rinse

Tare mass of effluent bottle	25.6	g				
Final mass of effluent bottle	68	g				
Mass of feed processed	42.4	g, or	41.6	mL		
Start date and time	8/30/01 1:12					
Finish date and time	8/30/01 2:43					
Average flow rate	27.5	mL/h or	2.4	BV/h		
Activity in bulk composite sample	2160	counts in	1200	seconds of mass	5.091	g
Composite bulk activity concentration	21.2	CPM/g				
Total activity in effluent	8.99E+02	CPM, or	0.1%	of feed		
Bed height	6.2	cm				
Bed volume	11.0	mL				

Elution

Total mass of eluant processed	152.303	g				
Average flow rate	10.00	mL/h, or	0.9	BV/h		
Activity in bulk composite sample	26492	counts in	300	seconds of mass	1.015	g
Composite bulk activity concentration	5220.1	CPM/g				
Total activity in effluent	7.95E+05	CPM, or	74.7%	of feed		
Bed height	5.3	cm				
Bed volume	9.4	mL				

Water Rinse

Tare mass of effluent bottle	25.7	g				
Final mass of effluent bottle	71.5	g				
Mass of feed processed	45.8	g, or	45.5	mL		
Start date and time	8/31/01 17:48					
Finish date and time	8/31/01 19:18					
Average flow rate	30.3	mL/h or	2.6	BV/h		
Activity in bulk composite sample	2480	counts in	30000	seconds of mass	5.036	g
Composite bulk activity concentration	1.0	CPM/g				
Total activity in effluent	4.51E+01	CPM, or	0.0%	of feed		
Bed height	5.3	cm				
Bed volume	9.4	mL				

Total

Total activity in all effluents	978790.8	CPM
Total activity in feed	1064515.7	CPM
Activity recovery, as fraction of feed	91.9%	

CYCLE 3 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	319.1	g		
Feed density	1.25	g/mL		
Bed height in 0.25M NaOH	6.9	cm		
Bed volume in 0.25M NaOH	12.2	mL		
Cesium concentration	6.09E-05	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	30.0	mL/h or	2.5	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	16181	300	6.335	639
Feed 2	16766	300	6.335	662
Average				650

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
431.8	112.7	9/10/01 13:30	9/10/01 16:40	28.5	95.4	7.8	24	3000	6.575	5.260	0.091	0.01
969.4	650.3	9/10/01 16:50	9/11/01 7:06	30.1	530.2	43.4	13	3000	5.899	4.719	0.055	0.01
1087.6	768.5	9/11/01 7:16	9/11/01 10:25	30.0	628.0	51.4	15	3000	4.000	3.200	0.094	0.01
1900.1	1581.0	9/11/01 10:35	9/12/01 8:15	30.0	1283.0	105.1	563	3000	6.294	5.035	2.236	0.34
2015.7	1696.6	9/12/01 8:25	9/12/01 11:31	29.8	1380.4	113.0	1079	3000	6.157	4.926	4.381	0.67
2151.9	1832.8	9/12/01 11:41	9/12/01 15:20	29.9	1494.4	122.4	2326	3000	6.335	5.068	9.179	1.41

CYCLE 4 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	319.9	g
Feed density	1.25	g/mL
Bed height in 0.25M NaOH	5.6	cm
Bed volume in 0.25M NaOH	9.9	mL
Cesium concentration	9.38E-05	M
Potassium concentration	9.51E-02	M
Hydroxide concentration	1.72	M
Nitrate concentration	1.23	M
Nitrite concentration	1.21	M
Flow rate	30.1	mL/h or 3.0 BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	15990	300	6.410	624
Average				624

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
404.6	84.7	9/18/01 11:50	9/18/01 14:12	28.6	72.9	7.4	73	1800	6.441	5.153	0.472	0.08
1064.2	744.3	9/18/01 14:22	9/19/01 7:51	30.2	605.6	61.1	38	1800	6.243	4.994	0.254	0.04
1198.4	878.5	9/19/01 8:01	9/19/01 11:35	30.1	718.1	72.4	109	1800	6.437	5.150	0.706	0.11
1348.1	1028.2	9/19/01 11:45	9/19/01 15:44	30.1	843.0	85.1	270	1800	6.450	5.160	1.744	0.28
1927.0	1607.1	9/19/01 15:54	9/20/01 7:18	30.1	1311.2	132.3	7358	1800	6.287	5.030	48.765	7.82
2059.6	1739.7	9/20/01 7:28	9/20/01 11:01	29.9	1422.4	143.5	11924	1800	6.500	5.200	76.436	12.26
2157.5	1837.6	9/20/01 11:11	9/20/01 13:48	29.9	1506.0	151.9	16370	1800	6.482	5.186	105.227	16.87

CYCLE 5 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	312.9	g		
Feed density	1.250	g/mL		
Bed height in 0.25M NaOH	5.6	cm		
Bed volume in 0.25M NaOH	9.9	mL		
Cesium concentration	1.22E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	30.1	mL/h or	3.0	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	11250	200	6.469	652
Feed 2	9991	180	6.469	644
Feed 3	9840	180	6.394	641
Feed 4	12359	200	6.469	716
Feed 5	10928	180	6.394	712
Feed 6	9974	180	6.469	642
Feed 7	10014	180	6.394	653
Average				666

VOA sample mass	29.57	g, taken from	9/26/01 14:04	to	9/26/01 14:52
SVOA sample mass	27.445	g, taken from	9/26/01 12:20	to	9/26/02 13:00

Effluent bottle mass (g)	Mass of feed collected in bottle & VOA/SVOA (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)	Activity in sample (CPM)
432.2	119.3	9/24/01 13:30	9/24/01 16:50	28.6	100.5	10.1	27	2000	6.352	5.082	0.159	0.02	0.81
552.4	239.5	9/24/01 17:00	9/24/01 20:10	30.4	201.6	20.3	37	2000	6.108	4.886	0.227	0.03	1.11
670.9	358.0	9/24/01 20:20	9/24/01 23:30	29.9	301.4	30.4	22	1800	6.240	4.992	0.147	0.02	0.73
925.9	613.0	9/24/01 23:40	9/25/01 6:28	30.0	510.4	51.5	22	2000	6.306	5.045	0.131	0.02	0.66
1034.0	721.1	9/25/01 6:38	9/25/01 9:30	30.2	601.9	60.7	93	2000	6.220	4.976	0.561	0.08	2.79
1155.4	842.5	9/25/01 9:40	9/25/01 12:53	30.2	704.0	71.0	293	2000	6.276	5.021	1.751	0.26	8.79
1272.6	959.7	9/25/01 13:03	9/25/01 16:10	30.1	802.9	81.0	855	2000	6.399	5.119	5.011	0.75	25.65
1391.6	1078.7	9/25/01 16:20	9/25/01 19:30	30.1	903.1	91.1	1976	2000	6.295	5.036	11.771	1.77	59.28
1509.5	1196.6	9/25/01 19:40	9/25/01 22:50	29.8	1002.5	101.1	4609	2000	6.344	5.075	27.244	4.09	138.27
1756.1	1443.2	9/25/01 23:00	9/26/01 5:36	29.9	1204.8	121.6	16224	2000	6.317	5.054	96.312	14.47	486.72
1871.8	1558.9	9/26/01 5:46	9/26/01 8:50	30.2	1302.8	131.4	25789	2000	6.743	5.394	143.421	21.54	773.67
1991.5	1678.6	9/26/01 9:00	9/26/01 12:10	30.2	1403.6	141.6	36563	2000	6.327	5.062	216.708	32.55	1096.89
2050.7	1794.8	9/26/01 12:20	9/26/01 15:20	31.0	1501.9	151.5	51755	2000	6.571	5.257	295.360	44.36	1552.65

CYCLE 5 - ELUTION

0.5M HNO3 density 1.015 g/mL

Eluant mass processed (g)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
9.084	9.084	8.948	0.903	441	1800	5.042	4.967	2.960	26.484	26	0.00256	0.0044
9.741	18.825	18.543	1.871	621	1800	5.047	4.971	4.164	39.952	66	0.00642	0.0063
9.843	28.668	28.239	2.849	3687	1200	5.110	5.033	36.625	355.099	422	0.04076	0.0550
9.853	38.521	37.944	3.828	82115	600	0.100	0.099	83363.148	809079.095	809501	78.26786	125.2117
9.521	48.042	47.323	4.774	34153	900	0.100	0.099	23114.750	216780.475	1026281	99.22763	34.7184
10.135	58.177	57.306	5.781	32219	900	5.101	5.025	427.481	4267.654	1030549	99.64025	0.6421
10.141	68.318	67.295	6.789	26478	1800	5.104	5.028	175.552	1753.614	1032302	99.80980	0.2637
10.306	78.624	77.447	7.813	12872	1800	5.083	5.007	85.695	869.951	1033172	99.89392	0.1287
9.893	88.517	87.192	8.797	6970	1800	5.077	5.001	46.458	452.723	1033625	99.93769	0.0698
10.152	98.669	97.192	9.805	3811	1800	5.081	5.005	25.382	253.817	1033879	99.96223	0.0381
10.057	108.726	107.098	10.805	2301	1800	5.091	5.015	15.295	151.517	1034030	99.97688	0.0230
10.152	118.878	117.098	11.814	1407	1800	5.049	4.973	9.430	94.302	1034125	99.98600	0.0142
10.092	128.970	127.039	12.817	1011	1800	5.199	5.121	6.581	65.417	1034190	99.99232	0.0099
10.330	139.300	137.214	13.843	671	1800	5.102	5.026	4.451	45.286	1034235	99.99670	0.0067
10.217	149.517	147.278	14.859	506	1800	5.049	4.973	3.391	34.131	1034270	100.00000	0.0051

CYCLE 5 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	26	g				
Final mass of effluent bottle	85.6	g				
Mass of feed processed	59.6	g, or	59.0	mL		
Start date and time	9/24/01 7:40					
Finish date and time	9/24/01 13:45					
Average flow rate	9.7	mL/h or	1.0	BV/h		
Bed height	5.5	cm				
Bed volume	9.7	mL				

Feed

Starting mass of effluent bottle	312.9	g				
Final mass of effluent bottle and voa/svoa samples	2107.715	g				
Mass of all samples	82.498					
Mass of feed processed	1877.313	g or	1502	mL		
Average flow rate	30.1	mL/h or	3.1	BV/h		
Activity concentration in feed	532.6	CPM/g				
Total activity processed	1.00E+06	CPM				
Bed height	5.0	cm				
Bed volume	8.9	mL				

Simulated LAW Effluent

Total acitvity in samples	4148.0	CPM				
Estimated activity in VOA/SVOA samples	11678.2	CPM				
Activity in effluent bottle sample	2076	counts in	1800	seconds of mass	6.262	g
Effluent bottle activity concentration	11.1	CPM/g				
Total mass of effluent in bottle	1737.8	g				
Total activity in bulk effluent	19204.0	CPM				
Total activity in simulated LAW effluent	3.50E+04	CPM, or	3.5%	of feed		

Feed Displacement

Tare mass of effluent bottle	25.8	g				
Final mass of effluent bottle	76.9	g				
Mass of feed processed	51.1	g or	43.0	mL		
Start date and time	9/26/01 15:30					
Finish date and time	9/26/01 17:00					
Average flow rate	28.7	mL/h or	2.4	BV/h		
Activity in bulk composite sample	32389	counts in	1800	seconds of mass	5.941	g
Composite bulk activity concentration	181.7	CPM/g				
Total activity in effluent	9.29E+03	CPM, or	0.9%	of feed		
Bed height	NM	cm				

