

**Ion Exchange Removal of Cesium from
Simulated and Actual Supernate from
Hanford Tanks 241-SY-101 and 241-SY-103**

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Summary

Pacific Northwest Laboratory (PNL)^(a), in conjunction with the Process Chemistry and Statistics Section of Westinghouse Hanford Company (WHC), conducted this study as part of the Supernatant Treatment Development Task for the Initial Pretreatment Module (IPM) Applied Engineering Project. The study assesses the performance of the CS-100 ion exchange material for removing cesium from simulated and actual alkaline supernate from Hanford tanks 241-SY-101 and 241-SY-103 (hereinafter identified as tanks 101-SY and 103-SY). The objective of these experiments is to compare the cesium ion exchange loading and elution profiles of actual and simulated wastes. Specific experimental objectives include 1) demonstration of decontamination factors (DF) for cesium removal, 2) verification of simulant performance, 3) investigation of waste/exchanger chemistry, and 4) determination of the radionuclide content of the regenerated CS-100 resin prior to disposal.

The experiments were conducted using single, small columns containing ~ 12 mL of CS-100, a phenol-formaldehyde resin. Simulants were developed based on available analytical data, and shake-down testing with simulated tank wastes was conducted prior to the actual waste tests. The process steps generally followed those expected in a full-scale process. These steps included 1) resin preparation in caustic, 2) loading, 3) caustic wash to remove residual waste and prevent the precipitation of $\text{Al}(\text{OH})_3$, 4) water wash to remove caustic, 5) elution with nitric acid, 6) water wash to remove nitric acid, and 7) regeneration with caustic.

The results described in this report demonstrate that radioactive cesium from actual or simulated supernatant liquids can be removed by passing the solution through a cesium-selective material (e.g., CS-100 ion exchange resin). Cesium can be recovered and concentrated into a smaller volume by elution of the resin with dilute nitric acid. Although not specifically investigated in this study, the resin can then be re-used several times by initiating multiple load and elute cycles. The following specific results and conclusions were obtained from these experiments:

- The removal of cesium from actual and simulated 101-SY and 103-SY tank waste was demonstrated using ~ 12-mL columns containing CS-100 ion exchange material. DFs greater than 1000 were obtained for the first seven column volumes (CV) of 101-SY and the first five CV of 103-SY feed processed. These experiments demonstrate that, in principle, fairly large DFs can be achieved with actual waste and fresh CS-100 material (first cycle loading).
- The CS-100 column was effectively eluted to 0.1 C/C_0 with approximately 4 CV of dilute (0.5 M) nitric acid.

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- Cesium loading breakthrough curves generated for the simulated and actual tank waste were very similar and indicate an ability to mimic cesium ion exchange with the intelligent formulation of nonradioactive simulant solutions.
- The ion exchange process appeared to have little effect on the ^{90}Sr , ^{99}Tc , and total alpha (AT) content of the wastes. As expected, very little ($<0.001\%$) of the ^{90}Sr was removed from the waste during the loading step. Based on an analysis of the eluant solutions, the resin appears to concentrate cesium and nickel but has limited affinity for Al, Cr, K, and Sr.
- After elution, stripping the resin with 3 M HNO_3 released additional residual cesium and other components. After the actual waste elution cycle, approximately 0.085- and 0.50-Ci ^{137}Cs m^{-3} remained on the stripped (101-SY) and unstripped (103-SY) resins, respectively. These values correspond to a residual cesium level of 1.35E-07 and 8.36E-07 mmol per gram of dried resin.

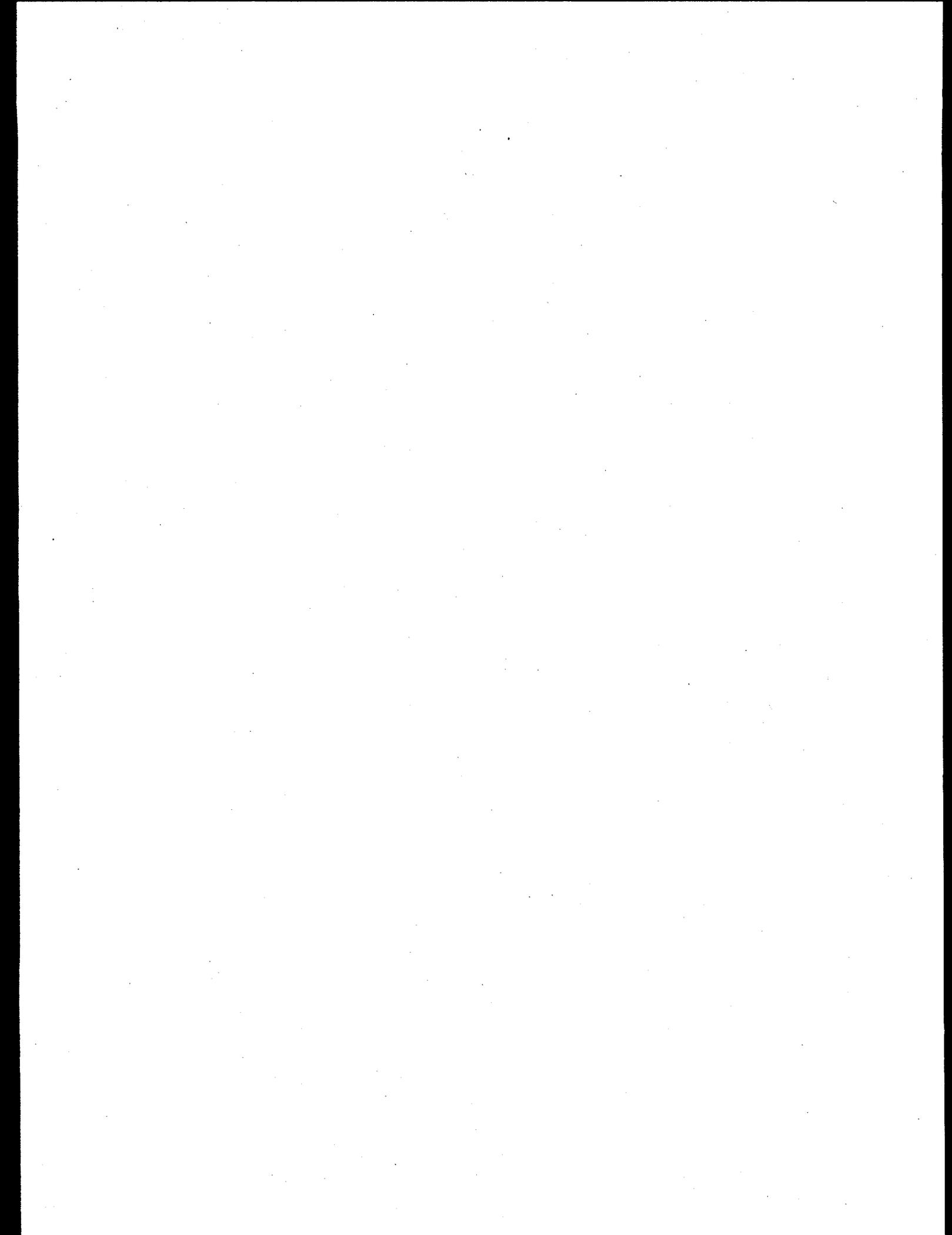
Based on the analytical data obtained during the experiment, several ion exchange loading and elution parameters can be calculated as described in Table S.1.

Table S.1. Ion Exchange Loading and Elution Data and Parameters for Actual and Simulated Waste from Tanks 101-SY and 103-SY

Parameter	Tank 101-SY		Tank 103-SY	
	Actual	Simulated	Actual	Simulated
Cs Loading (mmol g^{-1})	8.43E-03	8.26E-03	7.27E-03	9.97E-03
Cs Loading (mmol mL^{-1})	1.92E-03	1.81E-03	1.58E-03	2.19E-03
Load Volume to 0.5 C/C_0 (CV)	4.06E+01	4.14E+01	3.60E+01	3.68E+01
Elute Volume to 0.1 C/C_0 (CV)	4.20E+00	3.50E+00	4.20E+00	3.70E+00
Elute Volume to Peak (CV)	3.12E+00	2.54E+00	3.00E+00	2.64E+00
Elution Peak (C/C_0)	3.77E+01	6.00E+01	2.80E+01	4.94E+01
Mass Balance (%)	9.86E+01	9.51E+01	7.93E+01	8.78E+01
Residual Cesium (Ci m^{-3})	8.50E-02		5.00E-01	
Sodium (<u>M</u>)	4.84E+00	5.00E+00	4.86E+00	5.00E+00
Cesium (<u>M</u>)	4.74E-05	4.20E-05	5.40E-05	5.30E-05
Na/Cs Ratio (<u>M</u>)	1.02E+05	1.20E+05	9.00E+04	9.43E+04

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1.0 Introduction

1.1 Background

The contents of Hanford's 177 underground storage tanks (UST) include a mixture of sludge, salt cake, and alkaline supernatant liquid. The salt cake, generated by extensive evaporation of aqueous solution, consists primarily of damp sodium salts. The supernate consists of concentrated aqueous solutions of sodium nitrate/nitrite salts, with smaller quantities of hydroxide, aluminum, potassium, carbonate, sulfate, and phosphate. The bulk of the water-soluble radionuclides such as ^{137}Cs are contained in the interstitial liquid of the salt cake and supernatant liquid fractions. The insoluble sludge fraction of the waste consists of metal oxides and hydroxides and contains the bulk of the ^{90}Sr and many of the transuranic radionuclides (TRU).

1.2 Cesium Decontamination Requirements

Although the pretreatment and disposal strategy is still being defined, one of the first steps in most pretreatment scenarios will be a solids/liquid separation of the pumpable waste liquor, followed by ion exchange removal of cesium from the resulting supernatant liquid. Next, a salt cake dissolution and sludge wash step will be initiated, followed by another solids/liquid separation. Most of the cesium is expected to be in the aqueous liquids from these processes, and it is these solutions that are the focus of the cesium ion exchange removal process. Specific decontamination requirements will depend upon the composition of the waste being pretreated and future decisions made by the U.S. Department of Energy (DOE) with respect to the amount of cesium that will be acceptable in the low activity waste disposal form. The separated cesium will be concentrated and vitrified with the high-level waste sludge in the high-level waste repository. The decontaminated supernatant liquid and salt cake fractions will be immobilized in a low activity waste form.

1.3 Objectives

The overall objectives of the Cesium Ion Exchange Testing Task of the PNL Tank Waste Remediation System (TWRS) Initial Pretreatment Module (IPM) Applied Engineering Project are to 1) evaluate available materials for the ion exchange recovery of cesium from alkaline wastes, 2) determine the loading and elution efficiency of these processes, 3) determine the physical life cycle (including radiation and chemical stability) of these materials, and 4) develop basic ion exchange data that can be applied to a broad range of tank wastes.

The primary goal of the experiments described in this report is to determine the cesium loading and elution efficiency for the baseline ion exchange material (CS-100) in actual Hanford alkaline waste supernate and compare these results to those obtained with simulated solutions. The specific experimental objectives described in this report are to 1) demonstrate decontamination factors for cesium

removal, 2) investigate the potential impact of exchanger fouling/poisoning, 3) verify simulant performance, 4) investigate waste/exchanger chemistry, and 5) determine radionuclide content of regenerated resin prior to disposal.

1.4 Scope

The work described in this report involves the ion exchange removal of cesium from simulated and actual Hanford tank waste using the current project baseline material (CS-100). The experimental parameters tested were constrained by the availability of actual waste (volume and type) and do not necessarily reflect optimum column dimension, size, material, or solutions. To achieve the test objectives, small volumes of actual waste composite from Hanford tanks 241-SY-101 and 241-SY-103 (hereinafter identified as tanks 101-SY and 103-SY) were diluted with 2.0 M NaOH to approximately 5 M Na and 5.0E-05 M Cs. These solutions were passed through separate 12-mL CS-100 ion exchange columns and the extent of material efficiency was determined by monitoring the column effluent for ^{137}Cs , ^{90}Sr , ^{99}Tc , total alpha (AT), total organic carbon (TOC), and various metals. The columns were then washed with caustic and water before eluting the cesium with 0.5 M HNO_3 .

2.0 Experimental Approach

The Ion Exchange Loading and Elution task was designed to provide a small-scale demonstration of the baseline supernatant pretreatment process for cesium removal using simulated and actual Hanford waste. Small volumes of actual and simulated complexant concentrate (CC) waste from tanks 101-SY and 103-SY (ca. 200-mL in-tank) were decontaminated by passing the solutions through ~12-mL columns of a cesium-selective ion exchange material (CS-100). Cesium and small amounts of other components exchanged with Na^+ during the loading cycle and were eluted with dilute nitric acid into a smaller solution volume. The simulant experiments were completed by PNL personnel in the 325 Building. The actual waste tests were completed by WHC personnel at the 222-S laboratory under the direction of PNL. The resin performance was evaluated by determining the cesium concentration exiting the column by gamma energy analysis (GEA) as a function of solution volume passed through the material. The material loading (mmol Cs per gram of resin) was calculated from the loading curve and the mass balance was determined by comparing the loading and elution data. In addition, the loading and elution characteristics of other radionuclides (e.g., Sr, Pu, Am, etc.), nonradioactive species (e.g., Na, K, Fe, Ca, Ni, Mg, Mn, Zn, Zr, etc.), and the adsorption of organics on the resin were evaluated similarly.

Table 2.1 summarizes the experimental parameters and the specific conditions used during each separate waste loading and elution test. The process steps generally followed those expected in a full-scale process. These steps included 1) resin preparation with caustic, 2) loading, 3) caustic wash to remove residual waste and prevent the precipitation of $\text{Al}(\text{OH})_3$, 4) water wash to remove caustic, 5) elution with nitric acid, 6) water wash to remove nitric acid, and 7) regeneration with caustic. During two of the runs, a stripping step with 3.0 M nitric acid was used to assess the extent to which residual exchanged components could be removed after the elution step. This step would not be used during an actual operation because of its deleterious effect on the organic resin. The following sections provide additional details on this and other steps.