Water Rinse

Tare mass of effluent bottle	25.6	g				
Final mass of effluent bottle	69.4	g				
Mass of feed processed	43.8	g, or	43.1	mL		
Start date and time	9/26/01 17:00					
Finish date and time	9/26/01 18:30					
Average flow rate	28.7	mL/h or	2.9	BV/h		
Activity in bulk composite sample	1411	counts in	1800	seconds of mass	5.085	g
Composite bulk activity concentration	9.2	CPM/g				
Total activity in effluent	4.05E+02	CPM, or	0.0%	of feed		
Bed height	NM	cm				

Elution

Total mass of eluant processed	149.517	g				
Average flow rate	9.82	mL/h, or	1.0	BV/h		
Activity in bulk composite sample	16650	counts in	1800	seconds of mass	0.097	g
Composite bulk activity concentration	5721.6	CPM/g				
Total activity in effluent	8.55E+05	CPM, or	85.6%	of feed based on composite analysis		
Total activity in effluent	1.03E+06	CPM, or	103.4%	of feed based on eluant fraction analysis		
Bed height	NM	cm				

Water Rinse

Tare mass of effluent bottle	25.5	g				
Final mass of effluent bottle	70.3	g				
Mass of feed processed	44.8	g, or	44.4	mL		
Start date and time	9/27/01 14:07					
Finish date and time	9/27/01 15:37					
Average flow rate	29.6	mL/h or	3.0	BV/h		
Activity in bulk composite sample	74	counts in	1200	seconds of mass	5.048	g
Composite bulk activity concentration	0.7	CPM/g				
Total activity in effluent	3.28E+01	CPM, or	0.0%	of feed		
Bed height	3.8	cm				
Bed volume	6.7	mL				

Total

Total activity in all effluents	900238.3	CPM				
Total activity in feed	999898.2	CPM				
Activity recovery, as fraction of feed	90.0%					

CYCLE 6 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	309.8	g		
Feed density	1.25	g/mL		
Bed height in 0.25M NaOH	5.5	cm		
Bed volume in 0.25M NaOH	9.7	mL		
Cesium concentration	2.35E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	30.0	mL/h or	3.1	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	13263	300	6.398	518.248
Average				518.248

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
970.9	661.1	10/1/01 13:45	10/2/01 7:32	29.7	534.1	54.9	354	1800	6.552	5.242	2.251	0.43
1139.0	829.2	10/2/01 7:42	10/2/01 12:12	29.9	673.8	69.2	2554	1800	6.477	5.182	16.430	3.17
1280.4	970.6	10/2/01 12:22	10/2/01 16:07	30.2	792.0	81.4	8641	1800	6.412	5.130	56.151	10.83
1868.8	1559.0	10/2/01 16:17	10/3/01 7:56	30.1	1267.7	130.2	9505	300	6.153	4.922	386.194	74.52
1992.0	1682.2	10/3/01 8:06	10/3/01 11:23	30.0	1371.2	140.9	11429	300	6.197	4.958	461.070	88.97
2143.2	1833.4	10/3/01 11:33	10/3/01 15:35	30.0	1497.3	153.8	10194	240	6.420	5.136	496.203	95.75

CYCLE 7 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	320.4	g		
Feed density	1.26	g/mL		
Bed height in 0.25M NaOH	5.65	cm		
Bed volume in 0.25M NaOH	10.0	mL		
Cesium concentration	3.48E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	29.9	mL/h or	3.0	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	11656	300	6.303	466.018
Feed 2	12752	300	6.303	509.837
Average				487.928

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
453.2	132.8	10/8/01 13:00	10/8/01 16:47	27.9	110.7	11.1	30	1800	6.744	5.352	0.187	0.04
1007.7	687.3	10/8/01 16:57	10/9/01 7:40	29.9	555.9	55.6	4646	1200	6.368	5.054	45.964	9.42
1148.4	828.0	10/9/01 7:50	10/9/01 11:33	30.0	672.5	67.2	8700	900	6.213	4.931	117.624	24.11
1339.5	1019.1	10/9/01 11:43	10/9/01 16:46	30.0	829.2	82.9	11408	600	6.385	5.067	225.123	46.14
1847.1	1526.7	10/9/01 16:56	10/10/01 6:28	29.8	1237.0	123.7	11729	300	6.231	4.945	474.355	97.22
1982.2	1661.8	10/10/01 6:38	10/10/01 10:13	29.9	1349.4	134.9	12815	300	6.461	5.128	499.827	102.44
2101.7	1781.3	10/10/01 10:23	10/10/01 13:33	29.9	1449.2	144.9	11879	300	6.307	5.006	474.633	97.28
2143.6	1823.2	10/10/01 13:43	10/10/01 14:50	29.8	1487.6	148.8	12193	300	6.482	5.144	474.026	97.15

CYCLE 8 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	310.6	g		
Feed SG	1.26			
Bed height in 0.25M NaOH	5.7	cm		
Bed volume in 0.25M NaOH	10.1	mL		
Cesium concentration	4.61E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	29.9	mL/h or	3.0	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	14518	300	6.617	552.900
Feed 2				
Feed 3				
Feed 4				
Feed 5				
Average				552.900

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
438.5	127.9	10/16/01 12:55	10/16/01 16:30	28.3	106.4	10.5	25	1800	6.153	4.883	0.171	0.03
965.8	655.2	10/16/01 16:40	10/17/01 6:40	29.9	529.9	52.5	14934	1800	6.276	4.981	99.941	18.08
1107.7	797.1	10/17/01 6:50	10/17/01 10:36	29.9	647.5	64.2	35599	1800	6.378	5.062	234.424	42.40
1239.3	928.7	10/17/01 10:46	10/17/01 14:15	30.0	756.9	75.0	12210	600	6.228	4.943	247.023	44.68
1319.3	1008.7	10/17/01 14:25	10/17/01 16:32	30.0	825.4	81.8	23806	600	6.228	4.943	481.624	87.11
1907.8	1597.2	10/17/01 16:42	10/18/01 8:16	30.0	1297.5	128.6	14057	300	6.398	5.078	553.667	100.14
2025.3	1714.7	10/18/01 8:26	10/18/01 11:32	30.1	1395.8	138.4	13608	300	6.406	5.084	535.313	96.82
2140.0	1829.4	10/18/01 11:42	10/18/01 14:45	29.8	1492.0	147.9	14113	300	6.419	5.094	554.055	100.21

CYCLE 9 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	311.1	g		
Feed density	1.26	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	5.64E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	29.9	mL/h or	2.9	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	14367	300	6.379	567.563
Average				567.563

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
422.5	111.4	10/22/01 12:50	10/22/01 15:55	28.7	93.2	9.1	49	1800	6.062	4.811	0.339	0.06
592.0	280.9	10/22/01 16:05	10/22/01 20:38	29.6	232.8	22.7	26	1800	6.321	5.017	0.173	0.03
1010.0	698.9	10/22/01 20:48	10/23/01 7:55	29.8	568.4	55.4	13093	900	4.844	3.844	227.046	40.00
1147.0	835.9	10/23/01 8:05	10/23/01 11:43	29.9	682.1	66.4	10443	300	6.264	4.971	420.121	74.02
1296.1	985.0	10/23/01 11:53	10/23/01 15:51	29.8	805.4	78.5	13093	300	6.316	5.013	522.393	92.04
1924.4	1613.3	10/23/01 16:01	10/24/01 8:39	30.0	1309.2	127.5	14901	300	6.442	5.113	582.902	102.70

CYCLE 10 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	313.8	g		
Feed density	1.260	g/mL		
Bed height in 0.25M NaOH	5.9	cm		
Bed volume in 0.25M NaOH	10.443	mL		
Cesium concentration	1.22E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	28.9	mL/h or	2.8	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	15300	300	6.297	612
Feed 2	16414	300	6.297	657
Feed 3	16144	300	6.301	646
Feed 4	16543	300	6.297	662
Feed 5	15616	300	6.301	625
Feed 6	12187	240	6.297	610
Feed 7	12856	240	6.301	643
Average				636

VOA sample mass	30.413	g, taken from	10/31/01 12:40	to	10/31/01 13:30
SVOA sample mass	30.205	g, taken from	10/31/01 13:35	to	10/31/01 14:25

Effluent bottle mass (g)	Mass of feed collected in bottle & VOA/SVOA (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)	Activity in sample (CPM)
427.0	113.2	10/29/01 13:00	10/29/01 16:20	27.0	95.0	9.1	20	1800	6.479	5.142	0.130	0.02	0.67
545.7	231.9	10/29/01 16:30	10/29/01 19:40	29.7	194.1	18.6	36	1800	6.144	4.876	0.246	0.04	1.20
664.0	350.2	10/29/01 19:50	10/29/01 23:00	29.6	292.9	28.0	33	1800	6.179	4.904	0.224	0.04	1.10
987.9	674.1	10/29/01 23:10	10/30/01 7:50	29.7	554.7	53.1	59	1800	5.972	4.740	0.415	0.07	1.97
1056.8	743.0	10/30/01 8:00	10/30/01 9:50	29.8	614.2	58.8	139	1800	6.136	4.870	0.951	0.15	4.63
1145.7	831.9	10/30/01 10:00	10/30/01 12:23	29.6	690.0	66.1	323	1800	6.632	5.263	2.046	0.32	10.77
1263.8	950.0	10/30/01 12:33	10/30/01 15:40	30.1	789.2	75.6	1020	1800	6.809	5.404	6.292	0.99	34.00
1380.1	1066.3	10/30/01 15:50	10/30/01 19:00	29.1	886.4	84.9	2804	1800	6.255	4.964	18.828	2.96	93.47
1498.2	1184.4	10/30/01 19:10	10/30/01 22:20	29.6	985.1	94.3	6623	1800	6.161	4.890	45.149	7.10	220.77
1911.4	1597.6	10/30/01 22:30	10/31/01 9:35	29.6	1318.3	126.2	41840	1800	6.715	5.329	261.695	41.13	1394.67
1983.6	1669.8	10/31/01 9:45	10/31/01 11:41	29.6	1380.6	132.2	16312	600	6.306	5.005	325.930	51.23	1631.20
2030.9	1777.7	10/31/01 11:51	10/31/01 15:50	21.5	1471.0	140.9	19218	600	5.963	4.733	406.082	63.82	1921.80

CYCLE 10 - ELUTION

0.5M HNO_3 density 1.015 g/mL

Eluant mass processed (g)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
10.308	10.308	10.154	0.972	667	1800	5.011	4.936	4.504	46	46	0.00573	0.0071
10.273	20.581	20.273	1.941	1239	1800	5.030	4.955	8.336	84	130	0.01629	0.0131
10.407	30.988	30.524	2.923	10472	600	0.101	0.099	10525.915	107903	108033	13.52903	16.5437
10.506	41.494	40.873	3.914	10009	100	0.098	0.097	62211.042	643803	751837	94.15277	97.7780
10.432	51.926	51.149	4.898	12077	1800	0.098	0.097	4170.262	42853	794689	99.51925	6.5545
10.483	62.409	61.475	5.887	9480	600	5.087	5.011	189.190	1954	796643	99.76390	0.2974
10.383	72.792	71.702	6.866	11993	1800	5.076	5.000	79.953	818	797461	99.86630	0.1257
10.454	83.246	82.000	7.852	6548	1800	5.074	4.998	43.671	450	797910	99.92262	0.0686
10.525	93.771	92.367	8.845	3326	1800	5.074	4.998	22.182	230	798140	99.95141	0.0349
10.471	104.242	102.681	9.833	2068	1800	5.077	5.001	13.784	142	798283	99.96922	0.0217
10.527	114.769	113.051	10.825	1238	1800	5.094	5.018	8.224	85	798368	99.97990	0.0129
10.553	125.322	123.446	11.821	878	1800	5.095	5.019	5.832	61	798428	99.98749	0.0092
10.400	135.722	133.690	12.802	638	1800	5.097	5.021	4.236	43	798472	99.99292	0.0067
10.513	146.235	144.046	13.793	457	1800	5.087	5.011	3.040	31	798503	99.99687	0.0048
10.408	156.643	154.298	14.775	367	1800	5.088	5.012	2.441	25	798528	100.00000	0.0038