Because of the limited availability of actual radioactive waste and experimental costs associated with using radioactive materials, replicate experiments (12-mL column volume (CV) loading and elution tests) were completed using simulated solutions. Before using actual waste in any process (e.g., Cs ion exchange), it is imperative to complete adequate testing with simulated solutions to accurately assess process performance, safety, and hazardous conditions, and to provide personnel with on-the-job training or experience with a mock experimentation process. Experimental data and procedures for the actual and simulant waste experiments can be found in controlled laboratory notebooks WHC-N-384-2 (pp. 90-126) and BNW55750 (pp. 1-95), respectively.

Table 2.1. Summary of Experimental Parameters and Conditions for the CS-100 Column Ion Exchange Loading and Elution Experiment

Parameter	Simulant Waste Test		Actual Waste Test	
	101-SY	103-SY	101-SY	103-SY
"As Received" CS-100 Weight (g)	4.354	4.354	4.363	4.367
CS-100 Resin F-Factor (g/g)	0.6148	0.6148	0.6148	0.6148
CS-100 Column Volume (mL)	12.2	12.2	11.8	12.3
In-tank Waste Volume (mL)	NA ^(a)	NA	200	200
Diluted Feed Volume (mL)	826	925	684	434
Feed Na Concentration (M) ^(b)	5.00	5.00	5.27	6.02
Feed Na Concentration (M) ^(c)	5.00	5.00	4.84	4.86
Feed Cs Concentration (M) ^(d)	4.20E-05	5.30E-05	4.74E-05	5.40E-05
Feed Cs Concentration (μ Ci/mL) ^(e)	Trace	Trace	140	161
Feed Na/Cs Ratio (M/M) ^(f)	1.20E+05	9.43E+04	1.11E+05	1.11E+05
Feed Na/Cs Ratio (M/M) ^(g)	1.20E+05	9.43E+04	1.02E+05	9.00E+04
Feed Flowrate (CV/hr)	5.04	5.63	6.10	6.30
2 M NaOH Wash Volume (CV)	6.4	2.7	3.3	3.3
2 M NaOH Wash Flowrate (CV/hr)	5.12	5.47	6.70	6.60
Water Wash Volume (CV)	4.8	2.9	3.2	3.3
Water Wash Flowrate (CV/hr)	6.37	5.86	6.31	6.60
0.5 M HNO ₃ Elution Volume (CV)	7.1	7.4	7.4	7.8
0.5 M HNO ₃ Elution Flowrate (CV/hr)	1.01	1.05	1.05	1.31
3.0 M HNO ₃ Strip Volume (CV)	NA	2.0	3.0	NA
3.0 M HNO ₃ Strip Flowrate (CV/hr)	NA	0.94	5.9	NA
Final Water Wash Volume (CV)	NA	NA	2.8	2.9
Final Water Wash Flowrate (CV/hr)	NA	NA	4.25	5.8
2 M NaOH Regen Volume (CV)	NA	NA	2.8	3.2
2 M NaOH Regen Flowrate (CV/hr)	NA	NA	2.28	6.4
Experimental Temperature (°C)	25°	25°	22°	22°

(a) Not applicable.

(b) Sodium analysis on the actual wastes completed at the WHC 222-S analytical laboratory.

(c) Sodium analysis on the actual wastes completed at the PNL 325 Building analytical laboratory.

(d) Cesium analysis on the actual wastes completed at the PNL 325 Building analytical laboratory.

(e) Cesium analysis on the actual wastes completed at the WHC 222-S analytical laboratory.

(f) Sodium analysis on the actual wastes completed at the WHC 222-S analytical laboratory.

(g) Sodium analysis on the actual wastes completed at the PNL 325 Building analytical laboratory.

2.1 Batch Distribution

Although batch distribution coefficients (K_d s) were not determined for the experiments described in this report, the information in this section is useful for understanding the relationship of the current column loading experiment to previous batch K_d results. Carefully analyzing these data provides an estimate of the column size.

The batch distribution coefficient ($K_d = [Cs]_{\text{solid}} \div [Cs]_{\text{liquid}}$) is an equilibrium measure of the overall ability of the solid phase ion exchange material to remove an ion from solution under the particular experimental conditions which exist during the contact. In most batch K_d tests, a known quantity of ion exchange material is placed in contact with a known volume of solution containing the particular ions of interest (in this case, cesium). The material is allowed to contact the solution long enough to reach equilibrium at a constant temperature, after which the solid ion exchange material and liquid supernate are separated and analyzed. The equation for determining the K_d can be simplified by determining the concentration of the analyte before and after contact and calculating the quantity of analyte on the ion exchanger by difference (Equation 2.1).

$$K_d = \frac{(C_i - C_f)}{C_f} * \frac{V}{M * F} \quad (2.1)$$

where C_i is the initial concentration of the ion of interest in the feed solution prior to contact, C_f is the concentration after contact, V is the solution volume, M is the "as received" exchanger mass, and F is the mass of dry ion exchanger divided by the mass of wet ion exchanger (F -factor). K_d (mL/g) represents the theoretical volume of solution at equilibrium that can be processed per mass of exchanger under equilibrium conditions. λ , the theoretical number of bed volumes of solution at the final equilibrium concentration that can be processed per volume of exchanger, is obtained by multiplying K_d by the exchanger bed density, ρ_b (g/mL) as shown in Equation 2.2. λ is useful for estimating the 0.5 C/C_0 point in column ion exchange loading experiments. The 0.5 C/C_0 point is the number of column volumes which have passed through the column when the target species concentration exiting the column reaches one half the column inlet feed concentration.

$$\lambda = K_d * \rho_b \quad (2.2)$$

2.2 Simulant Feed Preparation

The feed solutions used for the cesium removal experiments were prepared using actual waste from tanks 101-SY and 103-SY. Although technically classified as CC waste, the actual waste supernates contain little total organic carbon (ca. 4 g C/L TOC), soluble ^{90}Sr , or total alpha (AT). In this respect the solutions are much different than the CC waste in tanks AN-102 or AN-107, which contain much higher levels of TOC, soluble ^{90}Sr , and AT. The simulant solutions (Table 2.2) were designed to mimic the cesium ion exchange properties of actual 101-SY and 103-SY tank waste.

Table 2.2. Comparison of Simulant Compositions for Tanks 101-SY and 103-SY (designated Sim101SY-Cs5 and Sim103SY-Cs5)

Species	Simulant Species Molarity (M)	
	101-SY	103-SY
Na	5.00E+00	5.00E+00
K	3.38E-02	2.84E-02
Rb	4.20E-06	5.30E-06
Cs	4.20E-05	5.30E-05
Ca	4.20E-03	3.16E-03
Sr	2.86E-07	9.10E-07
Al	4.15E-01	4.74E-01
Ni	2.49E-04	3.95E-04
Fe	1.96E-04	2.48E-04
Mo	4.20E-04	3.95E-04
Zn	5.00E-04	6.32E-04
CO ₃	3.75E-02	2.08E-01
F	9.18E-02	8.53E-02
NO ₂	1.09E+00	6.76E-01
NO ₃	1.29E+00	1.54E+00
OH (added)	3.78E+00	3.68E+00
OH (Free)	2.11E+00	1.77E+00
Theoretical pH	1.47E+01	1.46E+01
SO ₄	4.75E-03	3.60E-02
PO ₄	2.04E-02	9.47E-03
TOC (g/L)	3.42E+00	6.34E+00
Na/Cs Ratio:	1.20E+05	9.43E+04
K/Cs Ratio:	8.05E+02	5.36E+02
Na/Sr Ratio:	1.76E+07	5.49E+06
K/Sr Ratio:	1.18E+05	3.12E+04

The compositions were formulated using the best available analytical characterization data (Van Vleet 1993), organic complexant composition (Lokken et al. 1991), and previous simulant data (Bryan and Pederson 1994). No attempt was made to accurately mimic other properties of the actual tank waste (e.g., viscosity, solids, gas generation, ^{90}Sr , ^{99}Tc , etc). Use of this simulant for other than the intended purpose is not recommended.

The actual composite samples from the whole tank (estimated to be 14.1 M Na and 11.5 M Na for 101-SY and 103-SY, respectively) are saturated with respect to several components and contain significant quantities of undissolved solids (NaNO_3 , Na_2CO_3 , etc.) that can be brought into solution upon dilution of the waste. Ion exchange processing of the undiluted solutions at or near saturation is not advised since a precipitate may form during operation and effectively plug the column. However, dilution of tanks 101-SY and 103-SY must be completed with sodium hydroxide to prevent the precipitation of aluminum as $\text{Al}(\text{OH})_{3(s)}$ (Barney 1976). This is the recommended concentration for passive mitigation of waste in 101-SY and would also satisfy the cross site transfer criteria (Stewart et al. 1994). Based on the historical tank characterization data (Van Vleet 1993), Table 2.3 displays an estimate of the process volumes and anticipated concentration for selected species during a theoretical dilution of the actual wastes to 5 M Na total with 2 M NaOH.

Table 2.3. Estimated Dilution Parameters to Prepare Simulants Based on Historical Characterization Data for 101-SY and 103-SY Actual Waste Composites^(a)

Estimated Parameter	101-SY Tank Composite		103-SY Tank Composite	
	Undiluted	Diluted	Undiluted	Diluted
Volume (mL)	2.00E+02	8.00E+02	2.00E+02	6.33E+02
Na Concentration (<u>M</u>)	1.41E+01	5.00E+00	1.15E+01	5.00E+00
Cs Concentration ($\mu\text{Ci}/\text{mL}$)	6.00E+02	1.50E+02	4.00E+02	1.26E+02
Cs Concentration (<u>M</u>)	1.68E-04	4.20E-05	1.68E-04	5.30E-05
K Concentration (<u>M</u>)	1.35E-01	3.38E-02	9.00E-02	2.84E-02
Al Concentration (<u>M</u>)	1.66E+00	4.15E-01	1.50E+00	4.74E-01
OH Concentration (<u>M</u>)	2.30E+00	2.11E+00	1.27E+00	1.77E+00
Estimated Na/Cs	7.33E+04	1.20E+05	6.85E+04	9.43E+04

(a) The components listed in this table are useful for estimating the volume of simulated waste required to obtain the cesium loading breakthrough curve. Not every component of the actual waste is listed.

2.3 Exchanger Selection and Preparation

Duolite CS-100, a granular (20-50 mesh) phenol-formaldehyde condensate polymer resin, was chosen as the material for the column ion exchange removal of cesium from simulated and actual 101-SY and 103-SY tank waste. The material is considered to be the baseline material for alkaline-side cesium removal at Hanford (Eager et al. 1994)^(a). The resin was purchased from Rohm & Haas (Batch #6-8144, Lot#2-850001, November 25, 1991) and has been stored under an air atmosphere as it was received (H⁺-form) since that time. Approximately 4.35 g of "as received" material was accurately weighed into a beaker and contacted with 50 mL of 2 M NaOH for 30 minutes. This resin was slurry transferred into the ion exchange column in preparation for the loading and elution experiments. A fresh portion of resin was used for each column loading and elution test. The ratio of dried to "as received" resin (F-Factor) was determined by drying a separate 1-g portion of "as received" material for 24 hours at 105°C and measuring the total weight loss.

2.4 Estimation of Column Size

To achieve a meaningful and complete breakthrough curve during a typical ion exchange decontamination experiment, the volume and composition of available solution must be effectively matched to the quantity and type of sorbent material. Any number of factors may control this solid/liquid contact ratio, but during the current experiment, the volume of available waste (ca. 200 mL in-tank) was the limiting factor for column sizing considerations. In general, the material's total ion exchange capacity, selectivity, analyte loading (e.g., Cs loading is a function of the material's overall capacity and selectivity for Cs over other matrix ions like Na⁺, K⁺, etc.), and material density regulate the quantity of analyte ion that can be removed from a particular waste stream at a specific temperature. Each ion exchange material has a distinct column packing density, capacity, and selectivity as a function of solution temperature and composition. It is entirely possible for one exchanger to exhibit superior performance over another material under one set of circumstances and inferior performance under a second set.

The primary objective of this experiment was to obtain loading and elution breakthrough data for the ion exchange removal of cesium from actual Hanford tank waste and compare the results to those obtained for simulant solutions. The column bed volume (CV) required to fully load CS-100 was estimated from previous K_d data collected in simulated CC or NCAW waste matrices (Kurath et al. 1994) (see Section 2.1 for a discussion of K_d). At an equilibrium Na/Cs ratio near 1.00E+05 (e.g., that of the diluted 101-SY and 103-SY solutions used in this experiment), the lambda value was estimated to be 52 and 50 CV for CS-100, respectively. To exceed the 0.5 C/C₀ point and obtain a more complete cesium breakthrough curve, approximately 25% greater volume should be used (62.5 CV). From Table 2.3, estimated volumes of available actual waste feed were calculated to be approximately 800 and 630 mL for 101-SY and 103-SY wastes, respectively. Therefore, to achieve the intended

(a) Gallagher, S. A. 1986. *Report of Current NCAW Ion Exchange Laboratory Data*. Internal Letter #65453-86-088, Rockwell International, Richland, Washington.

breakthrough curve, the column volume was estimated to be 12.8 and 10.1 mL for each respective solution. Experimentally, the actual column size varied from 11.8 to 12.3 mL with an average of 12.1 mL (Table 2.1), and the actual waste volumes were 684 mL for tank 101-SY and 434 mL for tank 103-SY. The usable waste volume was smaller than expected because of solids, interstitial liquid in the solids, and sampling of the feed solution for analysis.

2.5 Column Loading and Elution

As was discussed in Section 2.1, batch K_d s are a rapid and cost-effective method of determining a material's ion exchange loading under certain equilibrium conditions. However, since the baseline cesium decontamination method will be a column ion exchange process, the behavior of the material in a flowing system must be assessed. In this case, column loading and elution provides information concerning the extent of analyte breakthrough as a function of flow rate, feed composition, temperature, column dimension, and ion exchange material.

A generalized discussion of the column loading and elution equipment and process flowsheet follows. Table 2.1 displays a detailed list of the exact experimental conditions. The ion exchange column system consisted of a single ion exchange column, pressure gauge, feed storage bottle, pump, and sample effluent collection bottles. The glass column (1.0-cm i.d.) had a maximum capacity of 24 mL of ion exchange material. The volume of the column could be adjusted between 2 and 22 mL (including head space for solution above the resin bed). Temperature was not controlled and all testing was completed at ambient (22°C to 25°C). The solution was processed by pumping down through the column from a large feed reservoir. The column effluent was sampled periodically (ca. every 30 minutes) through a valve located at the column exit. The volume of solution processed in CV was determined by dividing the effluent solution weight by the specific gravity of the feed solution.

For all tests a single column was partially filled with 2 M NaOH, and ~4.35 g of resin ("as received" CS-100 in the H⁺-form) was contacted with 50 mL of 2 M NaOH for 30 minutes and then slurried into the column. Approximately 3 CV of 2 M NaOH (36 mL) was passed up through the column bed at approximately 12 CV/hr. The flow was reversed to downflow and 3 CV of additional NaOH was passed through the resin while the pump was calibrated to 1.2 mL/min (6 CV/hr). The column was allowed to sit overnight before it was loaded with the simulated or actual waste solutions. The resin bed was approximately 12 mL (1.0 cm i.d. x 15.5 cm) at the end of the column preparation. The diluted feed solution (101-SY or 103-SY) was passed down through the column at 6 CV/hr. Column effluent composite samples were collected twice per hour (ca. 3 CV or 36 mL) throughout the experiment.

The loading phase was terminated after the feed had been exhausted. The column was then washed sequentially with 2 M NaOH (3 CV or 36 mL at 6 CV/hr or 1.2 mL/min) and water (3 CV at 6 CV/hr). Two composite samples for each wash were collected every 1.5 CV (18 mL). The cesium was eluted from the column with 0.5 M HNO₃ at 1 CV/hr (12 mL/hr). Composite samples were collected every 30 minutes (0.5 CV or 6 mL). Following the elution step, for the actual 101-SY and simulated 103-SY tests only, the column was stripped with 3 M HNO₃ to remove any additional

components remaining on the CS-100. The material was prepared for disposal by washing with water and 2 M NaOH as above. In the 101-SY and 103-SY actual waste tests, these steps were denoted by "clean" and "final" for the water and 2 M NaOH caustic washes, respectively. A fresh, unused portion of CS-100 ion exchange material was used for each waste type.

3.0 Results and Discussion

The ion exchange removal of cesium from simulated and actual alkaline Hanford tank waste (tanks 241-SY-101 and 241-SY-103) has been demonstrated using ~12-mL columns of CS-100. Previous researchers have reported the utility of using various ion exchange materials to remove cesium from simulated tank waste (Brown et al. 1995; Kurath et al. 1994). The results described in this report provide vital information concerning the ability to mimic cesium ion exchange properties of actual Hanford tank waste with simulated solutions.

3.1 Actual Waste Feed Preparation

Composite samples from tanks 101-SY and 103-SY were used for these tests. Each composite sample contained representative amounts of each core segment or layer, so that the composition of the composite sample represented a fully blended tank waste. For tank 101-SY, the sample was taken from an archived jar labeled "101-SY Tank Comp 93D," which had been stored in the 1E-1 hotcell at the 222-S laboratory since it was prepared in January, 1993. It contained waste from both the Window C (May, 1991) and Window E (December, 1991) core samples. The 103-SY whole-tank composite sample was provided by Andrew Rice (WHC) from the core sample taken from that tank earlier this year.

The composite samples were diluted with 2 M NaOH to achieve an estimated 5 M sodium concentration. Table 3.1 shows the dilution data. Each dilution was made by adding 2 M NaOH to the

Table 3.1. Dilution Data for Tank Composite Samples

Dilution Parameter	Tank 101-SY	Tank 103-SY
Total undiluted sample weight (g)	320.1	306.8
Estimated density, based on core data (g/mL)	1.6	1.5
Total undiluted sample volume (mL)	200.1	204.5
Weight of 2 <u>M</u> NaOH added (g)	648.2	432.8
Density of 2 <u>M</u> NaOH (CRC Handbook value)	1.082	1.082
Calculated volume of 2 <u>M</u> NaOH added (mL)	599.1	400.0
Total slurry volume if additive (mL)	799.1	604.5
Total slurry volume measured (mL)	789.0	588.0
Total slurry weight (g)	968.3	739.6
Density of dilute slurry (g/mL)	1.227	1.258
Total supernatant liquid volume (mL)	758.0	566.0
Total centrifuged solids volume (mL)	31.0	22.0
% centrifuged solids (v/v%)	3.92	3.74
Supernatant liquid density (g/mL)	1.213	1.242

undiluted sample in a 1-L Erlenmeyer flask and stirring at ambient temperature (approximately 22°C) for 24 hours before transferring the slurry into a series of 50-mL centrifuge cones. The "total slurry volume measured" entry in Table 3.1 is the sum of the volumes in each of the centrifuge cones. Each cone was centrifuged for approximately one hour. The centrifuged solids accounted for approximately 4% (v/v) or 5% (w/w) of the diluted waste. The supernatant solutions were decanted into a 1-L plastic bottle labeled "Diluted 101-SY [or 103-SY] Feed for IX Test." Duplicate samples were taken from each feed bottle for analysis.

Analytical results obtained by the WHC 222-S laboratory for the feed solution samples are displayed in Table 3.2. These results show that the two tanks are very similar in composition. The

Table 3.2. WHC Analytical Results for Tank 101-SY and 103-SY Actual Waste Samples

Analyte	Units	Tank 101-SY		Tank 103-SY	
		Sample	Duplicate	Sample	Duplicate
Al	<u>M</u>	5.71E-01	5.26E-01	6.56E-01	5.89E-01
B	<u>M</u>	2.96E-03	2.68E-03	3.89E-03	3.42E-03
Ca	<u>M</u>	1.52E-03	1.32E-03	nd ^(a)	nd
Cr	<u>M</u>	9.62E-04	9.42E-04	8.85E-04	7.69E-04
Cu	<u>M</u>	8.20E-05	6.30E-05	nd	nd
K	<u>M</u>	3.68E-02	3.43E-02	4.25E-02	3.96E-02
Mo	<u>M</u>	4.69E-04	4.27E-04	6.05E-04	5.21E-04
Na	<u>M</u>	5.18E+00	5.35E+00	6.26E+00	5.79E+00
Ni	<u>M</u>	1.12E-03	1.06E-03	3.58E-04	2.90E-04
P	<u>M</u>	3.87E-02	3.68E-02	5.23E-02	4.62E-02
S	<u>M</u>	2.30E-02	2.16E-02	3.08E-02	2.09E-02
Si	<u>M</u>	4.73E-03	4.63E-03	3.13E-03	2.63E-03
% H ₂ O	wt %	7.36E+01	7.37E+01	7.25E+01	7.26E+01
Cl	<u>M</u>	9.90E-02	9.87E-02	1.17E-01	1.16E-01
NO ₂ ⁻	<u>M</u>	9.45E-01	9.30E-01	1.06E+00	1.07E+00
NO ₃ ⁻	<u>M</u>	9.26E-01	8.74E-01	1.13E+00	1.13E+00
PO ₄ ³⁻	<u>M</u>	2.45E-02	3.17E-02	3.58E-02	3.51E-02
SO ₄ ²⁻	<u>M</u>	1.82E-02	1.73E-02	2.29E-02	2.22E-02
Oxalate	<u>M</u>	6.14E-03	7.23E-03	<1.25E-02	<1.25E-02
Na/K	<u>M/M</u>	1.41E+02	1.56E+02	1.45E+02	1.46E+02
TOC	g/L	4.27E+00	4.27E+00	3.05E+00	2.62E+00
TIC	g/L	2.69E+00	2.83E+00	2.99E+00	2.86E+00
¹³⁷ Cs	μCi/mL	1.38E+02	1.42E+02	1.61E+02	1.61E+02
AT	μCi/mL	< 1.00E-03	< 5.00E-04	9.10E-04	7.60E-04
⁹⁰ Sr	μCi/mL	1.82E+00	1.84E+00	8.20E-01	8.40E-01
⁹⁹ Tc	μCi/mL	2.71E-02	3.40E-02	7.20E-02	7.90E-02
²⁴¹ Am	μCi/mL	< 2.90E-04	< 2.70E-04	6.30E-04	6.00E-04
^{239/240} Pu	μCi/mL	4.10E-05	3.60E-05	9.70E-05	< 3.90E-05

(a) Not detected.

103-SY feed solution, having been diluted less than the 101-SY sample, is slightly more concentrated in most of the major components. Notable exceptions include TOC and ^{90}Sr , which have substantially higher concentrations in the 101-SY feed solution. The dilutions were calculated to produce 5 M sodium, based on the historical analytical data available for the core samples (Van Vleet 1993). From the resulting concentrations in the feed solutions, it would appear that the amount of sodium in the undiluted 103-SY composite sample had been underestimated. Table 3.3 displays the analytical results obtained by PNL's analytical laboratory in the 325 Building for the feed samples. In addition to metals analysis by inductively coupled plasma atomic emission (ICP-AES), cesium and rubidium were determined by graphite furnace atomic absorbance spectroscopy (GFAAS), and Cs, Rb, and Sr isotopic mass ratios were determined by thermal ionization mass spectrometry (TIMS). In general, the results are similar to those obtained by the 222-S laboratory, with the exception of those for sodium concentration. The PNL data suggest that the diluted feeds were 4.84 M and 4.86 M for the 101-SY and 103-SY solutions, respectively.

3.2 Cesium Loading of CS-100

Figure 3.1 displays the results of the column ion exchange removal of cesium from simulated and actual Hanford tank waste. The Cs loading breakthrough data are plotted in log-probability format where the cesium concentration (C) is normalized to the initial feed concentration (C_0). The Appendix provides a summary of the Cs loading data (Tables A.2 through A.5). When plotted using this format, the standard sigmoidally shaped loading curve is represented by a straight line (Buckingham 1967). Normalization of the cesium data provides the reader an easy way to estimate the cesium concentration and decontamination factor (DF). From Figure 3.1, approximately 7 CV of 101-SY and 5 CV of 103-SY solution can be processed before the Cs DF exceeds 1000. Zero volume is defined as the point at which the feed solution initially reaches the CS-100 resin at the top or head of the column, and therefore these values include one CV void volume. The initial cesium feed concentration of the diluted 101-SY tank waste was 140 $\mu\text{Ci/mL}$ and is estimated to be equivalent to approximately 5.03E-05 M total cesium (the ratio of ^{137}Cs to total cesium was determined by TIMS to be 23.01%). Cesium was also determined to be 4.74E-05 M by GFAAS, in agreement with GEA and TIMS results. The corresponding values for the 103-SY waste were 161- $\mu\text{Ci/mL}$ ^{137}Cs , 5.92E-05 M total Cs (22.82% ^{137}Cs by TIMS), and 5.40E-05 M total Cs (GFAAS). For comparison, the simulants were prepared with 4.20E-05 and 5.30E-05 M Cs for 101-SY and 103-SY, respectively.

The most important aspect of these data is the remarkable similarity of the actual and simulated waste loading curves. The simulants were prepared using historical radionuclide and nonradionuclide data (Van Vleet 1993) and assuming 30% of the total cesium was ^{137}Cs . Based on the analysis completed for total sodium and cesium (Table 3.3) and the cesium loading breakthrough data (see Figure 3.1), it appears that the simulant composition accurately modeled the Cs ion exchange behavior of the actual waste. The difference between the 101-SY and 103-SY loading curves is related to various parameters as described in Section 2.2, including temperature, flow rate, Na, Cs, and K concentration. Since 103-SY contains more cesium than 101-SY, the breakthrough curve is shifted towards less volume.

Table 3.3. PNL Analytical Results for Tank 101-SY and 103-SY Actual Waste Samples

Analyte	Units	Tank 101-SY		Tank 103-SY	
		Sample	Duplicate	Sample	Duplicate
Al	<u>M</u>	4.61E-01	4.63E-01	4.50E-01	na ^(a)
B	<u>M</u>	8.87E-03	7.66E-03	8.58E-03	na
Ca	<u>M</u>	1.25E-03	1.31E-03	8.11E-04	na
Cd	<u>M</u>	5.78E-06	5.69E-06	3.56E-06	na
Co	<u>M</u>	1.04E-05	1.07E-05	7.64E-06	na
Cr	<u>M</u>	8.78E-04	9.49E-04	6.97E-04	na
Cs	<u>M</u>	4.66E-05	4.82E-05	5.40E-05	na
Cu	<u>M</u>	6.67E-05	6.35E-05	2.98E-05	na
Fe	<u>M</u>	2.65E-04	4.94E-04	1.40E-04	na
K	<u>M</u>	2.99E-02	3.01E-02	3.11E-02	na
Mn	<u>M</u>	3.82E-06	4.19E-06	4.00E-06	na
Mo	<u>M</u>	3.72E-04	3.75E-04	4.09E-04	na
Na	<u>M</u>	4.75E+00	4.92E+00	4.86E+00	na
Ni	<u>M</u>	9.92E-04	1.00E-03	2.57E-04	na
P	<u>M</u>	3.30E-02	3.31E-02	3.64E-02	na
Pb	<u>M</u>	1.33E-05	1.34E-05	9.94E-06	na
Rb	<u>M</u>	3.28E-05	3.28E-05	3.51E-05	na
Sb	<u>M</u>	4.63E-05	4.70E-05	4.54E-05	na
Si	<u>M</u>	1.05E-02	9.74E-03	7.52E-03	na
Sr	<u>M</u>	2.02E-05	2.08E-06	nd ^(b)	na
Tl	<u>M</u>	1.07E-04	1.06E-04	1.04E-04	na
W	<u>M</u>	2.66E-04	2.69E-04	3.22E-04	na
Zn	<u>M</u>	8.38E-05	8.40E-05	1.24E-05	na
Zr	<u>M</u>	7.34E-06	7.24E-06	7.13E-06	na
Na/Cs	<u>M/M</u>	1.02E+05	1.02E+05	9.00E+04	na
Na/K	<u>M/M</u>	1.59E+02	1.63E+02	1.56E+02	na
¹³⁷ Cs	%	2.35E+01	2.26E+01	2.28E+01	na
	Isotopic				
⁸⁷ Rb	%	6.50E+01	2.90E+01 ^(c)	6.81E+01	na
	Isotopic				
⁹⁰ Sr	%	1.50E+00	2.96E+00	3.03E+00	na
	Isotopic				

(a) Duplicate analyses not completed.