CYCLE 10 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	26	g				
Final mass of effluent bottle	105.1	g				
Mass of feed processed	79.1	g, or	78.4	mL		
Start date and time	10/29/01 5:16					
Finish date and time	10/29/01 13:00					
Average flow rate	10.1	mL/h or	1.0	BV/h		
Bed height	5.9	cm				
Bed volume	10.4	mL				

Feed

Starting mass of effluent bottle	313.8	g				
Final mass of effluent bottle and voa/svoa samples	2091.518	g				
Mass of all samples	75.751					
Mass of feed processed	1853.469	g or	1471	mL		
Average flow rate	28.9	mL/h or	2.8	BV/h		
Acivity concentration in feed	505.0	CPM/g				
Total activity processed	9.36E+05	CPM				
Bed height	5.0	cm				
Bed volume	8.9	mL				

Simulated LAW Effluent

Total acitvity in samples	5316.2	CPM				
Estimated activity in VOA/SVOA samples	17608.4	CPM				
Activity in effluent bottle sample	617	counts in	1800	seconds of mass	6.325	g
Effluent bottle activity concentration	3.3	CPM/g				
Total mass of effluent in bottle	1717.1	g				
Total activity in bulk effluent	5583.4	CPM				
Total activity in simulated LAW effluent	2.85E+04	CPM, or	3.0%	of feed		

Feed Displacement

Tare mass of effluent bottle	26.1	g				
Final mass of effluent bottle	77.5	g				
Mass of feed processed	51.4	g or	45.0	mL		
Start date and time	10/31/01 16:02					
Finish date and time	10/31/01 17:32					
Average flow rate	30.0	mL/h or	2.9	BV/h		
Activity in bulk composite sample	12927	counts in	600	seconds of mass	5.712	g
Composite bulk activity concentration	226.3	CPM/g				
Total activity in effluent	1.16E+04	CPM, or	1.2%	of feed		
Bed height	NM	cm				

Water Rinse

Tare mass of effluent bottle	25.7	g				
Final mass of effluent bottle	70.2	g				
Mass of feed processed	44.5	g, or	44.5	mL		
Start date and time	10/31/01 16:32					
Finish date and time	10/31/01 18:02					
Average flow rate	29.7	mL/h or	2.8	BV/h		
Activity in bulk composite sample	1841	counts in	1800	seconds of mass	4.965	g
Composite bulk activity concentration	12.4	CPM/g				
Total activity in effluent	5.50E+02	CPM, or	0.1%	of feed		
Bed height	NM	cm				

Elution

Total mass of eluant processed	156.643	g				
Average flow rate	10.29	mL/h, or	1.0	BV/h		
Activity in bulk composite sample	11802	counts in	1200	seconds of mass	0.099	g
Composite bulk activity concentration	5960.6	CPM/g				
Total activity in effluent	9.34E+05	CPM, or	99.8%	of feed based on composite analysis		
Total activity in effluent	7.99E+05	CPM, or	85.3%	of feed based on eluant fraction analysis		
Bed height	NM	cm				

Water Rinse

Tare mass of effluent bottle	25.6	g				
Final mass of effluent bottle	71.2	g				
Mass of feed processed	45.6	g, or	45.6	mL		
Start date and time	11/1/01 15:22					
Finish date and time	11/1/01 16:53					
Average flow rate	30.1	mL/h or	2.9	BV/h		
Activity in bulk composite sample	83	counts in	1800	seconds of mass	5.002	g
Composite bulk activity concentration	0.6	CPM/g				
Total activity in effluent	2.52E+01	CPM, or	0.0%	of feed		
Bed height	3.5	cm				
Bed volume	6.2	mL				

Total

Total activity in all effluents	974402.9	CPM				
Total activity in feed	935925.4	CPM				
Activity recovery, as fraction of feed	104.1%					

CYCLE 11 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	310.9	g		
Feed density	1.22	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	2.35E-04	M		
Potassium concentration	8.00E-03	M		
Hydroxide concentration	2.2	M		
Nitrate concentration	0.1	M		
Nitrite concentration	1.46	M		
Flow rate	60.4	mL/h or	5.9	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	16511	300	6.044	666.559
Average				666.559

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
429.0	118.1	11/5/01 14:06	11/5/01 15:51	55.32	102.0	9.9	38	1800	6.332	5.190	0	0.04
860.3	549.4	11/5/01 15:56	11/5/01 21:43	61.13	460.6	44.9	38	1800	6.195	5.078	0	0.04
1546.0	1235.1	11/5/01 21:48	11/6/01 7:06	60.44	1027.9	100.1	19716	1800	6.401	5.247	125	18.79
1645.0	1334.1	11/6/01 7:11	11/6/01 8:32	60.11	1114.1	108.5	14039	900	6.117	5.014	187	28.00
1765.2	1454.3	11/6/01 8:37	11/6/01 10:16	59.71	1217.3	118.6	13969	600	5.753	4.716	296	44.44

CYCLE 12 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	311.4	g		
Feed density	1.22	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	6.58E-05	M		
Potassium concentration	8.00E-03	M		
Hydroxide concentration	2.2	M		
Nitrate concentration	0.1	M		
Nitrite concentration	1.46	M		
Flow rate	14.6	mL/h or	1.4	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	16461	300	6.095	658.980
Average				658.980

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
375.6	64.2	11/13/01 13:00	11/13/01 16:13	16.36	57.8	5.6	24	1800	6.332	5.190	0.154	0.02
650.3	338.9	11/13/01 16:18	11/14/01 8:25	13.97	288.1	28.1	18	1800	6.195	5.078	0.118	0.02
748.3	436.9	11/14/01 8:30	11/14/01 14:19	13.81	373.6	36.4	15	1800	6.401	5.247	0.095	0.01
1055.2	743.8	11/14/01 14:24	11/15/01 7:58	14.32	630.2	61.4	16	900	6.117	5.014	0.213	0.03
1158.7	847.3	11/15/01 8:03	11/15/01 14:10	13.87	719.8	70.1	14	600	5.753	4.716	0.297	0.05

CYCLE 13 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	311.5	g		
Feed density	1.220	g/mL		
Bed height in 0.25M NaOH	5.9	cm		
Bed volume in 0.25M NaOH	10.4	mL		
Cesium concentration	2.35E-04	M		
Potassium concentration	8.00E-03	M		
Hydroxide concentration	2.2	M		
Nitrate concentration	0.1	M		
Nitrite concentration	1.46	M		
Flow rate	30.2	mL/h or	2.9	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	14231	300	6.075	571.583
Feed 2	14012	300	6.075	562.787
Average				567.185

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
550.4	238.9	11/26/01 14:05	11/26/01 20:43	29.52	200.8	19.2	19	1800	6.020	4.934	0.128	0.023
943.9	632.4	11/26/01 20:53	11/27/01 7:35	30.14	528.4	50.6	16	1800	6.260	5.131	0.104	0.018
1048.6	737.1	11/27/01 7:45	11/27/01 10:35	30.29	619.1	59.3	25	1800	5.982	4.903	0.170	0.030
1278.4	966.9	11/27/01 10:45	11/27/01 16:58	30.30	812.8	77.8	532	1800	6.493	5.322	3.332	0.587
1843.9	1532.4	11/27/01 17:08	11/28/01 8:32	30.10	1281.5	122.7	9548	300	6.235	5.111	373.651	65.878
1965.5	1654.0	11/28/01 8:42	11/28/01 12:01	30.05	1386.2	132.7	12872	300	6.173	5.060	508.791	89.705
2102.2	1790.7	11/28/01 12:11	11/28/01 15:55	30.01	1503.6	144.0	13568	300	6.495	5.324	509.714	89.867

CYCLE 14 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	311.3	g		
Feed density	1.240	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	2.35E-04	M		
Potassium concentration	8.00E-03	M		
Hydroxide concentration	1.2	M		
Nitrate concentration	0.5	M		
Nitrite concentration	2.46	M		
Flow rate	14.8	mL/h or	1.4	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	18776	300	7.466	624
Feed 2	18928	300	7.466	629
Average				626

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
353.9	42.6	12/3/01 13:47	12/3/01 16:15	13.93	39.4	3.8	18	1800	6.203	5.002	0.120	0.019
632.4	321.1	12/3/01 16:35	12/4/01 7:55	14.65	268.7	26.2	14	1800	5.867	4.731	0.099	0.016
698.0	386.7	12/4/01 8:15	12/4/01 11:55	14.43	326.5	31.8	8	1800	6.037	4.869	0.055	0.009
765.4	454.1	12/4/01 12:15	12/4/01 15:55	14.82	385.6	37.6	9	1800	5.965	4.810	0.062	0.010
1052.5	741.2	12/4/01 16:15	12/5/01 7:55	14.78	622.2	60.6	16	1800	6.202	5.002	0.107	0.017
1121.6	810.3	12/5/01 8:15	12/5/01 12:00	14.86	682.8	66.5	78	1800	6.040	4.871	0.534	0.085
1178.8	867.5	12/5/01 12:20	12/5/01 15:27	14.80	733.8	71.5	419	1800	6.090	4.911	2.844	0.454

CYCLE 14 - ELUTION

0.5M HNO3 density 1.015 g/mL
Flow rate 19.8 mL/h or 1.9 BV/h

Eluant mass processed (g)	Start time	Finish time	Flow rate (mL/h)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
19.601	12/5/01 19:00	12/5/01 20:00	19.3	19.601	19.308	1.881	15	1800	5.022	4.947	0.101	2	2	0.00038	0.000
10.018	12/5/01 20:00	12/5/01 20:30	19.7	29.619	29.176	2.842	36968	1800	0.100	0.099	12509.971	123448	123450	23.76960	19.977
10.116	12/5/01 20:30	12/5/01 21:00	19.9	39.735	39.140	3.813	84799	1800	0.099	0.098	28985.840	288831	412281	79.38209	46.288
9.707	12/5/01 21:00	12/5/01 21:29	19.8	49.442	48.702	4.744	31458	1800	0.098	0.097	10862.640	103865	516146	99.38061	17.347
20.378	12/5/01 21:29	12/5/01 22:30	19.7	69.820	68.775	6.699	17484	1800	5.082	5.006	116.422	2337	518483	99.83058	0.186
20.197	12/5/01 22:30	12/5/01 23:30	19.9	90.017	88.669	8.637	3563	1800	5.075	4.999	23.758	473	518955	99.92158	0.038
21.935	12/5/01 23:30	12/6/01 0:35	19.9	111.952	110.276	10.742	1681	1800	5.074	4.998	11.211	242	519198	99.96822	0.018
18.699	12/6/01 0:35	12/6/01 1:30	20.1	130.651	128.695	12.536	813	1800	5.071	4.995	5.425	100	519298	99.98746	0.009
20.349	12/6/01 1:30	12/6/01 2:30	20.0	151.000	148.739	14.489	487	1800	5.074	4.998	3.248	65	519363	100.00000	0.005