(b) Not detected.

(c) The determined Rb isotopic ratios are close to those expected for natural Rb, suggesting contamination of the analytical sample.

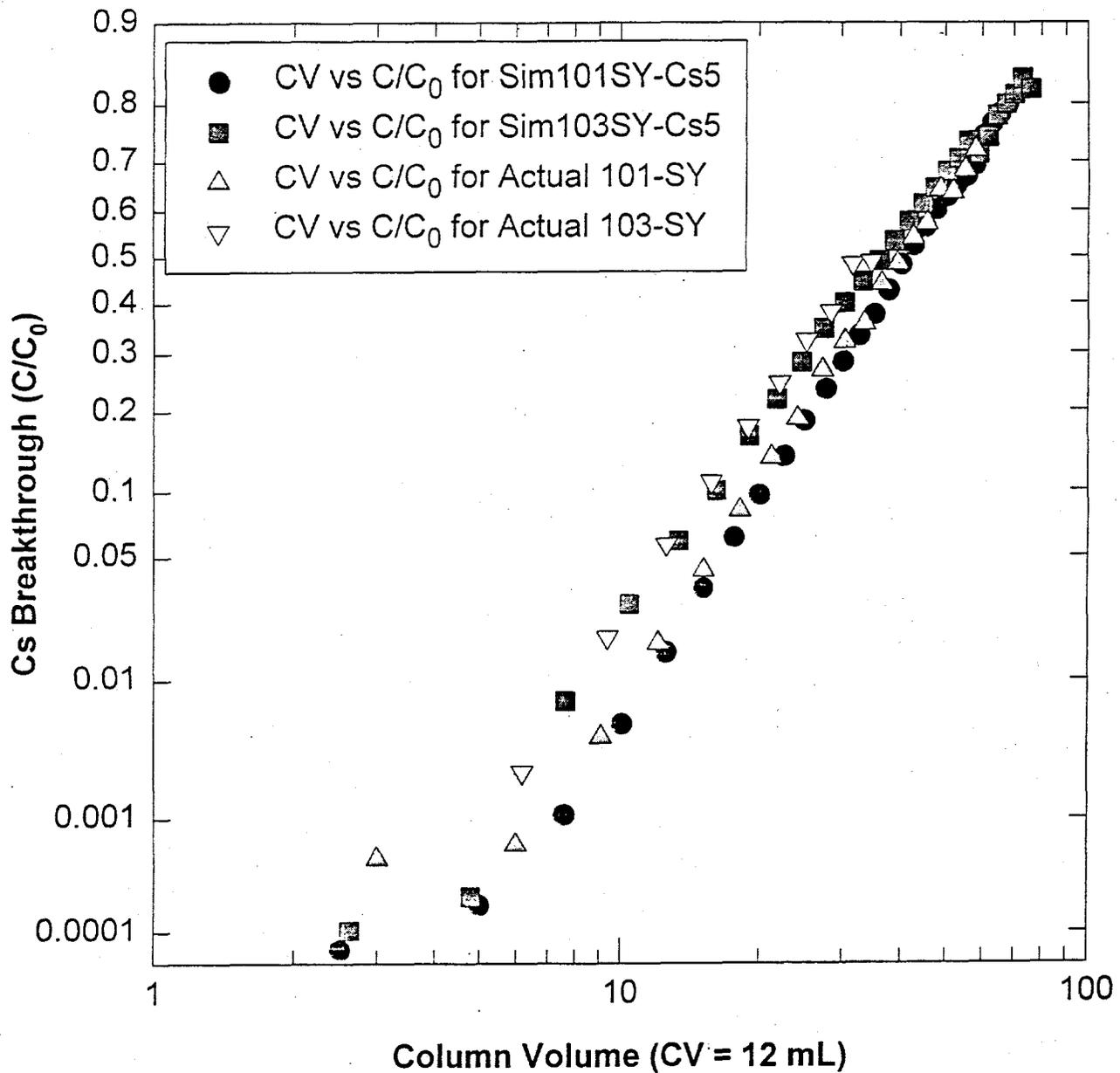


Figure 3.1. Cesium Breakthrough Curves for CS-100 Ion Exchange Column Loading of Simulated and Actual Waste Supernate from Tanks 101-SY and 103-SY Loaded at 6 CV/hr (CV = 12 mL) and Ambient Temperature

As was discussed in Section 2.4, previous CS-100 batch K_d data obtained in the NCAW simulant at various Na/Cs ratios were used to estimate the column volume size required to achieve 0.5 C/C_0 breakthrough with the volume of actual waste available. The estimated volume to reach 0.5 C/C_0 was approximately 52 and 50 for simulated NCAW solutions with Na/Cs ratios of 1.20E+05 and 9.43E+04, respectively (Kurath et al. 1994). However, in the current experiment with simulated and

actual 101-SY and 103-SY waste samples (see Figure 3.1), approximately 41.4 and 36.8 CV were achieved, respectively. The difference between the two curves is likely related to differences in the total Na and Cs concentrations (or Na/Cs ratio) of the wastes with respect to the simulants, as described in Section 2.2. The actual waste solutions may not have been diluted to exactly 5.0 M Na as estimated. The WHC analytical results indicate an average sodium concentration of 5.25 M and 6.05 M for the actual 101-SY and 103-SY waste, respectively. However, PNL analytical results suggest lower values (4.84 M and 4.86 M). Previous researchers have shown that the cesium loading for CS-100 and many other ion exchange materials decreases with increasing sodium concentration (Kurath et al. 1994). Differences between the chemical composition of the NCAW and 101-SY/103-SY simulants may reduce the cesium loading with a concurrent increase in the loading of other constituents. In addition, degradation of the CS-100 resin during storage, or reduced loading because of poor ion exchange kinetics or channeling during the present 12-mL column experiments, might also account for these results. However, a more probable explanation could be variations between the two tests in the bed density.

The cesium loading of the CS-100 resin was determined by integrating the area under the breakthrough curve and accounting for the column inlet and outlet cesium concentrations and the resin mass. The calculation yields a value of 8.26E-03 and 9.97E-03 mmol Cs per gram (H⁺-form dry weight basis) of CS-100 for the 101-SY and 103-SY simulant solutions, respectively. For the 101-SY and 103-SY actual waste experiments, the values are 8.43E-03 and 7.23E-03 mmol Cs per gram, respectively. These values are quite low when compared to previously reported values (Brown et al. 1995) and compared to the total capacity of CS-100. These lower values are to be expected since the NCAW simulant used in the previous work contains approximately ten times the cesium of the 101-SY and 103-SY solutions. The reason for these poor loading results is thus related to interfering ions (mostly Na) in the waste matrix. Based on this data, greater than 99% of the total capacity is occupied by chemical species (e.g., sodium) other than cesium.

3.3 Non-Cesium Loading of CS-100

In addition to the analysis for cesium loading on CS-100 during the ion exchange column experiment, several other radionuclide and nonradionuclide species were analyzed for possible breakthrough behavior. These species include AT, TOC, ⁹⁰Sr, ⁹⁹Tc, and various elements analyzed by ICP-AES. Table A.1 (see Appendix) displays a complete list of these elements. The listed detection limits and linear range are instrumental parameters and do not reflect dilution during sample preparation. Species not listed in the analytical report but specified in Table A.1 were below the sample detection limit. To reduce analytical costs, not every analytical method was completed for each sample. In general, none of the species exhibited a loading breakthrough curve similar to cesium (i.e., initially undetectable followed by a sigmoidal concentration profile which eventually approached the feed concentration). In contrast, all of the analytical data suggest immediate breakthrough of these components. Tables A.10 through A.20 in the Appendix display a complete listing of the data. Total alpha was below the detection limit in virtually every sample. TOC, ⁹⁰Sr, and ⁹⁹Tc remained fairly constant during the load cycle, with concentrations essentially at or near the feed concentration (e.g., 4.27 g/L, 1.83 μCi/mL, and 30.6 nCi/mL, respectively).

The ICP-AES analytical results for the first loading sample are all lower than the feed concentration by approximately a factor of two. This is consistent with the experimental procedure. The first sample contains one column volume (void volume) of 2 M NaOH left over from the column preparation step. Since the total volume passed during the first sample was about 3 CV, the dilution factor should approach three. Beginning with the second loading sample, the concentrations essentially reach the feed concentration. Notable exceptions for the 101-SY experiment include Al (13,800 vs. 14,800 $\mu\text{g/mL}$ feed), Ni (52 vs. 64 $\mu\text{g/mL}$), Ca (49 vs. 57 $\mu\text{g/mL}$), and K (1280 vs. 1390 $\mu\text{g/mL}$). Similar results are observed for the 103-SY test as well. At this time, it is not known if these differences are significant since there is wide variability in the analytical data. A better indication of what may have loaded can be obtained by analyzing the species eluted from the resin since the material will act as a concentrating agent prior to analytical determination (Section 3.5).

During the caustic wash (3 CV of 2 M NaOH) and water wash (3 CV) phases after loading and prior to elution of the resin, the concentrations of all species decreased significantly as the interstitial solution was rinsed from the column. For the 101-SY experiments, TOC, ^{90}Sr , and ^{99}Tc decreased to approximately 0.04 g/L, 655 nCi/mL, and 0.4 nCi/mL, respectively. The corresponding values for the 103-SY test were <0.06 g/L, 1.0 nCi/mL, and 2.3 nCi/mL, respectively.

3.4 Cesium Elution of CS-100

Figure 3.2 displays the Cs elution results for the previously loaded CS-100 columns. The data are normalized to the initial cesium concentration of the respective feeds (actual or simulated 101-SY and 103-SY). As was the case for the loading breakthrough curves, data plotted in this way are useful for determining DF and analyte concentration over the course of the experiment. In all cases, the cesium concentration initially starts at a value of approximately 0.03 C/C_0 or $1.5\text{E-}06$ M Cs. This value will depend upon the volume of caustic and water washes used after the loading cycle. The Cs concentration begins to increase as the 0.5 M HNO_3 reacts with the residual NaOH remaining in the column after the water-washing phase. As the pH drops, the distribution coefficient drops and the cesium is eluted. The peak concentration reaches approximately 60 to 70 C/C_0 , or $3.0\text{E-}03$ M Cs at approximately 2.5 to 3.0 CV. Beyond this point, the Cs concentration in the eluant decreases exponentially as the Cs on the column is exhausted to approximately $1.0\text{E-}03$ C/C_0 , or $5.0\text{E-}08$ M Cs with an additional 3 CV of acid. The data indicate that the cesium on the CS-100 resin can be easily removed with ~ 4 CV of 0.5 M HNO_3 .

The elution results are similar to results obtained previously (Kurath et al. 1994) and display a standard elution curve shape. However, there is a difference between the actual and simulant elution data which was not noted in the loading data. The simulant data for the 101-SY and 103-SY elution are nearly identical and exhibit a peak at 2.5 CV of acid. The data generated from elution of the actual 101-SY and 103-SY waste are also nearly identical but have a peak at approximately 3.1 CV. The wastes have different levels of cesium (and Na/Cs ratios), and yet demonstrate identical elution when normalized. It does not seem to matter how much cesium is present. However, there is a difference between the simulant and actual waste elutions. Evidently something which was not properly simulated is interfering with elution. For the actual waste experiments, approximately 0.6 CV of additional acid

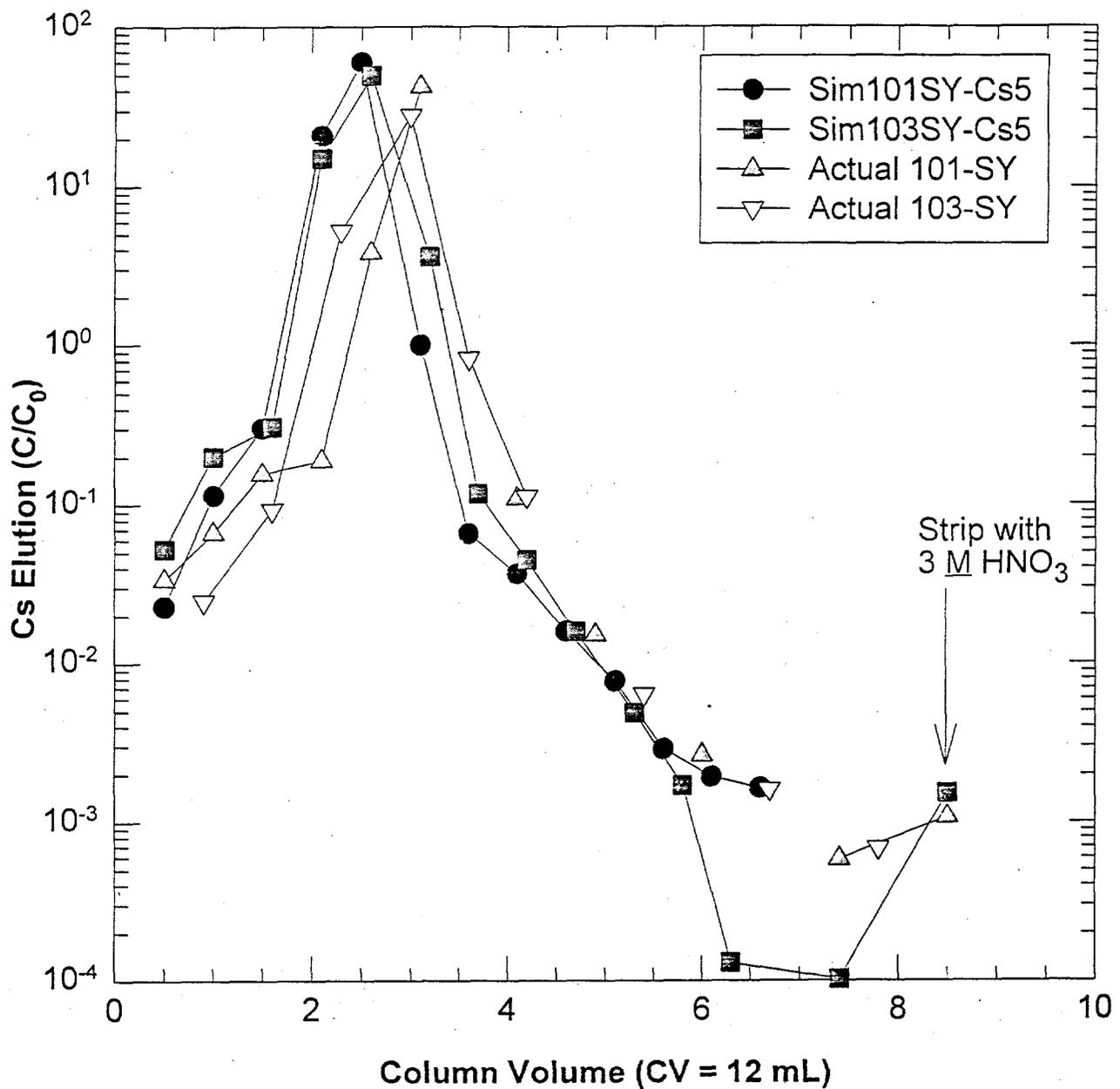


Figure 3.2. Cesium Breakthrough Curves for CS-100 Ion Exchange Column Elution of Simulated and Actual Waste Supernate from Tanks 101-SY and 103-SY Eluted with 0.5M HNO₃ at 1 CV/hr (CV = 12 mL) and Ambient Temperature

(3.6 mmol H⁺) is required before the elution of cesium can proceed. The observed difference cannot be due to a variation in the caustic or water wash volumes since less volume was used in the 103-SY simulant test (Table 2.1). This interference can be estimated to correspond to 1.35 mmol/g resin,

which appears to be a significant fraction of the resin capacity. No metal component (e.g., Ni, Cr, Sr, etc.) of the actual waste that can account for this interference has been identified (Section 3.5). It is interesting to note that two different wastes, each with differing components and volumes loaded on the resin, each shift the elution the same extent. The reason for the observed shift is not known, but could be related to variation in experimental temperature, acid eluant composition, or resin loading (or retention) of certain species present only in the actual waste that required additional acid to be eluted and/or acidified. For example, salts of various carboxylic acids (e.g., oxalate) may account for the observed differences.

3.5 Non-Cesium Elution of CS-100

In general, the elution of any ion exchange resin provides a unique opportunity for analysis of trace components because of the relative preconcentration achieved for species which can be easily removed. However, one must be careful since other species may remain fixed to the resin and interfere with loading during subsequent cycles. In addition to cesium, the removal of various radionuclides and nonradionuclides by CS-100 from actual waste solution is of interest to scientists and engineers investigating various pretreatment options. Selected elution samples were analyzed for the same species described in Section 3.3 to assess the extent to which noncesium species interact with the CS-100 ion exchange resin. Tables A.11 through A.20 in the Appendix provide a complete listing of the data.

In addition to ^{137}Cs , elution data were also collected for TOC, AT, ^{99}Tc , ^{90}Sr , and various metals analyzed by ICP-AES. Unfortunately, the removal of alpha-emitting radionuclides could not be properly investigated since nearly all of the samples for 101-SY and most of the samples for 103-SY (feed, load, wash, and elute) contained less-than-detectable quantities of AT (variable from <0.5 nCi/mL to <5.7 nCi/mL). Technetium-99 did not load or elute from the column to any significant extent as would be expected for an anionic species. The TOC elution results suggest that some loading does occur. For the 101-SY actual waste test, TOC increases from 0.046- to 0.104-g C/L at the peak before decreasing to 0.091. For 103-SY, the increase is from <0.06 - to 1.05-g C/L before decreasing to 0.06. It is difficult to determine if the trends are significant since only minimal samples were analyzed for the two actual waste experiments. The source of TOC could be from loading and elution of carbon-containing species in the waste or degradation of the organic ion exchange resin during processing.

Figures 3.3 and 3.4 display the analytical results for several species as a function of 0.5 M HNO_3 volume for the elution of CS-100 columns that were previously loaded with actual 101-SY and 103-SY supernate, respectively. Tables A.11 through A.20 in the Appendix show the data, which are normalized to each species' respective feed concentration, as described previously. It appears that several species are eluted from the resin and therefore must have been loaded, but that only nickel is actually concentrated (peak elution approximately 10 times the original feed concentration). The data are fairly consistent and exhibit very similar elution profiles for each waste (actual 101-SY and 103-SY). The elution peaks also coincide with the cesium elution profile (Figure 3.2).

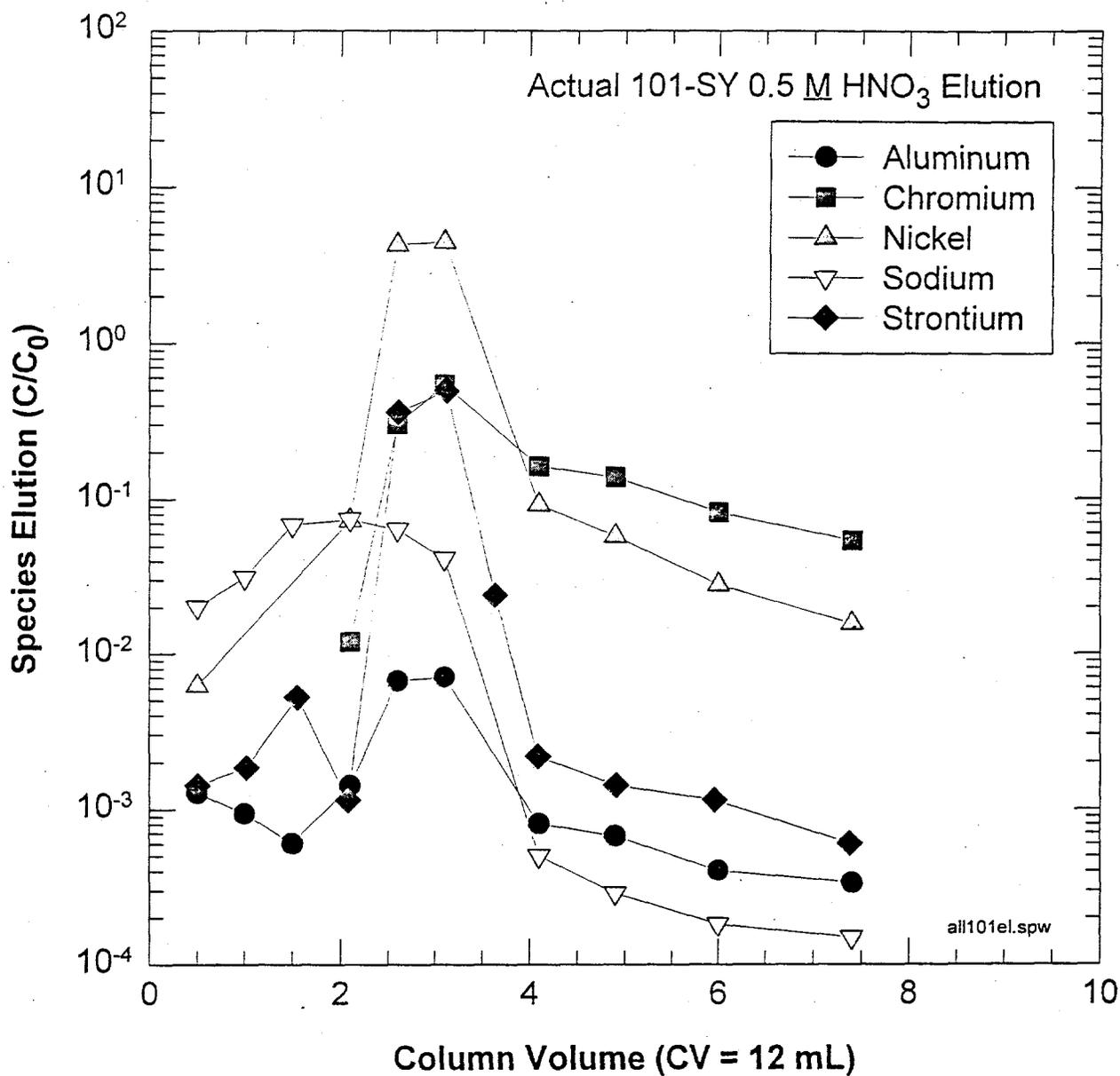


Figure 3.3. Al, Cr, Na, Ni, and Sr Breakthrough Curves for CS-100 Ion Exchange Column Elution of Actual Waste Supernate from Tank 101-SY Eluted with 0.5 M HNO₃ at 1 CV/hr (CV = 12 mL) and Ambient Temperature (22°C)

The data suggest that several elements (Al, B, Ca, Cr, Cu, K, Na, Ni) can be eluted from the resin as determined by ICP-AES. It appears that nickel has the greatest effect. For example, in the 101-SY column elution samples, Ni started at 0.4, peaked at 285, and decreased again to 0.6 µg/mL. Chromium increased from below the detection limit to 27 µg/mL before decreasing to 2.7. Similar results

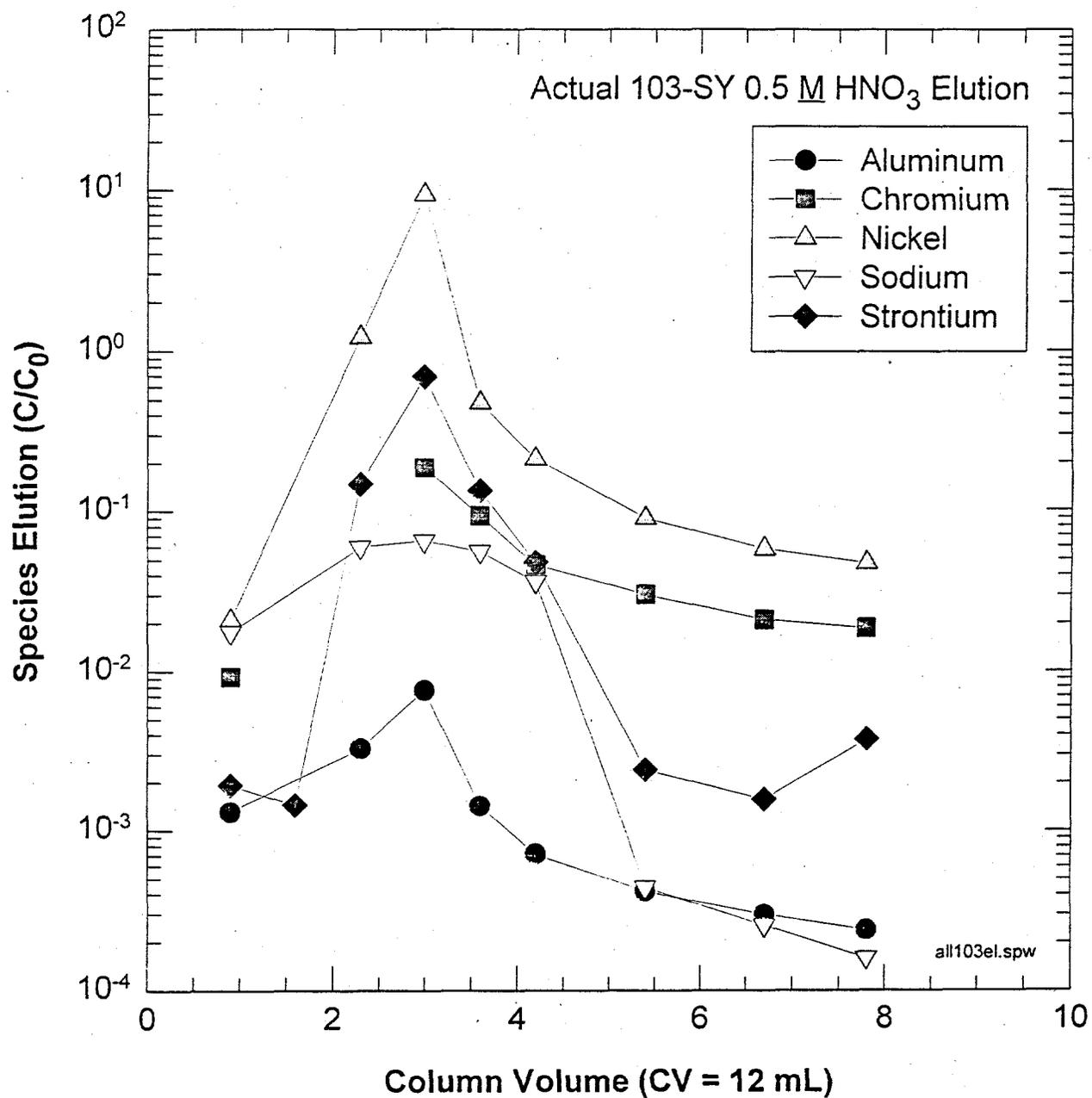


Figure 3.4. Al, Cr, Na, Ni, and Sr Breakthrough Curves for CS-100 Ion Exchange Column Elution of Actual Waste Supernate from Tank 103-SY Eluted with 0.5 M HNO₃ at 1 CV/hr (CV = 12 mL) and Ambient Temperature (22°C)

were observed for aluminum, potassium, and sodium and are to be expected because of the high concentration of these species in the waste feed. Even though the peak Na concentration is only 0.05 C/C₀ (9000 μg/mL), this translates into 4.5E-03 mol Na eluted, which is equivalent to approximately 4 mmol/g.

The quantity of each species eluted from the resin can be calculated (Table 3.4) by integrating the area under each curve in Figures 3.3 and 3.4. The calculations were completed for the 101-SY and 103-SY actual waste tests only. The results for strontium were calculated from radioanalytical data and assuming that 3% of the total strontium was ⁹⁰Sr (as determined by TIMS). The results for cesium were calculated using the Cs GFAAS data from Table 3.3. The data corroborate the theory that a large fraction of the resin capacity is used by sodium. It is interesting to note that the resin appears to have a large affinity for nickel and chromium. The initial concentration of these species is 10 to 20 times the Cs level.