CYCLE 15 - SIMULATED AN-105 PROCESSING

Effluent bottle tare	309.7	g		
Feed density	1.260	g/mL		
Bed height in 0.25M NaOH	6	cm		
Bed volume in 0.25M NaOH	10.62	mL		
Cesium concentration	1.22E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	30.0	mL/h or	2.8	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	15779	300	6.203	641
Feed 2	12135	240	6.203	616
Feed 3	12209	240	6.260	614
Feed 4	13017	240	6.203	661
Feed 5	12632	240	6.260	636
Feed 6	15308	300	6.203	622
Feed 7	13212	240	6.260	665
Feed 8	12548	240	6.203	637
Average				637

VOA sample mass	30.425	g, taken from	12/12/01 8:13	to	12/12/01 9:04
SVOA sample mass	29.91	g, taken from	12/12/01 7:24	to	12/12/01 8:13

Effluent bottle mass (g)	Mass of feed collected in bottle & VOA/SVOA (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)	Activity in sample (CPM)
428.9	119.2	12/10/01 15:09	12/10/01 18:29	28.4	99.5	9.4	23	1800	6.127	4.863	0.158	0.025	0.77
549.0	239.3	12/10/01 18:39	12/10/01 21:49	30.1	199.8	18.8	19	1800	6.350	5.040	0.126	0.020	0.63
593.7	284.0	12/10/01 21:59	12/10/01 23:10	30.0	240.7	22.7	26	1800	6.748	5.356	0.162	0.025	0.87
905.8	596.1	12/10/01 23:20	12/11/01 7:35	30.0	493.6	46.5	1284	1800	6.654	5.281	8.105	1.273	42.80
1034.8	725.1	12/11/01 7:45	12/11/01 11:09	30.1	600.9	56.6	3816	1800	6.214	4.932	25.792	4.052	127.20
1156.2	846.5	12/11/01 11:19	12/11/01 14:32	30.0	702.3	66.1	9013	1800	6.365	5.052	59.473	9.343	300.43
1273.0	963.3	12/11/01 14:42	12/11/01 17:49	29.7	800.1	75.3	16312	1800	6.342	5.033	108.026	16.971	543.73
1393.1	1083.4	12/11/01 17:59	12/11/01 21:09	30.1	900.5	84.8	13048	900	6.474	5.138	169.297	26.597	869.87
1457.5	1147.8	12/11/01 21:19	12/11/01 23:00	30.4	956.7	90.1	15327	900	6.420	5.095	200.540	31.505	1021.80
1763.5	1453.8	12/11/01 23:10	12/12/01 7:14	30.1	1204.6	113.4	17817	600	6.307	5.006	355.945	55.920	1781.70
1819.9	1570.5	12/12/01 7:24	12/12/01 10:29	30.0	1302.3	122.6	20484	600	6.396	5.076	403.531	63.396	2048.40
1941.1	1691.7	12/12/01 10:39	12/12/01 13:50	30.2	1403.6	132.2	11165	300	6.353	5.042	442.874	69.577	2233.00
2018.9	1769.5	12/12/01 14:00	12/12/01 16:05	29.6	1465.3	138.0							
2047.0	1797.6	12/14/01 9:40	12/14/01 10:25	29.7	1492.7	140.6	11803	300	6.441	5.112	461.785	72.548	2360.60

CYCLE 15 - ELUTION

0.5M HNO3 density 1.015 g/mL

Eluant mass processed (g)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
10.256	10.256	10.102	0.951	3732	1800	5.007	4.932	25.223	254.813	255	0.03740	0.0396
10.191	20.447	20.141	1.897	1973	1800	5.008	4.933	13.332	133.831	389	0.05705	0.0209
10.147	30.594	30.136	2.838	11615	1000	0.099	0.098	7146.393	71428.730	71817	10.54134	11.2272
10.526	41.120	40.504	3.814	27391	300	0.101	0.099	55064.046	570926.071	642743	94.34174	86.5071
10.318	51.438	50.668	4.771	7783	1800	0.095	0.094	2772.387	28177.191	670921	98.47759	4.3555
10.068	61.506	60.585	5.705	72967	1800	5.038	4.963	490.116	4860.604	675781	99.19102	0.7700
10.491	71.997	70.919	6.678	35733	1800	5.031	4.956	240.351	2483.767	678265	99.55559	0.3776
10.602	82.599	81.362	7.661	18889	1800	5.054	4.978	126.475	1320.810	679586	99.74946	0.1987
10.099	92.698	91.310	8.598	10474	1800	5.059	4.983	70.061	696.955	680283	99.85176	0.1101
10.489	103.187	101.642	9.571	6162	1800	5.041	4.966	41.365	427.384	680710	99.91449	0.0650
10.436	113.623	111.922	10.539	3460	1800	5.021	4.946	23.319	239.717	680950	99.94968	0.0366
10.419	124.042	122.185	11.505	2177	1800	5.045	4.969	14.603	149.866	681100	99.97167	0.0229
10.311	134.353	132.341	12.462	1380	1800	5.050	4.974	9.247	93.922	681194	99.98546	0.0145
10.283	144.636	142.470	13.415	844	1800	5.038	4.963	5.669	57.423	681251	99.99389	0.0089
10.280	154.916	152.597	14.369	611	1800	5.027	4.952	4.113	41.649	681293	100.00000	0.0065

CYCLE 15 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	26	g				
Final mass of effluent bottle	40.6	g, and VOA & SVOA samples	44.361	g		
Mass of feed processed	58.961	g, or	58.4	mL		
Start date and time	12/10/01 9:09					
Finish date and time	12/10/01 15:09					
Average flow rate	9.7	mL/h or	0.9	BV/h		
Bed height	6.0	cm				
Bed volume	10.6	mL				

Feed

Starting mass of feed bottle	2330.5	g				
Final mass of feed bottle	474	g				
Mass of feed processed	1857	g or	1474	mL		
Average flow rate	30.0	mL/h or	2.8	BV/h		
Acivity concentration in feed	505.2	CPM/g				
Total activity processed	9.38E+05	CPM				
Bed height	5.0	cm				
Bed volume	8.9	mL				

Simulated LAW Effluent

Total acitvity in samples	11331.8	CPM				
Estimated activity in VOA/SVOA samples	20265.0	CPM				
Activity in effluent bottle sample	4883	counts in	1800	seconds of mass	6.191	g
Effluent bottle activity concentration	26.3	CPM/g				
Total mass of effluent in bottle	1737	g				
Total activity in bulk effluent	45675.1	CPM				
Total activity in simulated LAW effluent	7.73E+04	CPM, or	8.2%	of feed		

Feed Displacement

Tare mass of effluent bottle	26.2	g				
Final mass of effluent bottle	77.9	g				
Mass of feed processed	51.7	g or	44.3	mL		
Start date and time	12/14/01 10:38					
Finish date and time	12/14/01 12:08					
Average flow rate	29.5	mL/h or	2.8	BV/h		
Activity in bulk composite sample	18396	counts in	600	seconds of mass	5.838	g
Composite bulk activity concentration	315.1	CPM/g				
Total activity in effluent	1.63E+04	CPM, or	1.7%	of feed		
Bed height	5.7	cm				
Bed volume	10.1	mL				

Water Rinse

Tare mass of effluent bottle	26.6	g				
Final mass of effluent bottle	72.6	g				
Mass of feed processed	46.0	g, or	45.9	mL		
Start date and time	12/14/01 12:09					
Finish date and time	12/14/01 13:39					
Average flow rate	30.6	mL/h or	2.9	BV/h		
Activity in bulk composite sample	3426	counts in	1800	seconds of mass	5.011	g
Composite bulk activity concentration	22.8	CPM/g				
Total activity in effluent	1.05E+03	CPM, or	0.1%	of feed		
Bed height	5.9	cm				
Bed volume	10.4	mL				

Elution

Total mass of eluant processed	154.916	g				
Average flow rate	10.17	mL/h, or	1.0	BV/h		
Activity in bulk composite sample	14560	counts in	1800	seconds of mass	0.096	g
Composite bulk activity concentration	5055.6	CPM/g				
Total activity in effluent	7.83E+05	CPM, or	83.5%	of feed based on composite analysis		
Total activity in effluent	6.81E+05	CPM, or	72.6%	of feed based on eluant fraction analysis		
Bed height	4.0	cm				
Bed volume	7.1	mL				

Water Rinse

Tare mass of effluent bottle	25.6	g				
Final mass of effluent bottle	71	g				
Mass of feed processed	45.4	g, or	45.0	mL		
Start date and time	12/15/01 9:19					
Finish date and time	12/15/01 10:49					
Average flow rate	30.0	mL/h or	2.8	BV/h		
Activity in bulk composite sample	113	counts in	1800	seconds of mass	5.047	g
Composite bulk activity concentration	0.7	CPM/g				
Total activity in effluent	3.39E+01	CPM, or	0.0%	of feed		
Bed height	4.0	cm				
Bed volume	7.1	mL				

Total

Total activity in all effluents	877831.7	CPM
Total activity in feed	938118.3	CPM
Activity recovery, as fraction of feed	93.6%	

CYCLE 16 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	310.0	g		
Effluent bottle B tare	291.7	g		
Feed density	1.240	mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	6.58E-05	M		
Potassium concentration	8.00E-03	M		
Hydroxide concentration	1.2	M		
Nitrate concentration	0.5	M		
Nitrite concentration	2.46	M		
Flow rate	62.8	mL/h or	6.1	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	14654	300	6.325	575
Feed 2	15770	300	6.363	615
Average				595

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
1484.5	1174.5	1/8/02 16:30	1/9/02 7:56	61.37	952.4	92.8	2782	1800	6.479	5.225	17.748	3.089
1721.6	1411.6	1/9/02 8:01	1/9/02 11:02	63.38	1148.5	111.9	7524	1800	6.090	4.911	51.066	8.888
1986.6	1676.6	1/9/02 11:07	1/9/02 14:30	63.17	1367.9	133.2	19526	1800	6.965	5.617	115.876	20.167
459.7	1844.6	1/9/02 14:35	1/9/02 16:43	63.51	1508.7	147.0	14424	900	6.678	5.385	178.554	29.050
1648.6	3033.5	1/9/02 16:48	1/10/02 7:56	63.36	2473.0	240.9	39237	900	6.838	5.515	474.348	77.175
1892.1	3277.0	1/10/02 8:01	1/10/02 10:07	93.51	2674.6	260.5	26513	600	6.478	5.224	507.504	82.569
2118.9	3503.8	1/10/02 10:12	1/10/02 14:06	46.90	2862.7	278.9	13829	300	6.410	5.169	535.038	87.049

CYCLE 17 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	292	g		
Effluent bottle B tare	291.7	g		
Feed density	1.240	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	2.35E-04	M		
Potassium concentration	8.00E-03	M		
Hydroxide concentration	1.2	M		
Nitrate concentration	0.5	M		
Nitrite concentration	2.46	M		
Flow rate	30.0	mL/h or	2.9	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	15501	300	7.457	515.522
Feed 2	14987	300	7.457	498.428
Average				506.975

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
838.2	546.2	1/15/02 16:32	1/16/02 7:24	29.63	445.7	43.4	112	1800	6.479	5.225	0.715	0.141
1006.6	714.6	1/16/02 7:34	1/16/02 12:05	30.07	586.4	57.1	2060	1800	6.090	4.911	13.981	2.758
1167.9	875.9	1/16/02 12:15	1/16/02 16:35	30.02	722.1	70.3	12209	1800	6.965	5.617	72.454	14.291
1709.1	1417.1	1/16/02 16:45	1/17/02 7:18	30.00	1164.0	113.4	73593	1800	6.678	5.385	455.502	89.847
1900.6	1608.6	1/17/02 7:28	1/17/02 12:38	29.89	1323.9	129.0	13124	300	6.838	5.515	475.980	93.886
1973.5	1681.5	1/17/02 12:48	1/17/02 14:46	29.89	1387.9	135.2	13157	300	6.478	5.224	503.695	99.353
2100.8	1808.8	1/17/02 14:56	1/17/02 18:22	29.90	1495.8	145.7	13145	300	6.410	5.169	508.574	100.315

CYCLE 18 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	128.3	g
Effluent bottle B tare	291.7	g
Feed density	1.290	g/mL
Bed height in 0.25M NaOH	5.8	cm
Bed volume in 0.25M NaOH	10.3	mL
Cesium concentration	2.35E-04	M
Potassium concentration	8.00E-01	M
Hydroxide concentration	2.2	M
Nitrate concentration	1.36	M
Nitrite concentration	0.58	M
Flow rate	15.0	mL/h or 1.5 BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	14086	180	6.476	935
Feed 2	15393	180	6.476	1022
Average				979

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	CPM/mL	C/C0 (%)
410.4	282.1	1/22/02 17:00	1/23/02 7:53	14.69	223.3	21.8	628	1800	5.994	4.647	4.505	0.460
485.8	357.5	1/23/02 8:13	1/23/02 12:09	14.86	286.8	27.9	7674	1800	6.458	5.006	51.097	5.221
566.4	438.1	1/23/02 12:29	1/23/02 16:40	14.94	354.2	34.5	36348	1800	6.427	4.982	243.187	24.848
842.7	714.4	1/23/02 17:00	1/24/02 7:15	15.03	573.4	55.9	12818	180	6.433	4.987	856.792	87.545
946.7	818.4	1/24/02 7:35	1/24/02 12:57	15.02	659.0	64.2	13017	180	6.446	4.997	868.339	88.725
1051.6	923.3	1/24/02 13:17	1/24/02 18:40	15.11	745.4	72.6	14091	180	6.553	5.080	924.635	94.477

CYCLE 19 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	290.8	g		
Effluent bottle B tare	291.7	g		
Feed density	1.290	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	6.58E-05	M		
Potassium concentration	8.00E-01	M		
Hydroxide concentration	2.2	M		
Nitrate concentration	1.36	M		
Nitrite concentration	0.58	M		
Flow rate	63.7	mL/h or	6.2	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	16964	300	6.604	663
Feed 2	15732	300	6.506	624
Average				643

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
1756.2	1465.4	1/29/02 16:30	1/30/02 10:25	63.40	1141.1	111.2	15732	300	6.644	5.150	610.905	92.179
1964.0	1673.2	1/30/02 10:30	1/30/02 13:05	62.36	1308.9	127.5	18724	300	8.612	6.676	560.937	84.640
564.7	1946.2	1/30/02 13:10	1/30/02 16:28	64.13	1525.9	148.6	15825	300	6.925	5.368	589.581	94.505
1796.6	3178.1	1/30/02 16:33	1/31/02 7:25	64.24	2486.0	242.2	15449	300	6.720	5.209	593.131	95.074
2058.3	3439.8	1/31/02 7:30	1/31/02 10:41	63.73	2694.0	262.4	17557	300	6.567	5.091	689.768	110.564
2378.1	3759.6	1/31/02 10:46	1/31/02 14:39	63.84	2946.6	287.0	17236	300	6.023	4.669	738.318	118.346
2563.3	3944.8	1/31/02 14:44	1/31/02 17:00	63.34	3090.9	301.1	18651	300	7.561	5.861	636.418	102.012

CYCLE 20 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	292	g		
Feed density	1.260	g/mL		
Bed height in 0.25M NaOH	5.8	cm		
Bed volume in 0.25M NaOH	10.3	mL		
Cesium concentration	1.22E-04	M		
Potassium concentration	9.51E-02	M		
Hydroxide concentration	1.72	M		
Nitrate concentration	1.23	M		
Nitrite concentration	1.21	M		
Flow rate	29.7	mL/h or	2.9	BV/h

VOA sample mass	30.08	g, taken from	2/7/02 12:00	to	2/7/02 12:48
SVOA sample mass	23.062	g, taken from	2/7/02 12:48	to	2/7/02 13:25

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	11641	240	6.308	581
Feed 2	15002	300	6.308	599
Feed 3	12313	240	6.315	614
Feed 4	12102	240	6.315	604
Feed 5	12258	240	6.308	612
Feed 6	12349	240	6.315	616
Feed 7	12306	240	6.308	615
Feed 8	12208	240	6.315	609
Feed 9	11989	240	6.308	599
Feed 10	11957	240	6.315	596
Average				605

Effluent bottle mass (g)	Mass of feed collected in bottle & VOA/SVOA (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)	Activity in sample (CPM)
411.4	119.4	2/5/02 16:30	2/5/02 19:50	28.43	99.6	9.7	13	1800	6.135	4.869	0.089	0.015	0.43
530.9	238.9	2/5/02 20:00	2/5/02 23:10	29.95	199.5	19.4	17	1800	6.396	5.076	0.112	0.018	0.57
901.1	609.1	2/5/02 23:20	2/6/02 9:10	29.88	498.3	48.5	591	1800	6.221	4.937	3.990	0.660	19.70
1019.8	727.8	2/6/02 9:20	2/6/02 12:30	29.75	597.5	58.2	2550	1800	6.283	4.987	17.046	2.820	85.00
1139.0	847.0	2/6/02 12:40	2/6/02 15:50	29.87	697.1	67.9	8084	1800	6.315	5.012	53.765	8.894	269.47
1257.6	965.6	2/6/02 16:00	2/6/02 19:10	29.72	796.2	77.6	18160	1800	6.261	4.969	121.821	20.152	605.33
1376.2	1084.2	2/6/02 19:20	2/6/02 22:30	29.72	895.3	87.2	16292	900	6.292	4.994	217.503	35.980	1086.13
1793.9	1501.9	2/6/02 22:40	2/7/02 9:50	29.69	1231.6	120.0	18481	450	6.061	4.810	512.260	84.739	2464.13
1861.7	1569.7	2/7/02 10:00	2/7/02 11:50	29.35	1290.5	125.7	10840	240	6.356	5.044	537.225	88.868	2710.00
1926.8	1687.9	2/7/02 12:00	2/7/02 15:10	29.63	1389.3	135.3	11575	240	6.297	4.998	579.026	95.783	2893.75
2037.6	1798.7	2/7/02 15:20	2/7/02 18:20	29.31	1482.5	144.4	11857	240	6.539	5.190	571.181	94.485	2964.25

CYCLE 20 - ELUTION

0.5M HNO3 density 1.015 g/mL

Eluant mass processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
10.336	2/7/02 21:45	2/7/02 22:45	10.18	10.336	10.181	0.992	1180	1800	4.954	4.880	8.060	82	82	0.01241	0.01266
10.308	2/7/02 22:45	2/7/02 23:45	10.15	20.644	20.335	1.981	1990	1800	4.966	4.892	13.561	138	220	0.03322	0.02130
10.507	2/7/02 23:45	2/8/02 0:45	10.35	31.151	30.685	2.989	11078	900	0.096	0.095	7809.990	80831	81051	12.25226	12.26971
10.423	2/8/02 0:45	2/8/02 1:45	10.27	41.574	40.952	3.989	9726	120	0.092	0.091	53662.148	550946	631997	95.53764	84.30470
10.441	2/8/02 1:45	2/8/02 2:45	10.28	52.015	51.236	4.991	3150	1800	0.041	0.040	2599.902	26739	658736	99.57974	4.08452
10.525	2/8/02 2:45	2/8/02 3:45	10.37	62.540	61.604	6.001	20859	1800	5.049	4.973	139.804	1449	660185	99.79884	0.21964
10.589	2/8/02 3:45	2/8/02 4:45	10.43	73.129	72.034	7.017	8919	1800	4.909	4.836	61.483	641	660827	99.89578	0.09659
10.585	2/8/02 4:45	2/8/02 5:45	10.43	83.714	82.461	8.032	4551	1800	4.978	4.903	30.937	323	661149	99.94454	0.04860
11.806	2/8/02 5:45	2/8/02 6:52	10.41	95.520	94.090	9.165	2163	1800	5.002	4.927	14.633	170	661319	99.97027	0.02299
9.647	2/8/02 6:52	2/8/02 7:45	10.76	105.167	103.592	10.091	1141	1800	4.902	4.829	7.877	75	661394	99.98158	0.01237
10.410	2/8/02 7:45	2/8/02 8:45	10.25	115.577	113.847	11.090	672	1800	4.870	4.797	4.670	48	661442	99.98882	0.00734
10.930	2/8/02 8:45	2/8/02 9:48	10.25	126.507	124.613	12.138	455	1800	5.066	4.990	3.039	33	661475	99.99377	0.00477
10.158	2/8/02 9:48	2/8/02 10:45	10.53	136.665	134.619	13.113	265	1800	5.059	4.983	1.773	18	661493	99.99645	0.00278
10.575	2/8/02 11:45	2/8/02 12:45	10.42	157.748	155.386	15.136	131	1800	5.045	4.969	0.879	9	661502	99.99783	0.00138
7.460	2/8/02 15:03	2/8/02 15:45	10.50	189.447	186.611	18.178	79	1800	5.072	4.996	0.527	4	661506	99.99842	0.00083
10.581	2/8/02 15:45	2/8/02 16:45	10.42	200.028	197.033	19.193	80	1800	5.012	4.937	0.540	6	661511	99.99927	0.00085
10.653	2/8/02 16:45	2/8/02 17:45	10.49	210.681	207.527	20.215	68	1800	5.000	4.925	0.460	5	661516	100.00000	0.00072

VOA sample mass	24.239	g, taken from	2/8/2002 12:45	to	2/8/2002 15:03
SVOA sample mass	10.508	g, taken from	2/8/2002 10:45	to	2/8/2002 11:45

CYCLE 20 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	26	g				
Final mass of effluent bottle	57.7	g, and VOA SVOA samples	30.207	g		
Mass of feed processed	61.907	g, or	61.3	mL		
Start date and time	2/5/02 10:30					
Finish date and time	2/5/02 16:30					
Average flow rate	10.2	mL/h or	1.0	BV/h		
Bed height	5.8	cm				
Bed volume	10.3	mL				

Feed

Starting mass of feed bottle	2404.8	g				
Final mass of feed bottle	528	g				
Mass of feed processed	1877	g or	1489	mL		
Average flow rate	29.7	mL/h or	2.9	BV/h		
Acivity concentration in feed	479.8	CPM/g				
Total activity processed	9.00E+05	CPM				
Bed height	4.7	cm				
Bed volume	8.3	mL				

Simulated LAW Effluent

Total acitvity in samples	13098.8	CPM				
Estimated activity in VOA/SVOA samples	23539.6	CPM				
Activity in effluent bottle sample	9789	counts in	600	seconds of mass	6.297	g
Effluent bottle activity concentration	155.5	CPM/g				
Total mass of effluent in bottle	1746	g				
Total activity in bulk effluent	271362.2	CPM				
Total activity in simulated LAW effluent	3.08E+05	CPM, or	34.2%	of feed		

Feed Displacement

Tare mass of effluent bottle	26.3	g				
Final mass of effluent bottle	77.4	g				
Mass of feed processed	51.1	g or	44.4	mL		
Start date and time	2/7/02 18:35					
Finish date and time	2/7/02 20:05					
Average flow rate	29.6	mL/h or	2.9	BV/h		
Activity in bulk composite sample	17995	counts in	600	seconds of mass	5.755	g
Composite bulk activity concentration	312.7	CPM/g				
Total activity in effluent	1.60E+04	CPM, or	1.8%	of feed		
Bed height	5.7	cm				
Bed volume	10.1	mL				