Table 3.4. Selected Species Eluted During 101-SY and 103-SY Actual Waste Tests

Species	Cumulative Elution (meq)		Cumulative Elution (%) ^(a)	
	101-SY	103-SY	101-SY	103-SY
Cs	2.23E-02	1.54E-02	0.21%	0.11%
Sr	2.80E-06	1.83E-06	0.00%	0.00%
Ni	7.46E-02	3.01E-02	0.70%	0.22%
Al	8.46E-02	9.05E-02	0.80%	0.65%
Ca	3.31E-03	0.00E+00	0.03%	0.00%
Cr	1.11E-02	1.29E-02	0.10%	0.09%
K	6.20E-03	7.69E-03	0.06%	0.06%
Na	1.04E+01	1.38E+01	98.10%	98.87%
Total ^(b)	1.06E+01	1.39E+01	100.00%	100.00%
Total ^(c)	3.95E+00	5.18E+00		

(a) The value listed reflects an estimate of the percentage of species removed from the resin during the elution phase and is based on integration of the available analytical data. The reported values do not correspond to mass balances with respect to loaded species.

(b) Total species eluted from resin (meq).

(c) Total species eluted from the column (meq/g) on a dry resin H⁺-form weight basis.

The effect of analyte preconcentration on the resin is clearly demonstrated by comparing the ^{90}Sr loading and elution data. From the loading breakthrough data, it is not apparent that strontium was loaded onto the resin. The feed concentration was 1830 and 830 nCi/mL ^{90}Sr in 101-SY and 103-SY, respectively. All of the load samples exhibited approximately the same concentrations and, therefore, the data have little relevance. In contrast, the elution samples displayed a typical elution peak profile, with the ^{90}Sr concentration increasing from 2.6 (1.6) nCi/mL initially to a peak of 900 (572) nCi/mL before decreasing back to 2.1 (1.3) nCi/mL for the 101-SY (103-SY) experiment. The elution data indicate that trace strontium is picked up by the resin during the loading phase even though the effect is not visible from the loading breakthrough data. These data correspond to $1.42\text{E-}02$ ($1.93\text{E-}03$) Sr C/C_0 prior to elution and a peak of nearly 0.49 (0.69) Sr C/C_0 before returning to the previous value in the 101-SY (103-SY) actual waste tests. By integrating the area under the curve and making several assumptions, one can estimate that essentially no strontium is removed from the waste ($2.80\text{E-}09$ mol Sr removed vs. $6.0\text{E-}04$ mol Sr total in the 800 mL of 101-SY feed). Clearly, the extent of Sr loading is insignificant when compared to the total amount of strontium present in the feed solution.

Following the cesium ion exchange loading process, the solution exiting the column will have essentially the same chemical composition as the solution entering the column, except that the cesium concentration will be significantly lower. The extent of cesium decontamination will be a function of the volume of solution passed through the column. However, during the elution phase, the solution exiting the column will contain a higher concentration of cesium than existed in the original feed solution. In addition, other waste components (see Table 3.4) are removed from the resin during the elution process. Assuming that approximately 4 CV are required to elute the column and using the data from Table 3.4, the concentration of each species in the 0.5 M HNO_3 elution composite can be estimated (Table 3.5). Concentration of the eluant by evaporation would not change the relative composition of the composite, but would increase each species overall concentration.

Table 3.5. Species Concentration in Actual 101-SY and 103-SY Elution Composites

Species	Species Concentration (M)	
	101-SY	103-SY
Cs	4.44E-04	3.06E-04
Sr	6.73E-08	4.40E-08
Ni	1.55E-03	6.27E-04
Al	1.76E-03	1.88E-03
Ca	6.90E-05	0.00E+00
Cr	2.31E-04	2.69E-04
K	1.29E-04	1.60E-04
Na	2.17E-01	2.88E-01

3.6 3 M HNO₃ Stripping of CS-100

Numerous researchers have voiced concern that unknown species may remain on the CS-100 resin even after elution with 0.5 M HNO₃, and that these species may interfere with additional loading cycles. In the worst case, the entire capacity of an ion exchange resin might be occupied by species which exist only at ultratrace or even undetectable levels in the actual waste. The cesium loading would continually decrease as a larger and larger percentage of the ion exchange sites is filled with these retained species. For these reasons, a 3 M HNO₃ strip step was attempted to ascertain whether or not additional species are retained by the resin and removed with stronger acid. This step was attempted only for the 101-SY actual waste test and the 103-SY simulant test. Although the data are incomplete, some trends are apparent. The data clearly indicate that residual cesium and strontium remain on the solid ion exchange resin even after elution with nearly 8 CV of 0.5 M HNO₃. Although not specifically investigated in this experiment, these nonelutable species may contribute to resin fouling during subsequent loading cycles.

During the 103-SY simulant experiment, the cesium concentration increased 15-fold from 0.721 µg/L (1.02E-04 C/C₀) to 10.7 µg/L (1.52E-03 C/C₀) from the last elution sample to the strip sample. Additional elemental analysis by ICP-AES of the simulant effluent was not completed. During the 101-SY actual waste test, cesium in the effluent increased by a factor of two, from 0.082 to 0.15 µCi/mL. Strontium increased from 1.1 nCi/mL to 13.4 nCi/mL under the same conditions. It should be noted, however, that the feed Sr level was 1.81 µCi/mL and thus these values are 6.1E-04 and 7.4E-03 C/C₀, respectively. Unfortunately, an ICP-AES analysis was not completed because of an insufficient amount of sample. In addition, the stripping procedure was not performed during the 103-SY test for safety reasons. A large volume of gas was generated within the column after adding the 3 M HNO₃. The solution exiting the column was dark brown and contained bubbles. This behavior was not observed during simulant tests and, for safety reasons, the experiment was not repeated on the 103-SY waste. The reason for the degradation is unknown, but may be related to radiation exposure from the actual waste or the presence of additional species in the actual waste solution which catalyze the decomposition reaction. The 3 M HNO₃ strip cycle is not in the reference flow sheet for cesium ion exchange (Eager et al. 1994) and is not recommended as a process step because of the gas generation.

3.7 Resin Regeneration and Disposal

Following the elution or stripping phase (Sections 3.4 through 3.6), the CS-100 resin was prepared for additional column loading and elution cycles by rinsing with 3 CV H₂O followed by regeneration with 3 CV of 2 M NaOH. In general, the concentration of most species (excluding sodium) continued to decrease with the additional solution volumes passed through the column. The notable exception was TOC, which increased from 0.26- to 2.90-g C/L for the 101-SY actual waste test. However, the results were below the detection limit (<0.06 g C/L) for the 103-SY actual waste test. These results corroborate the visible observations and suggest that the 3 M HNO₃ stripping step (101-SY actual waste test only) is detrimental to the stability of the organic resin. The increased TOC observed in the regeneration cycle is likely due to the pH shock-induced degradation of the organic ion exchange resin.

After the actual waste experiments were completed, the resin was removed from the column and the residual radioactivity was measured by gamma energy analysis. For the 101-SY and 103-SY actual waste tests, a total of 0.995- and 6.15- μCi ^{137}Cs was measured. Assuming a 12-mL column containing 4.35 g of resin, these values can be converted into 0.085 Ci m^{-3} (230 nCi g^{-1}) and 0.50 Ci m^{-3} (1400 nCi g^{-1}), respectively. These values correspond to a residual cesium level of 1.35E-07 and 8.36E-07 mmol per gram of dried resin, negligible amounts when compared to the total resin capacity. The high HNO_3 stripping step can be used to further reduce the residual ^{137}Cs on the ion exchange resin.

3.8 Column Loading Parameters

The column loading parameters were determined using an optimization routine previously described in Kurath et al. (1994). These parameters are the Freundlich coefficient (K) and mass transfer coefficient (K_{pa}). The Freundlich isotherm was used to obtain the relationship between the cesium concentration on the exchanger and the cesium concentration in solution and is shown in Equation 3.1.

$$[\text{Cs}_s] = K [\text{Cs}_l]^n \quad (3.1)$$

where $[\text{Cs}_s]$ is the concentration of cesium on the exchanger at equilibrium with the cesium in the liquid, $[\text{Cs}_l]$ is the concentration of cesium in the liquid, and n is an experimentally derived exponent obtained from an empirical fit of previous cesium batch distribution data equal to 0.734. The number of transfer units, N , was computed using the following expression:

$$N = \frac{\lambda K_{pa} v}{f} \quad (3.2)$$

where K_{pa} is the mass transfer coefficient (min^{-1} or hr^{-1}), f is the solution flowrate (mL/hr), and v is the volume of the resin bed (mL).

Table 3.6 shows the results of these analyses. The mass transfer coefficients determined for the simulant runs are nearly identical, as expected, since the experimental conditions were similar. The mass transfer coefficients for the actual waste runs are somewhat higher, although it is not known if this difference is significant. It could be that this results from a slightly higher flowrate, which would enhance the rate of mass transfer in the fluid phase and increase K_{pa} . Previously reported values of K_{pa} derived from 200-mL column runs with NCAW and similar residence times were about 0.02 min^{-1} , roughly twice as high as the values determined for these experiments. This difference is most likely a reflection of the greater superficial velocities, factors that tend to increase the film-phase mass transfer coefficient.

Table 3.6. Column Loading Parameters

Feed Solution	Flowrate mL hr ⁻¹ (CV/hr ⁻¹)	K	K _{pa} (min ⁻¹)	λ	N
101-SY-Sim	61.1 (5.03)	0.51	0.0092	46.8	5.12
101-SY-Act	72.0 (6.10)	0.48	0.0116	43.7	4.84
103-SY-Sim	68.5 (5.63)	0.53	0.0090	46.0	4.39
103-SY-Act	77.7 (6.30)	0.40	0.0149	34.0	4.83

4.0 Conclusions

Based on the results of the column loading and elution experiments using simulated and actual waste from Hanford tanks 241-SY-101 and 241-SY-103, the following observations and conclusions are made:

- The removal of cesium from actual and simulated 101-SY and 103-SY tank waste was demonstrated using ~ 12-mL columns containing CS-100 ion exchange material. Decontamination factors > 1000 were obtained for the first seven column volumes of 101-SY feed and the first five column volumes of 103-SY feed processed. While these experiments do not demonstrate a DF during multiple load/elute cycles, they do demonstrate that in principle, fairly high DFs can be achieved with actual waste and fresh CS-100 material.
- Approximately 41.4 and 36.8 CV of 101-SY and 103-SY solutions were required to load the CS-100 exchanger to 0.5 C/C₀, which was slightly less than the values (52 and 50) predicted by previous Cs batch K_d analysis in either CC or NCAW simulants. The reason for this difference is not known and could be due to differences between the waste compositions, resin density, loading of additional noncesium species, or degradation of the CS-100 resin during storage.
- The ion exchange process appeared to have little effect on the ⁹⁰Sr, ⁹⁹Tc, and AT content of the wastes. As expected, very little (< 0.001 %) of the ⁹⁰Sr was removed from the waste during the loading step. This is expected because the ⁹⁰Sr is thought to be solubilized by organic complexants and is probably present as a neutral or anionic complex. The AT was generally below the detection limit, so it is not possible to assess the extent of removal of these components. Very little ⁹⁹Tc was removed since it is thought to be present as the pertechnetate anion and is not affected by cation exchange materials.
- Based on an analysis of the eluant solutions, the resin appears to concentrate only cesium and nickel. However, the resin has an affinity for small amounts of potassium, strontium, and possibly aluminum and chromium. The concentration of these components in the eluant composite relative to the feed was low.
- The performance of the simulants was very similar to that of the actual waste despite some small differences in composition. These differences were either not significant, a result of analytical inaccuracy, or canceled each other. Elution of cesium with 0.5 M HNO₃ required approximately 4.2 and 3.6 CV to reach 0.1 C/C₀ for the simulated and actual wastes. This indicates that an additional 0.6 CV of 0.5 M nitric acid is required during actual waste processing. The need for the additional nitric acid could be due to additional waste components that require neutralization.

- After elution, stripping the resin with 3 M HNO₃ released additional residual cesium and other components. After the actual waste testing was completed, analysis revealed approximately 0.085- and 0.50-Ci ¹³⁷Cs/m³ remained on the stripped (101-SY) and unstripped (103-SY) resins, respectively. These values correspond to a residual cesium level of 1.35E-07 and 8.36E-07 mmol per gram of dried resin.