Water Rinse

Tare mass of effluent bottle	25.8	g				
Final mass of effluent bottle	70.7	g				
Mass of feed processed	44.9	g, or	44.4	mL		
Start date and time	2/7/02 20:10					
Finish date and time	2/7/02 21:40					
Average flow rate	29.6	mL/h or	2.9	BV/h		
Activity in bulk composite sample	4020	counts in	1800	seconds of mass	5.051	g
Composite bulk activity concentration	26.5	CPM/g				
Total activity in effluent	1.19E+03	CPM, or	0.1%	of feed		
Bed height	6.0	cm				
Bed volume	10.6	mL				

Elution

Total mass of eluant processed	210.681	g				
Average flow rate	10.38	mL/h, or	1.0	BV/h		
Activity in bulk composite sample (first 15 samples only)	12613	counts in	1800	seconds of mass	0.096	g
Composite bulk activity concentration	4379.5	CPM/g				
Total activity in effluent	7.24E+05	CPM, or	80.4%	of feed based on composite analysis		
Total activity in effluent	6.62E+05	CPM, or	73.5%	of feed based on eluant fraction analysis		
Bed height	3.8	cm				
Bed volume	6.7	mL				

Water Rinse

Tare mass of effluent bottle	25.8	g				
Final mass of effluent bottle	70.9	g				
Mass of feed processed	45.1	g, or	44.7	mL		
Start date and time	2/8/02 17:45					
Finish date and time	2/8/02 19:15					
Average flow rate	29.8	mL/h or	2.9	BV/h		
Activity in bulk composite sample	38	counts in	1800	seconds of mass	5.05	g
Composite bulk activity concentration	0.3	CPM/g				
Total activity in effluent	1.13E+01	CPM, or	0.0%	of feed		
Bed height	3.3	cm				
Bed volume	5.8	mL				

Total

Total activity in all effluents	1049031.7	CPM
Total activity in feed	900299.5	CPM
Activity recovery, as fraction of feed	116.5%	

CYCLE 21 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	290.8	g		
Effluent bottle B tare	291.7	g		
Feed density	1.290	g/mL		
Bed height in 0.25M NaOH	5.9	cm		
Bed volume in 0.25M NaOH	10.4	mL		
Cesium concentration	6.58E-05	M		
Potassium concentration	8.00E-01	M		
Hydroxide concentration	2.2	M		
Nitrate concentration	1.36	M		
Nitrite concentration	0.58	M		
Flow rate	30.2	mL/h or	2.9	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	14090	300	7.512	484
Feed 2	13920	300	7.512	478
Average				481

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
863.6	572.8	2/12/02 16:36	2/13/02 7:28	29.87	449.1	43.0	24608	1800	6.527	5.060	162.118	33.501
1024.3	733.5	2/13/02 7:38	2/13/02 11:45	30.26	578.5	55.4	21005	900	6.179	4.790	292.350	60.413
1206.0	915.2	2/13/02 11:55	2/13/02 16:34	30.29	724.4	69.4	14758	420	6.612	5.126	411.326	86.037
1764.7	1473.9	2/13/02 16:44	2/14/02 7:07	30.11	1162.7	111.3	12882	300	6.688	5.184	496.943	103.945

CYCLE 22 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	291.7	g		
Effluent bottle B tare	291.6	g		
Feed density	1.290	g/mL		
Bed height in 0.25M NaOH	6	cm		
Bed volume in 0.25M NaOH	10.6	mL		
Cesium concentration	2.35E-04	M		
Potassium concentration	8.00E-01	M		
Hydroxide concentration	1.2	M		
Nitrate concentration	1.36	M		
Nitrite concentration	1.6	M		
Flow rate	62.5	mL/h or	5.9	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	8458	240	6.400	426
Feed 2	9540	300	6.179	398
Average				412

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
1756.2	1464.5	2/19/02 15:34	2/20/02 9:49	62.21	1140.4	107.4	10308	300	6.570	5.093	404.789	94.975
2118.8	1827.2	2/20/02 9:54	2/20/02 14:24	62.48	1426.9	134.4	10675	300	6.972	5.405	395.030	99.170

CYCLE 23 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	128.4	g		
Effluent bottle B tare	291.7	g		
Feed density	1.290	g/mL		
Bed height in 0.25M NaOH	6	cm		
Bed volume in 0.25M NaOH	10.6	mL		
Cesium concentration	6.58E-05	M		
Potassium concentration	8.00E-01	M		
Hydroxide concentration	1.2	M		
Nitrate concentration	1.36	M		
Nitrite concentration	1.6	M		
Flow rate	14.9	mL/h or	1.4	BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	10449	300	6.502	415
Feed 2	10364	300	6.502	411
Average				413

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
421.6	293.2	2/26/02 16:45	2/27/02 8:19	14.60	231.3	21.8	661	1800	5.201	4.032	5.465	1.323
576.3	447.9	2/27/02 8:39	2/27/02 16:38	15.02	356.2	33.5	7873	1800	6.396	4.958	52.930	12.818
865.9	737.5	2/27/02 16:58	2/28/02 8:00	14.93	585.7	55.1	41676	1800	6.446	4.997	278.012	67.327
1057.5	929.1	2/28/02 8:20	2/28/02 18:25	14.73	739.1	69.6	13650	420	6.264	4.856	401.580	97.251

CYCLE 24 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle A tare	332.2	g
Effluent bottle B tare	291.7	g
Feed density	1.290	g/mL
Bed height in 0.25M NaOH	5.7	cm
Bed volume in 0.25M NaOH	10.1	mL
Cesium concentration	6.58E-05	M
Potassium concentration	8.00E-01	M
Hydroxide concentration	1.2	M
Nitrate concentration	1.36	M
Nitrite concentration	1.6	M
Flow rate	29.7	mL/h or 2.9 BV/h

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	8723	300	6.450	349
Average				349

Effluent bottle mass (g)	Mass of feed processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)
987.7	655.5	3/5/02 15:25	3/6/02 9:50	27.59	513.2	50.9	10452	600	6.493	5.033	207.656	59.514
1142.6	810.4	3/6/02 10:00	3/6/02 13:05	38.94	638.1	63.2	9696	420	6.217	4.819	287.411	82.372
1269.9	937.7	3/6/02 13:15	3/6/02 16:25	31.16	741.5	73.5	7539	300	6.130	4.752	317.302	90.938

CYCLE 25 - SIMULATED AN-105 LAW PROCESSING

Effluent bottle tare	326.8	g				
Feed density	1.250	g/mL				
Bed height in 0.25M NaOH	5.6	cm				
Bed volume in 0.25M NaOH	9.9	mL				
Cesium concentration	1.22E-04	M				
Potassium concentration	9.51E-02	M				
Hydroxide concentration	1.72	M				
Nitrate concentration	1.23	M				
Nitrite concentration	1.21	M				
Flow rate	29.6	mL/h or	3.0	BV/h		
VOA sample mass	30.075	g, taken from	3/20/02 18:00	to	3/20/02 18:49	
SVOA sample mass	25.479	g, taken from	3/20/02 18:49	to	3/20/02 19:30	

	Counts	Count time (s)	Sample mass (g)	Sample activity (CPM/mL)
Feed 1	15461	300	6.451	599
Feed 2	15269	300	6.462	591
Feed 3	14948	300	6.451	579
Feed 4	14266	300	6.462	552
Feed 5	12607	240	6.451	611
Feed 6	11877	240	6.462	574
Average				584

Effluent bottle mass (g)	Mass of feed collected in bottle & VOA/SVOA (g)	Start time	Finish time	Volume flow rate (mL/hr)	Volume of feed processed (mL)	Bed volumes of feed processed	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	C/C0 (%)	Activity in sample (CPM)
466.8	140.0	3/19/02 8:30	3/19/02 12:26	28.47	116.4	11.7	5	1800	5.552	4.442	0.038	0.006	0.17
556.4	229.6	3/19/02 12:36	3/19/02 15:00	29.87	192.9	19.5	12	1800	6.015	4.812	0.083	0.014	0.40
679.5	352.7	3/19/02 15:10	3/19/02 18:30	29.54	296.3	29.9	25	1800	6.120	4.896	0.170	0.029	0.83
821.6	494.8	3/19/02 18:40	3/19/02 22:30	29.66	414.9	41.9	201	1800	6.154	4.923	1.361	0.233	6.70
1130.2	803.4	3/19/02 22:40	3/20/02 7:00	29.63	666.6	67.2	11746	1800	5.980	4.784	81.842	14.005	391.53
1277.8	951.0	3/20/02 7:10	3/20/02 11:10	29.52	789.6	79.7	35102	1800	6.217	4.974	235.255	40.258	1170.07
1400.9	1074.1	3/20/02 11:20	3/20/02 14:40	29.54	893.1	90.1	56551	1800	6.175	4.940	381.586	65.299	1885.03
1511.9	1185.1	3/20/02 14:50	3/20/02 17:50	29.60	986.8	99.6	23334	600	6.224	4.979	468.629	80.195	2333.40
1622.1	1350.9	3/20/02 18:00	3/20/02 22:30	29.47	1124.4	113.4	26834	600	6.248	4.998	536.852	91.870	2683.40
2053.3	1782.1	3/20/02 22:40	3/21/02 10:20	29.57	1474.4	148.7	23822	600	6.269	5.015	474.996	81.284	2382.20

CYCLE 25 - ELUTION

0.5M HNO3 density 1.015 g/mL

Eluant mass processed (g)	Start time	Finish time	Volume flow rate (mL/hr)	Cumulative eluant mass processed (g)	Cumulative eluant volume processed (mL)	Bed volumes of eluant generated	Counts	Count time (s)	Sample mass (g)	Sample volume (mL)	Sample activity (CPM/mL)	Total CPM in collected fraction	Cumulative CPM collected	Cumulative % of Cs collected	C/C0
10.224	3/21/02 13:32	3/21/02 14:32	10.07	10.224	10.071	0.981	1193	1800	5.047	4.971	7.999	80.558	80.558	0.01527	0.01257
10.211	3/21/02 14:32	3/21/02 15:32	10.06	20.435	20.129	1.961	1663	1800	5.019	4.944	11.213	112.777	193.335	0.03664	0.01762
10.287	3/21/02 15:32	3/21/02 16:32	10.13	30.722	30.262	2.948	9126	900	0.091	0.090	6787.337	68775.943	68969.278	13.07177	10.66309
10.413	3/21/02 16:32	3/21/02 17:32	10.26	41.135	40.519	3.947	21709	300	0.101	0.099	43641.538	447635.281	516604.559	97.91227	68.56205
10.333	3/21/02 17:32	3/21/02 18:32	10.18	51.468	50.697	4.938	2532	1800	0.099	0.098	865.484	8809.143	525413.703	99.58187	1.35970
10.285	3/21/02 18:32	3/21/02 19:32	10.13	61.753	60.828	5.925	16038	1800	5.093	5.017	106.563	1079.592	526493.294	99.78649	0.16741
10.343	3/21/02 19:32	3/21/02 20:32	10.19	72.096	71.017	6.918	7509	1800	5.103	5.027	49.795	507.320	527000.614	99.88264	0.07823
10.313	3/21/02 20:32	3/21/02 21:32	10.16	82.409	81.175	7.907	4084	1800	5.087	5.011	27.168	275.986	527276.601	99.93495	0.04268
10.299	3/21/02 21:32	3/21/02 22:32	10.14	92.708	91.320	8.895	2104	1800	5.108	5.032	13.939	141.406	527418.007	99.96175	0.02190
10.311	3/21/02 22:32	3/21/02 23:32	10.16	103.019	101.477	9.885	1182	1800	5.101	5.025	7.841	79.642	527497.649	99.97684	0.01232
10.406	3/21/02 23:32	3/22/02 0:32	10.25	113.425	111.727	10.883	596	1800	5.121	5.044	3.938	40.370	527538.018	99.98449	0.00619
10.487	3/22/02 0:32	3/22/02 1:32	10.33	123.912	122.057	11.889	366	1800	5.133	5.056	2.413	24.925	527562.944	99.98922	0.00379
10.498	3/22/02 1:32	3/22/02 2:32	10.34	134.410	132.398	12.897	254	1800	5.047	4.971	1.703	17.611	527580.555	99.99255	0.00268
10.392	3/22/02 2:32	3/22/02 3:32	10.24	144.802	142.634	13.894	167	1800	5.057	4.981	1.118	11.439	527591.994	99.99472	0.00176
10.474	3/22/02 3:32	3/22/02 4:32	10.32	155.276	152.951	14.899	109	1800	5.085	5.009	0.725	7.484	527599.478	99.99614	0.00114
5.221	3/22/02 6:55	3/22/02 7:25	10.29	185.232	182.459	17.773	70	1800	5.221	5.143	0.454	2.333	527601.811	99.99658	0.00071
5.176	3/22/02 9:00	3/22/02 9:30	10.20	206.929	203.831	19.855	64	1800	5.176	5.099	0.418	2.133	527603.945	99.99699	0.00066
20.614	3/22/02 9:30	3/22/02 11:30	10.15	227.543	224.136	21.833	63	1800	5.130	5.053	0.416	8.438	527612.383	99.99859	0.00065
20.704	3/22/02 11:30	3/22/02 13:30	10.20	248.247	244.530	23.819	54	1800	4.999	4.924	0.366	7.455	527619.838	100.00000	0.00057