5.0 References

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Appendix A

Laboratory Data from Test Runs

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Table A.1. ICP-AES Instrument Characteristics and Performance

<u>Element</u>	<u>Wavelength (nm)</u>	<u>Estimated IDL (ppm)</u>	<u>EQL (ppm)</u>	<u>Linear Dynamic Range (ppm)</u>
Ag	328.068	0.00206	0.01	0.003-150
Al	308.215	0.00984	0.05	0.025-500
As	193.696	0.02291	0.10	0.050-250
B	249.678	0.00626	0.05	0.006-150
Ba	455.403	0.00031	0.05	0.001-100
Be	313.042	0.00025	0.005	0.001-150
Bi	223.061	0.02461	0.10	0.030-500
Ca	317.933	0.00717	0.10	0.010-500
Cd	226.502	0.00186	0.01	0.004-200
Ce	413.765	0.03011	0.10	0.050-250
Co	228.616	0.00341	0.02	0.003-150
Cr	267.716	0.00204	0.01	0.005-150
Cu	324.754	0.00159	0.01	0.002-150
Eu	393.948	0.00411	0.05	0.005-100
Fe	259.940	0.00261	0.05	0.005-150
K	766.491	0.32211	0.50	0.400-1000
La	408.672	0.00231	0.05	0.004-150
Li	670.784	0.00063	0.01	0.006-200
Mg	279.079	0.01041	0.10	0.015-500
Mn	257.610	0.00042	0.01	0.001-150
Mo	202.030	0.00552	0.05	0.005-200
Na	588.995	0.00698	0.10	0.010-200
Nd	401.225	0.00469	0.10	0.010-200
Ni	231.604	0.01411	0.02	0.010-200
P	178.287	0.03041	0.20	0.060-250
Pb	220.353	0.02721	0.10	0.025-200
S	182.040	0.01971	0.10	0.080-500
Sb	206.838	0.01811	0.06	0.050-200
Se	196.026	0.03491	0.10	0.050-250
Si	251.611	0.00555	0.05	0.010-500
Sm	443.430	0.00943	0.10	0.020-100
Sr	421.552	0.00031	0.01	0.001-100
Th	283.730	0.00316	0.05	0.030-150
Ti	334.941	0.00112	0.01	0.002-200
Tl	190.864	0.03411	0.20	0.050-500
U	385.958	0.03451	0.50	0.100-300
V	292.402	0.00256	0.05	0.002-200
Zn	213.856	0.00238	0.01	0.004-150
Zr	339.198	0.00202	0.01	0.003-250

Table A.2. Cesium Loading Data for 101-SY Actual Waste

Time (Hr)	Sample Number	Vol. (CV)	Vol. (mL)	Cesium (uCi/mL)	Cesium (mg/L)	Cesium (C/C ₀)	Loaded (mmol Cs)
0.00	1SYFEED1	0.00	0.00	1.40E+02	6.30E+00	1.00E+00	3.25E-02
0.50	1SYLOD01	3.13	36.92	6.50E-02	2.93E-03	4.64E-04	1.75E-03
1.00	1SYLOD02	6.20	73.11	8.50E-02	3.83E-03	6.07E-04	3.46E-03
1.50	1SYLOD03	9.23	108.90	5.78E-01	2.60E-02	4.13E-03	5.16E-03
2.00	1SYLOD04	12.26	144.72	2.37E+00	1.07E-01	1.69E-02	6.84E-03
2.50	1SYLOD05	15.35	181.14	5.98E+00	2.69E-01	4.27E-02	8.51E-03
3.00	1SYLOD06	18.40	217.10	1.15E+01	5.18E-01	8.21E-02	1.01E-02
3.50	1SYLOD07	21.47	253.31	1.90E+01	8.55E-01	1.36E-01	1.16E-02
4.00	1SYLOD08	24.41	288.08	2.62E+01	1.18E+00	1.87E-01	1.30E-02
4.50	1SYLOD09	27.50	324.48	3.74E+01	1.68E+00	2.67E-01	1.44E-02
5.00	1SYLOD10	30.59	360.99	4.48E+01	2.02E+00	3.20E-01	1.56E-02
5.50	1SYLOD11	33.66	397.18	4.99E+01	2.25E+00	3.56E-01	1.67E-02
6.00	1SYLOD12	36.66	432.60	6.16E+01	2.77E+00	4.40E-01	1.77E-02
6.50	1SYLOD13	39.70	468.46	6.77E+01	3.05E+00	4.84E-01	1.86E-02
7.00	1SYLOD14	42.75	504.45	7.55E+01	3.40E+00	5.39E-01	1.95E-02
7.50	1SYLOD15	45.81	540.61	7.98E+01	3.59E+00	5.70E-01	2.02E-02
8.00	1SYLOD16	48.91	577.12	8.99E+01	4.05E+00	6.42E-01	2.09E-02
8.50	1SYLOD17	52.00	613.66	8.92E+01	4.01E+00	6.37E-01	2.15E-02
9.00	1SYLOD18	55.08	650.00	9.55E+01	4.30E+00	6.82E-01	2.21E-02
9.50	1SYLOD19	58.14	686.01	1.01E+02	4.55E+00	7.21E-01	2.26E-02
0.25	1SYWash1	1.65	19.46	5.34E+01	2.40E+00	3.81E-01	
0.50	1SYWash2	3.16	37.26	2.71E+01	1.22E+00	1.94E-01	
0.25	1SYRins1	1.47	17.35	2.18E+01	9.81E-01	1.56E-01	
0.50	1SYRins2	3.05	35.98	7.32E+00	3.29E-01	5.23E-02	

Table A.3. Cesium Elution Data for 101-SY Actual Waste

<u>Time</u> <u>(Hr)</u>	<u>Sample</u> <u>Number</u>	<u>Vol.</u> <u>(CV)</u>	<u>Vol.</u> <u>(mL)</u>	<u>Cesium</u> <u>(uCi/mL)</u>	<u>Cesium</u> <u>(mg/L)</u>	<u>Cesium</u> <u>(C/C₀)</u>	<u>Eluted</u> <u>(mmol Cs)</u>
0.50	1SYELU01	0.51	5.97	4.72E+00	2.12E-01	3.37E-02	4.77E-06
1.00	1SYELU02	1.02	12.07	9.35E+00	4.21E-01	6.68E-02	1.93E-05
1.50	1SYELU03	1.55	18.23	2.19E+01	9.86E-01	1.56E-01	5.19E-05
2.00	1SYELU04	2.08	24.54	2.64E+01	1.19E+00	1.89E-01	1.03E-04
2.50	1SYELU05	2.61	30.74	5.04E+03	2.27E+02	3.60E+01	5.42E-03
3.00	1SYELU06	3.12	36.80	5.28E+03	2.38E+02	3.77E+01	1.60E-02
3.50	1SYELU07	3.64	42.98	3.99E+02	1.80E+01	2.85E+00	2.19E-02
4.00	1SYELU08	4.09	48.31	1.52E+01	6.84E-01	1.09E-01	2.23E-02
4.50	1SYELU09	4.53	53.42		0.00E+00	0.00E+00	2.23E-02
5.00	1SYELU10	4.91	57.93	2.12E+00	9.54E-02	1.51E-02	2.23E-02
5.50	1SYELU11	5.30	62.58		0.00E+00	0.00E+00	2.23E-02
6.00	1SYELU12	5.96	70.28	3.71E-01	1.67E-02	2.65E-03	2.23E-02
6.50	1SYELU13	6.70	79.10		0.00E+00	0.00E+00	2.23E-02
7.00	1SYELU14	7.38	87.14	8.16E-02	3.67E-03	5.83E-04	2.23E-02
0.67	1SYStrip	3.16	37.34	1.50E-01	6.75E-03	1.07E-03	0.00E+00
0.67	1SYClean	3.05	35.95	2.17E-02	9.77E-04	1.55E-04	0.00E+00
0.75	1SYFinal	2.66	31.33	1.85E-02	8.33E-04	1.32E-04	0.00E+00

Table A.4. Cesium Loading Data for 101-SY Simulated Waste

Time (Hr)	Sample Number	Vol. (CV)	Vol. (mL)	Cesium (Counts)	Cesium (mg/L)	Cesium C/C ₀	Loaded (mmol Cs)
0.00	1SYA-LF _d	0.00	0.00	4.52E+04	5.54E+00	1.00E+00	3.44E-02
0.50	1SYA-L1	2.54	30.93	3.05E+00	3.74E-04	6.75E-05	1.29E-03
1.00	1SYA-L2	5.00	60.86	7.97E+00	9.79E-04	1.77E-04	2.54E-03
1.50	1SYA-L3	7.58	92.23	4.85E+01	5.95E-03	1.07E-03	3.84E-03
2.00	1SYA-L4	10.10	122.88	2.30E+02	2.82E-02	5.09E-03	5.12E-03
2.50	1SYA-L5	12.62	153.58	6.69E+02	8.21E-02	1.48E-02	6.39E-03
3.00	1SYA-L6	15.16	184.54	1.55E+03	1.91E-01	3.44E-02	7.65E-03
3.50	1SYA-L7	17.65	214.83	2.79E+03	3.42E-01	6.17E-02	8.85E-03
4.00	1SYA-L8	20.12	244.87	4.32E+03	5.30E-01	9.57E-02	1.00E-02
4.50	1SYA-L9	22.69	276.10	6.17E+03	7.57E-01	1.37E-01	1.12E-02
5.00	1SYA-L10	25.10	305.41	8.32E+03	1.02E+00	1.84E-01	1.22E-02
5.50	1SYA-L11	27.75	337.73	1.06E+04	1.30E+00	2.34E-01	1.32E-02
6.00	1SYA-L12	30.23	367.89	1.27E+04	1.56E+00	2.82E-01	1.42E-02
6.50	1SYA-L13	32.74	397.40	1.50E+04	1.84E+00	3.32E-01	1.51E-02
7.00	1SYA-L14	35.25	429.05	1.69E+04	2.08E+00	3.75E-01	1.59E-02
7.50	1SYA-L15	37.79	459.87	1.92E+04	2.36E+00	4.26E-01	1.67E-02
8.00	1SYA-L16	40.30	490.51	2.18E+04	2.67E+00	4.82E-01	1.74E-02
8.50	1SYA-L17	42.82	521.07	2.36E+04	2.90E+00	5.23E-01	1.80E-02
9.00	1SYA-L18	45.34	551.78	2.54E+04	3.12E+00	5.62E-01	1.86E-02
9.50	1SYA-L19	47.85	582.32	2.71E+04	3.32E+00	6.00E-01	1.91E-02
10.00	1SYA-L20	50.38	613.15	2.83E+04	3.48E+00	6.28E-01	1.96E-02
10.50	1SYA-L21	52.89	643.73	2.95E+04	3.62E+00	6.53E-01	2.01E-02
11.00	1SYA-L22	55.41	674.36	3.02E+04	3.71E+00	6.69E-01	2.05E-02
11.50	1SYA-L23	57.94	705.08	3.13E+04	3.84E+00	6.92E-01	2.09E-02
12.00	1SYA-L24	60.45	735.72	3.37E+04	4.14E+00	7.47E-01	2.13E-02
12.50	1SYA-L25	62.96	766.23	3.47E+04	4.25E+00	7.68E-01	2.16E-02
13.00	1SYA-L26	65.49	796.98	3.54E+04	4.35E+00	7.84E-01	2.19E-02
13.50	1SYA-L27	67.88	826.10	3.61E+04	4.43E+00	7.99E-01	2.21E-02
0.50	1SYA-N29	2.45	29.86	2.04E+04	2.51E+00	4.52E-01	
1.00	1SYA-N30	4.98	60.58	9.39E+03	1.15E+00	2.08E-01	
1.25	1SYA-N31	6.40	77.83	8.76E+03	1.07E+00	1.94E-01	
0.25	1SYA-W33	1.56	18.95	8.13E+03	9.98E-01	1.80E-01	
0.50	1SYA-W34	3.39	41.26	2.68E+03	3.29E-01	5.93E-02	
0.75	1SYA-W35	4.78	58.12	1.15E+03	1.41E-01	2.55E-02	

Table A.5. Cesium Elution Data for 101-SY Simulated Waste

<u>Time</u> <u>(Hr)</u>	<u>Sample</u> <u>Number</u>	<u>Vol.</u> <u>(CV)</u>	<u>Vol.</u> <u>(mL)</u>	<u>Cesium</u> <u>(Counts)</u>	<u>Cesium</u> <u>(mg/L)</u>	<u>Cesium</u> <u>(C/C₀)</u>	<u>Eluted</u> <u>(mmol Cs)</u>
0.50	1SYA-E40	0.50	6.12	1.04E+03	1.27E-01	2.29E-02	2.93E-06
1.00	1SYA-E41	1.01	12.29	5.19E+03	6.37E-01	1.15E-01	2.07E-05
1.50	1SYA-E42	1.53	18.58	1.37E+04	1.68E+00	3.04E-01	7.55E-05
2.00	1SYA-E43	2.06	25.02	9.36E+05	1.15E+02	2.07E+01	2.90E-03
2.50	1SYA-E44	2.54	30.85	2.71E+06	3.33E+02	6.00E+01	1.27E-02
3.00	1SYA-E45	3.06	37.26	4.52E+04	5.55E+00	1.00E+00	2.09E-02
3.50	1SYA-E46	3.56	43.27	2.97E+03	3.64E-01	6.57E-02	2.10E-02
4.00	1SYA-E47	4.08	49.70	1.65E+03	2.03E-01	3.66E-02	2.10E-02
4.50	1SYA-E48	4.58	55.76	7.25E+02	8.89E-02	1.60E-02	2.10E-02
5.00	1SYA-E49	5.09	61.91	3.49E+02	4.28E-02	7.72E-03	2.10E-02
5.50	1SYA-E50	5.59	67.99	1.32E+02	1.61E-02	2.91E-03	2.10E-02
6.00	1SYA-E51	6.09	74.17	8.75E+01	1.07E-02	1.94E-03	2.10E-02
6.50	1SYA-E52	6.60	80.32	7.47E+01	9.16E-03	1.65E-03	2.10E-02

Table A.6. Cesium Loading Data for 103-SY Actual Waste

<u>Time (Hr)</u>	<u>Sample Number</u>	<u>Vol. (CV)</u>	<u>Vol. (mL)</u>	<u>Cesium (uCi/mL)</u>	<u>Cesium (mg/L)</u>	<u>Cesium C/C₀</u>	<u>Loaded (mmol Cs)</u>
0.00	3SYFEED1	0.00	0.00	1.61E+02	7.20E+00	1.00E+00	2.39E-02
0.50	3SYLOD01	3.26	40.22	0.00E+00	0.00E+00	0.00E+00	2.18E-03
1.00	3SYLOD02	6.46	79.65	3.72E-01	1.66E-02	2.31E-03	4.31E-03
1.50	3SYLOD03	9.71	119.71	2.93E+00	1.31E-01	1.82E-02	6.46E-03
2.00	3SYLOD04	12.89	158.98	9.19E+00	4.11E-01	5.71E-02	8.51E-03
2.50	3SYLOD05	16.18	199.46	1.75E+01	7.83E-01	1.09E-01	1.05E-02
3.00	3SYLOD06	19.42	239.43	2.83E+01	1.27E+00	1.76E-01	1.24E-02
3.50	3SYLOD07	22.59	278.48	3.95E+01	1.77E+00	2.45E-01	1.40E-02
4.00	3SYLOD08	25.77	317.69	5.20E+01	2.33E+00	3.23E-01	1.56E-02
4.50	3SYLOD09	28.97	357.19	6.13E+01	2.74E+00	3.81E-01	1.70E-02
5.00	3SYLOD10	32.13	396.20	7.80E+01	3.49E+00	4.84E-01	1.82E-02
5.50	3SYLOD11	35.21	434.09	7.88E+01	3.52E+00	4.89E-01	1.92E-02
6.00	3SYLOD12	35.74	440.64	7.37E+01	3.30E+00	4.58E-01	1.94E-02
0.25	3SYWash1	1.57	19.30	5.70E+01	2.55E+00	3.54E-01	
0.50	3SYWash2	3.18	39.16	2.50E+01	1.12E+00	1.55E-01	
0.25	3SYRins1	1.66	20.42	1.70E+01	7.60E-01	1.06E-01	
0.50	3SYRins2	3.24	39.93	4.90E+00	2.19E-01	3.04E-02	