VOA sample mass 24.735 g, taken from 3/22/2002 4:32 to 3/22/2002 6:55
SVOA sample mass 16.521 g, taken from 3/22/2002 7:25 to 3/22/2002 9:00

CYCLE 25 - OPERATIONAL DETAILS AND ACTIVITY BALANCE

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	26	g				
Final mass of effluent bottle	51	g, and VOA SVOA samples	39.494	g		
Mass of feed processed	64.494	g, or	63.9	mL		
Start date and time	3/12/02 10:45					
Finish date and time	3/12/02 16:45					
Average flow rate	10.6	mL/h or	1.1	BV/h		
Bed height	5.6	cm				
Bed volume	9.9	mL				

Feed

Volume of feed processed	1474.4	mL				
Average flow rate	29.6	mL/h or	3.0	BV/h		
Activity concentration in feed	584.4	CPM/mL				
Total activity processed	8.62E+05	CPM				
Bed height	4.5	cm				
Bed volume	8.0	mL				

Simulated LAW Effluent

Total acitvity in samples	10853.7	CPM				
Estimated activity in VOA/SVOA samples	10247.5	CPM				
Activity in effluent bottle sample	4884	counts in	1800	seconds of mass	6.395	g
Effluent bottle activity concentration	25.5	CPM/g				
Total mass of effluent in bottle	1726.5	g				
Total activity in bulk effluent	43952.2	CPM				
Total activity in simulated LAW effluent	6.51E+04	CPM, or	7.6%	of feed		

Feed Displacement

Tare mass of effluent bottle	26.1	g				
Final mass of effluent bottle	77	g				
Mass of feed processed	50.9	g or	42.3	mL		
Start date and time	3/21/02 10:31					
Finish date and time	3/21/02 12:01					
Average flow rate	28.2	mL/h or	2.8	BV/h		
Activity in bulk composite sample	23831	counts in	600	seconds of mass	6.014	g
Composite bulk activity concentration	396.3	CPM/g				
Total activity in effluent	2.02E+04	CPM, or	2.3%	of feed		
Bed height	5.2	cm				
Bed volume	9.2	mL				

Water Rinse

Tare mass of effluent bottle	25.8	g				
Final mass of effluent bottle	70.3	g				
Mass of feed processed	44.5	g, or	44.0	mL		
Start date and time	3/21/02 12:02					
Finish date and time	3/21/02 13:32					
Average flow rate	29.6	mL/h or	3.0	BV/h		
Activity in bulk composite sample	4585	counts in	1800	seconds of mass	5.052	g
Composite bulk activity concentration	30.3	CPM/g				
Total activity in effluent	1.35E+03	CPM, or	0.2%	of feed		
Bed height	5.7	cm				
Bed volume	10.1	mL				

Elution

Total mass of eluant processed	245	g				
Average flow rate	10.21	mL/h, or	1.0	BV/h		
Activity in bulk composite sample (first 15 samples only)	10648	counts in	1800	seconds of mass	0.088	g
Composite bulk activity concentration	4033.3	CPM/g				
Total activity in effluent	8.35E+05	CPM, or	96.9%	of feed based on composite analysis		
Total activity in effluent	5.28E+05	CPM, or	61.2%	of feed based on eluant fraction analysis		
Bed height	3.3	cm				
Bed volume	5.8	mL				

Water Rinse

Tare mass of effluent bottle	25.8	g				
Final mass of effluent bottle	70.3	g				
Mass of feed processed	44.5	g, or	43.8	mL		
Start date and time	3/22/02 13:32					
Finish date and time	3/22/02 15:02					
Average flow rate	29.2	mL/h or	2.9	BV/h		
Activity in bulk composite sample	49	counts in	1800	seconds of mass	5.076	g
Composite bulk activity concentration	0.3	CPM/g				
Total activity in effluent	1.43E+01	CPM, or	0.0%	of feed		
Bed height	3.1	cm				
Bed volume	5.5	mL				

Total

Total activity in all effluents	921447.2	CPM
Total activity in feed	861588.9	CPM
Activity recovery, as fraction of feed	106.9%	

Appendix B: Statistical Analysis of Breakthrough Performance

The goal of the analyses is to explain variation in the Lambda values based on the values of the listed factors. Cycle is the order in which the experimental trials are performed, and Lambda tends to decrease throughout the cycle order. The remaining four factors were varied in the experimental trials as indicated below.

The following data were used in the analyses to be discussed. Note that the runs with Cycle numbers 2, 16, 17, and, 22 could not be used due to missing information. In addition the Cycle 1 run is a significant outlier that has considerable influence on the results. One might question the validity of this initial run when considered in the initial sequence below for Cycles 1, 5, 10, 15, 20, and 25, where the other factors were not varied. This gives a good estimate of the influence of the Cycle order, and when the associated Lambda values are observed for these Cycle runs, the unusual value of 120 for Lambda under Cycle 1 can be seen. For this reason it was omitted from subsequent analyses.

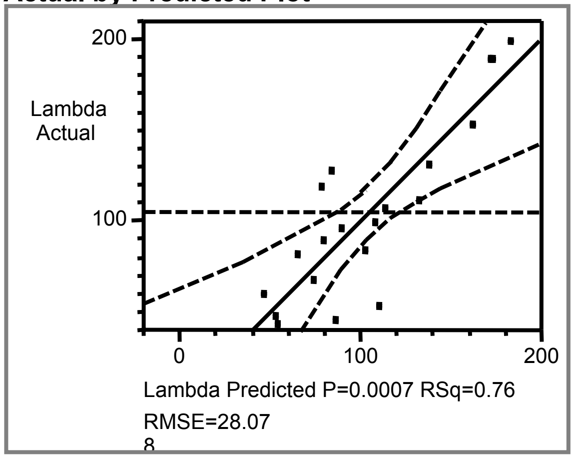
Cycle	Cs^+	K^+	OH^-	Flowrate	Lambda
1	13	0.095	1.7	3	120
5	13	0.095	1.7	3	153
10	13	0.095	1.7	3	132
15	13	0.095	1.7	3	108
20	13	0.095	1.7	3	96
25	13	0.095	1.7	3	82
11	25	0.008	2.2	6	120
22	25	0.8	1.2	6	.
18	25	0.8	2.2	1.5	128
14	25	0.008	1.2	1.5	100
12	7	0.008	2.2	1.5	190
23	7	0.8	1.2	1.5	90
19	7	0.8	2.2	6	44
16	7	0.008	1.2	6	.
13	25	0.008	2.2	3	54
17	25	0.008	1.2	3	.
24	7	0.8	1.2	3	48
21	7	0.8	2.2	3	46
2	.	.	.	.	120
3	7	0.095	1.7	3	200
4	10	0.095	1.7	3	190
6	25	0.095	1.7	3	112
7	37	0.095	1.7	3	84
8	49	0.095	1.7	3	68
9	60	0.095	1.7	3	60

Linear terms only – The first model considered includes only the linear terms for the five factors. The following plot gives a visual image of how well predicted values from this model match up with the values actually observed. The curved outside bands indicate the magnitude of variability in Lambda that could not be explained. Better models would have most points falling very close to the center line with very little spread between the outside bands. A very poor fit would have points exhibiting “shotgun” scatter and very wide bands. The curved lines should contain 95% of the points, so the excessive variability of the points in the center range of Lambda show that this is not a very good model for fitting the data.

Response Lambda

Whole Model

Actual by Predicted Plot



The model obtained for linear terms only is given by the following parameter estimates as obtained from the next table. Each estimate is the coefficient of the corresponding factor. For a given set of factor values, the predicted Lambda value would be approximately

Lambda = 225.89 – 4.79x**Cycle** – 2.04x**Cs** – 28.68x**K** + 17.94x**OH** – 13.86x**Flowrate**.

The impact on Lambda of the changing the factor levels can thus be observed. For example each consecutive cycle tended to have a Lambda value approximately 4.79 lower. Increasing hydroxide tended to increase Lambda (positive coefficient) while increases in the other factors tended to lower Lambda (negative coefficients).

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	225.88765	41.07851	5.50	<.0001
Cycle	-4.794881	1.223117	-3.92	0.0015
Cs^+	-2.042241	0.469985	-4.35	0.0007
K^+	-28.68083	25.94845	-1.11	0.2877
OH^-	17.943222	20.98828	0.85	0.4070
Flowrate	-13.85837	5.887821	-2.35	0.0337

The remaining columns in the above table reflect the “statistical significance” of the influence of the various factors. (Note that the intercept is the value expected when all factors are at level zero; this has essentially no meaning in the current application.) The “Prob>|t|” column is called a “p-value” or “significance level” of the factor. If in fact Lambda and a factor have no true relationship, this number would vary randomly between zero and one. However, the stronger the relationship, the closer to zero the value would be. Thus small values indicate statistical significance. Typically the value 0.05 is used as the borderline case for indicating significance with factors having p-values less than that being significant. Those with p-values slightly greater are considered marginally significant, and those with p-values above, say, 0.15 are considered not significant.

In the above, Cycle and Cs^+ would be considered extremely significant, Flowrate significant, and K and OH not significant.

The following tables give the overall summary of the model fit. The R-square value of 0.755 suggests that about 75.5% of the variation in Lambda can be explained by the influence of the varying factors (linear terms only). 24.5% of the variation thus remains unexplained and perhaps could be better explained through more complex models, which will be discussed below. The adjusted R-square value takes into consideration the number of terms included in the model, that is, the more terms, the lower the adjusted R-square values would be relative to the R-square value. The Root Mean Square Error (RMSE) is a standard deviation type measure of the variability one might expect around the predicted model Lambda values. A plus or minus two standard deviation range is therefore about ± 56.

The "Prob>F" value in the second table below gives a measure of the significance of the overall model. It too is a p-value, so being very small, it indicates the extreme statistical significance of the ability of the factors to explain why Lambda is varying.

Summary of Fit

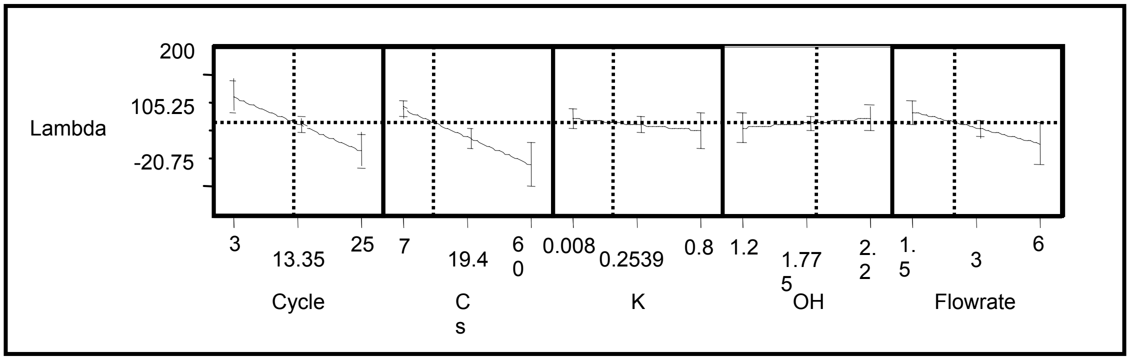
RSquare	0.755092
RSquare Adj	0.667625
Root Mean Square Error	28.07763
Mean of Response	105.25
Observations (or Sum Wgts)	20

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	34028.805	6805.76	8.6329
Error	14	11036.945	788.35	Prob > F
C. Total	19	45065.750		0.0007

The figure below gives a graphical depiction of the significance of the factors. The slopes of the following lines indicate whether the influence is an increase or decrease, and the magnitude of the change between factor levels shows the significance of the factor. As indicated above, the most important factors are Cycle and Cs$^+$, next is Flowrate, with K$^+$ and OH$^-$ contributing very little.

Prediction Profiler



In summary, the model with only linear terms is relatively good with fairly large R-square, moderate RMSE, and several factors significant. An improvement for the model would be to consider "interaction terms", that is, products of the factors. A Cs$^+$ by K$^+$ interaction, for example, would be a measure of the combined impact of the two factors. For example, the influence of Cs on Lambda may depend on whether high or low levels of K are present. The linear-only model assumes this is not the case and that the influence of all the factors is simply "additive", that is, the increases or decreases due to each factor simply accumulate in an additive fashion regardless of how the factors interact with each other.

Additional terms that may improve the explanation of variability in Lambda are the second order terms, that is, the square of the linear terms. Such terms would be important if Lambda does not increase linearly with a changing factor. Instead Lambda may initially increase with increasing factor levels when such levels are low, but then may decrease with increasing factor levels when such levels are high.

The number and particular selection of specific experimental trials used do not support including all such interaction and quadratic terms with the linear terms. Instead a stepwise approach is needed to add the more significant contributing factors to the model first. In such a stepwise approach, if an interaction term is included, so too will be the two corresponding linear terms. And if a quadratic term is included, the corresponding linear term will be as well. The stepwise approach thus successively picks the groups of factors that are most significant in explaining the changes in Lambda.

The following tabled values indicate that p-values had to be less then 0.25 for the terms to be entered into the model. Then when the resulting model is evaluated, p-values had to be less than 0.15 for the associated terms to stay in the model, both for the newly entered terms and the ones that had already been in the model.

Stepwise Regression Control

Prob to Enter	0.250
Prob to Leave	0.150

The following gives the sequence of steps for these data as various factors are entered and/or removed. The gradual improvement in R-square can be observed. When terms are removed, R-square will in fact decrease somewhat, but not a statistically significant amount.

Step History

Step	Parameter	Action	"Sig Prob"	Seq SS	RSquare	Cp	p
1	Cycle*Cs	Entered	0.0001	33349.54	0.7400	289.88	4
2	Cs*Flowrate	Entered	0.0223	4910.754	0.8490	167.35	6
3	Cs*K	Entered	0.1104	2091.739	0.8954	117.45	8
4	Cycle*Cs	Removed	0.7063	58.50815	0.8941	116.96	7
5	Cs*Cs	Entered	0.0306	1591.791	0.9294	77.948	8
6	Flowrate*Flowrate	Entered	0.0034	1772.448	0.9688	34.278	9
7	OH	Entered	0.0041	813.9692	0.9868	15.305	10
8	Cycle*Cycle	Entered	0.0407	230.335	0.9919	11.371	11
9	K*OH	Entered	0.0571	138.7414	0.9950	9.7957	12

In Step 1, the Cycle by Cs interaction, and thus the associated linear terms, Cycle and Cs, enter the model. This gives R-square of 0.74, almost as large as that of the model considered earlier that includes just the linear terms, but all of them. Apparently the variability explained by this interaction term can also be explained by other interaction terms that enter the model in subsequent steps since in Step 4 the Cycle by Cs interaction term is removed.

Note by end of the stepwise process, R-square has increased to 0.995. One might question the importance of the terms added in the later steps since the increase in R-square is minimal even though it is statistically significant. In fact, at Step 6, R-square has already reached 0.969, and at that point, OH has not entered the model. This suggests little would be lost in omitting OH completely, and this is done in the following. A similar stepwise approach is used but without terms involving OH.

Step History

Step	Parameter	Action	"Sig Prob"	Seq SS	RSquare	Cp	p
1	Cycle*Cs	Entered	0.0001	33349.54	0.7400	289.88	4
2	Cs*Flowrate	Entered	0.0223	4910.754	0.8490	167.35	6
3	Cs*K	Entered	0.1104	2091.739	0.8954	117.45	8
4	Cycle*Cs	Removed	0.7063	58.50815	0.8941	116.96	7
5	Cs*Cs	Entered	0.0306	1591.791	0.9294	77.948	8
6	Flowrate*Flowrate	Entered	0.0034	1772.448	0.9688	34.278	9
7	Cycle*Cycle	Entered	0.0058	774.1746	0.9859	16.331	10

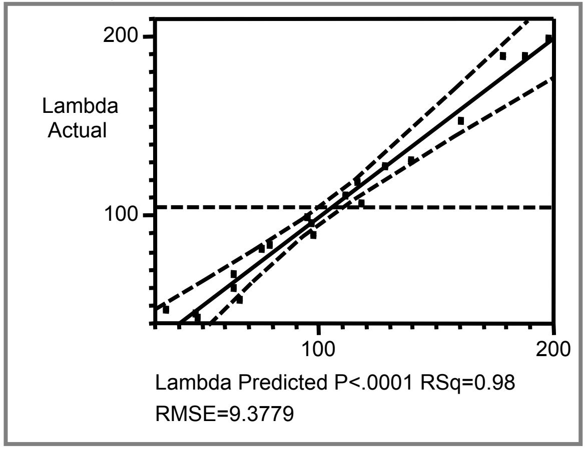
In this sequence of steps, once the Cycle by Cs interaction is removed, no other interactions involving Cycle enter the model, and the Cycle second order terms first enters in Step 7 with minimal impact. This suggests that simply including Cycle as a linear term and still omitting OH would likely be sufficient to give a reasonable model. This is done in the following stepwise sequence, that is, no OH terms are considered as candidates to enter the model and Cycle can enter only as a linear term.

Step History

Step	Parameter	Action	"Sig Prob"	Seq SS	RSquare	Cp	p
1	Cs*K	Entered	0.0009	28450.77	0.6313	162.22	4
2	Cycle	Entered	0.0007	9019.732	0.8315	69.641	5
3	Cs*Cs	Entered	0.0334	2159.446	0.8794	48.998	6
4	Flowrate*Flowrate	Entered	0.0137	2777.243	0.9410	23.877	8
5	K*Flowrate	Entered	0.0024	1547.622	0.9753	9.6489	9
6	K*K	Entered	0.1358	231.4857	0.9805	9.2216	10

A pretty reasonable model that is somewhat simplified is thus obtained. The features of the complete model that corresponds to this stepwise result is given in the following.

Response Lambda
Whole Model
Actual by Predicted Plot



The above figure indicates the improvement over the corresponding figure for the model with only linear terms considered at the beginning of this discussion. R-square has increased from 0.755 to 0.980. The RMSE has decreased from 28.08 to 9.38. A plus or minus two standard deviation range (that one might expect around the predicted model Lambda values) is therefore decreased from about ± 56 in the first model discussed to ± 18.8. A small amount of excessive variability in the middle Lambda range remains (where points fall outside the 95% bands), but it is not nearly as severe as before.

Summary of Fit

RSquare	0.980485
RSquare Adj	0.962922
Root Mean Square Error	9.377924
Mean of Response	105.25
Observations (or Sum Wgts)	20

Note that the prediction equation is considerably more complex. It can be obtained from the following parameter coefficient estimates. Note also that K and the quadratic term using K are not particularly useful, but the interaction terms involving K are. If all terms involving K were to be omitted from the model, R-square would drop to about 0.77.

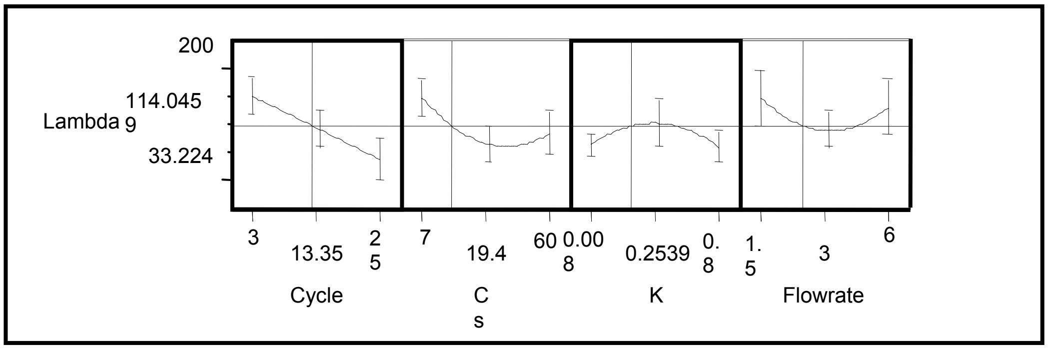
Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	350.03016	19.60135	17.86	<.0001
Cycle	-4.252866	0.426905	-9.96	<.0001
Cs	-6.579024	0.77429	-8.50	<.0001
Cs*Cs	0.0583321	0.011213	5.20	0.0004
K	105.31377	119.9295	0.88	0.4005
K*K	-215.322	132.719	-1.62	0.1358
Flowrate	-58.38979	12.69277	-4.60	0.0010

Term	Estimate	Std Error	t Ratio	Prob>|t|
Flowrate*Flowrate	8.0576442	1.631282	4.94	0.0006
Cs*K	6.5570604	1.139683	5.75	0.0002
K*Flowrate	-21.04764	4.94629	-4.26	0.0017

The profiles below show the quadratic nature of the relationships between Lambda and the factors.

Prediction Profiler



In summary, a model based on only linear terms for the five factors can only explain about 75% of the variability in the Lambda values. Adding interaction and quadratic terms considerably improves this. OH is not needed since it only adds minimal explanation of Lambda variability. Cycle need only be included as a linear term. A good model for explaining the variability of Lambda based on Cycle, Cs, K, and Flowrate (including interaction and quadratic terms involving the last three factors) was derived. It appears to have strong predictive capability with R-square value 0.96 and RMSE of about only 9.4.

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