Table A.7. Cesium Elution Data for 103-SY Actual Waste

<u>Time (Hr)</u>	<u>Sample Number</u>	<u>Vol. (CV)</u>	<u>Vol. (mL)</u>	<u>Cesium (uCi/mL)</u>	<u>Cesium (mg/L)</u>	<u>Cesium (C/C₀)</u>	<u>Eluted (mmol Cs)</u>
0.50	3SYELU01	0.87	10.78	3.99E+00	1.78E-01	2.48E-02	7.24E-06
1.00	3SYELU02	1.61	19.83	1.50E+01	6.71E-01	9.32E-02	3.62E-05
1.50	3SYELU03	2.31	28.51	8.44E+02	3.77E+01	5.24E+00	1.29E-03
2.00	3SYELU04	3.04	37.46	4.50E+03	2.01E+02	2.80E+01	9.34E-03
2.50	3SYELU05	3.62	44.66	1.43E+02	6.40E+00	8.88E-01	1.50E-02
3.00	3SYELU06	4.21	51.97	9.40E+01	4.20E+00	5.84E-01	1.53E-02
3.50	3SYELU07	4.81	59.27				
4.00	3SYELU08	5.41	66.65	1.03E+00	4.61E-02	6.40E-03	1.54E-02
4.50	3SYELU09	6.00	73.96				
5.00	3SYELU10	6.66	82.09	2.60E-01	1.16E-02	1.61E-03	1.54E-02
5.50	3SYELU11	7.24	89.30				
6.00	3SYELU12	7.84	96.66	1.10E-01	4.92E-03	6.83E-04	1.54E-02
0.50	3SYClean	3.07	37.81	9.60E-02	4.29E-03	5.96E-04	3.70E-04
1.00	3SYFinal	3.03	37.34	3.60E-02	1.61E-03	2.24E-04	

Table A.8. Cesium Loading Data for 103-SY Simulated Waste

Time (Hr)	Sample Number	Vol. (CV)	Vol. (mL)	Cesium (Counts)	Cesium (mg/L)	Cesium C/C ₀	Loaded (mmol Cs)
0.00	3SYB-LF _d	0.0	0.00	4.55E+04	7.04E+00	1.00E+00	4.90E-02
0.50	3SYB-L1	2.62	31.94	4.72E+00	7.31E-04	1.04E-04	1.69E-03
1.00	3SYB-L2	4.79	58.33	9.69E+00	1.50E-03	2.13E-04	3.09E-03
1.50	3SYB-L3	7.65	93.05	3.32E+02	5.14E-02	7.29E-03	4.92E-03
2.00	3SYB-L4	10.49	127.72	1.28E+03	1.99E-01	2.82E-02	6.73E-03
2.50	3SYB-L5	13.38	162.83	2.71E+03	4.19E-01	5.95E-02	8.51E-03
3.00	3SYB-L6	16.22	197.40	4.55E+03	7.05E-01	1.00E-01	1.02E-02
3.50	3SYB-L7	19.07	232.14	7.41E+03	1.15E+00	1.63E-01	1.18E-02
4.00	3SYB-L8	21.86	266.09	9.85E+03	1.53E+00	2.17E-01	1.33E-02
4.50	3SYB-L9	24.70	300.64	1.28E+04	1.98E+00	2.81E-01	1.46E-02
5.00	3SYB-L10	27.55	335.24	1.57E+04	2.44E+00	3.46E-01	1.59E-02
5.50	3SYB-L11	30.38	369.69	1.82E+04	2.81E+00	3.99E-01	1.70E-02
6.00	3SYB-L12	33.21	404.22	2.02E+04	3.13E+00	4.45E-01	1.81E-02
6.50	3SYB-L13	36.11	439.42	2.23E+04	3.45E+00	4.90E-01	1.91E-02
7.00	3SYB-L14	38.93	473.74	2.42E+04	3.75E+00	5.33E-01	2.00E-02
7.50	3SYB-L15	41.73	507.86	2.62E+04	4.05E+00	5.75E-01	2.08E-02
8.00	3SYB-L16	44.67	543.68	2.78E+04	4.31E+00	6.12E-01	2.15E-02
8.50	3SYB-L17	47.42	577.10	2.94E+04	4.55E+00	6.46E-01	2.22E-02
9.00	3SYB-L18	50.28	611.93	3.09E+04	4.78E+00	6.79E-01	2.28E-02
9.50	3SYB-L19	53.12	646.45	3.21E+04	4.97E+00	7.05E-01	2.34E-02
10.00	3SYB-L20	55.93	680.73	3.35E+04	5.19E+00	7.37E-01	2.39E-02
10.50	3SYB-L21	58.91	716.98	3.24E+04	5.02E+00	7.13E-01	2.44E-02
11.00	3SYB-L22	61.75	751.49	3.38E+04	5.24E+00	7.44E-01	2.49E-02
11.50	3SYB-L23	64.57	785.77	3.55E+04	5.50E+00	7.81E-01	2.54E-02
12.00	3SYB-L24	67.38	819.98	3.64E+04	5.63E+00	7.99E-01	2.57E-02
12.50	3SYB-L25	70.20	854.34	3.70E+04	5.73E+00	8.14E-01	2.61E-02
13.00	3SYB-L26	73.04	888.90	3.80E+04	5.89E+00	8.36E-01	2.64E-02
13.50	3SYB-L27	76.01	924.99	3.74E+04	5.79E+00	8.21E-01	2.67E-02
0.13	3SYB-N31	0.68	8.29	8.96E+03	1.39E+00	1.97E-01	
0.25	3SYB-N32	1.30	15.88	9.69E+03	1.50E+00	2.13E-01	
0.38	3SYB-N33	2.03	24.65	9.55E+03	1.48E+00	2.10E-01	
0.50	3SYB-N34	2.73	33.27	9.25E+03	1.43E+00	2.03E-01	
0.13	3SYB-W35	0.67	8.15	9.07E+03	1.41E+00	1.99E-01	
0.25	3SYB-W36	1.44	17.53	7.50E+03	1.16E+00	1.65E-01	
0.38	3SYB-W37	2.20	26.72	3.53E+03	5.47E-01	7.77E-02	
0.50	3SYB-W38	2.93	35.67	2.45E+03	3.80E-01	5.40E-02	

Table A.9. Cesium Elution Data for 103-SY Simulated Waste

Time (Hr)	Sample Number	Vol. (CV)	Vol. (mL)	Cesium (Counts)	Cesium (mg/L)	Cesium (C/C ₀)	Eluted (mmol Cs)
0.50	3SYB-E39	0.51	6.25	2.41E+03	3.73E-01	5.29E-02	8.77E-06
1.00	3SYB-E40	1.04	12.61	9.15E+03	1.42E+00	2.01E-01	5.16E-05
1.50	3SYB-E41	1.57	19.08	1.41E+04	2.18E+00	3.09E-01	1.39E-04
2.00	3SYB-E42	2.11	25.65	6.83E+05	1.06E+02	1.50E+01	2.81E-03
2.50	3SYB-E43	2.64	32.08	2.25E+06	3.48E+02	4.94E+01	1.38E-02
3.00	3SYB-E44	3.16	38.51	1.63E+05	2.53E+01	3.59E+00	2.28E-02
3.50	3SYB-E45	3.69	44.87	5.34E+03	8.27E-01	1.17E-01	2.34E-02
4.00	3SYB-E46	4.22	51.32	2.04E+03	3.16E-01	4.48E-02	2.35E-02
4.50	3SYB-E47	4.75	57.78	7.23E+02	1.12E-01	1.59E-02	2.35E-02
5.00	3SYB-E48	5.27	64.19	2.22E+02	3.44E-02	4.88E-03	2.35E-02
5.50	3SYB-E49	5.79	70.49	7.78E+01	1.21E-02	1.71E-03	2.35E-02
6.00	3SYB-E50	6.31	76.79	5.91E+00	9.16E-04	1.30E-04	2.35E-02
6.50	3SYB-E51	6.84	83.25	-9.35E+00	-1.45E-03	-2.06E-04	2.35E-02
7.00	3SYB-E52	7.37	89.64	4.65E+00	7.21E-04	1.02E-04	2.35E-02
8.00	3SYB-E53	8.45	102.86	6.92E+01	1.07E-02	1.52E-03	2.35E-02
9.00	3SYB-E54	8.45	102.86	3.28E+02	5.08E-02	7.22E-03	2.35E-02

Table A.10. TOC and Radionuclide Analyses for Actual 101-SY Load/Wash/Rinse Samples

Sample Number	Vol. (mL)	TOC (g/L)	¹³⁷ Cs (μCi/mL)	AT (μCi/mL)	⁹⁰ Sr (μCi/mL)	⁹⁹ Tc (μCi/mL)
1SYFEED1 ^(a)	-- ^(b)	4.27	140	< 0.0010	1.83	0.030
1SYLOD01	36.3	--	< 0.065	< 0.0005	--	--
1SYLOD02	36.2	--	< 0.085	< 0.0005	1.73	--
1SYLOD03	35.8	--	0.58	< 0.0005	--	--
1SYLOD04	35.8	4.30	2.37	< 0.0005	1.81	0.027
1SYLOD05	36.4	--	5.98	< 0.0008	--	--
1SYLOD06	36.0	--	11.5	< 0.0005	1.69	--
1SYLOD07	36.2	--	19.0	< 0.0005	--	--
1SYLOD08	34.8	--	26.2	< 0.0008	1.78	--
1SYLOD09	36.4	--	37.4	< 0.0005	--	--
1SYLOD10	36.5	4.41	44.8	< 0.0005	1.69	0.030
1SYLOD11	36.2	--	49.9	< 0.0008	--	--
1SYLOD12	35.4	--	61.6	< 0.0006	1.70	--
1SYLOD13	35.9	--	67.7	< 0.0005	--	--
1SYLOD14	36.0	--	75.5	< 0.0007	1.69	--
1SYLOD15	36.2	--	79.8	< 0.0011	--	--
1SYLOD16	36.5	--	89.9	< 0.0005	1.66	--
1SYLOD17	36.5	--	89.2	< 0.0008	--	--
1SYLOD18	36.3	4.14	95.5	< 0.0005	1.85	0.024
1SYLOD19	36.0	--	101.0	< 0.0009	--	--
1SYWASH1	20.2	2.85	53.4	< 0.0011	1.01	0.0379
1SYWASH2	17.7	0.34	27.1	< 0.0011	0.0204	0.0029
1SYRINS1	17.7	0.205	21.8	< 0.0014	0.0077	0.0009
1SYRINS2	18.6	0.039	7.3	< 0.0007	0.0065	0.0004

(a) Average of duplicate samples.

(b) -- denotes analysis was not requested.

Table A.11. ICP-AES Results for Actual 101-SY Load/Wash/Rinse Samples

Sample Number	Elemental ICP-AES Results ($\mu\text{g/mL}$)											
	Si	Al	Cu	Ni	Ca	Cr	P	S	Mo	B	K	Na
1SYFEED1 ^(a)	132	14800	4.6	64	57	50	1170	720	43	30	1390	121000
1SYLOD01	121	9000	2.8	34	33	27	728	432	26	26	601	86200
1SYLOD02	130	13800	nd ^(b)	52	48	41	1070	650	39	30	1260	109000
1SYLOD04	128	13800	4.4	52	49	43	1090	658	40	30	1280	110000
1SYLOD10	125	13900	nd	53	49	45	1090	662	40	29	1280	111000
1SYLOD18	130	13700	4.4	57	50	42	1070	650	40	31	1280	110000
1SYWASH1	101	9310	2.8	29	32	13	720	428	24	20	1100	101000
1SYWASH2	119	263	nd	nd	nd	nd	nd	nd	nd	nd	330	43700
1SYRINS1	158	77	nd	nd	nd	nd	nd	nd	nd	17	nd	32600
1SYRINS2	54	22	nd	nd	nd	nd	nd	nd	nd	6	nd	4830

(a) Average of duplicate samples.

(b) not detected

Table A.12. TOC and Radionuclide Analyses for Actual 101-SY Elute/Regenerate Samples

Sample Number	Vol. (mL)	TOC (g/L)	¹³⁷ Cs ($\mu\text{Ci/mL}$)	AT ($\mu\text{Ci/mL}$)	⁹⁰ Sr ($\mu\text{Ci/mL}$)	⁹⁹ Tc ($\mu\text{Ci/mL}$)	
1SYFEED1	--	4.27	140	< 0.0010	1.83	0.030	
1SYELU01	6.0	--	4.7	< 0.0005	0.0026	--	
1SYELU02	6.1	0.046	9.4	< 0.0007	0.0034	0.0002	
1SYELU03	6.1	--	21.9	< 0.0014	0.096	--	
1SYELU04	6.3	0.070	26.4	< 0.0014	0.0021	--	
1SYELU05	5.7	0.104	5036	< 0.054	0.66	4.9E-4	
1SYELU06	5.6	--	5278	< 0.051	0.90	--	
1SYELU07	6.1	--	399	--	0.044	--	
1SYELU08	5.3	--	15.2	< 0.0014	0.004	--	
1SYELU09	5.1	--	--	--	--	--	
1SYELU10	4.5	0.091	2.12	< 0.0014	0.0026	7.3E-5	
1SYELU11	4.7	--	--	--	--	--	
1SYELU12	7.7	--	0.371	1.5E-5	0.0021	--	
1SYELU13	8.8	--	--	--	--	--	
1SYELU14	8.0	--	0.082	7.3E-6	0.0011	--	
1SYSTRIP	34.5	0.55	0.15	3.0E-5	0.0134	--	
1SYCLEAN	35.6	0.26	0.022	4.6E-6	0.0013	< 3.5E-5	
1SYFINAL	32.0	2.90	0.018	3.2E-6	7.5E-5	2.5E-5	
1SYRESIN			¹³⁷ Cs = 0.995 μCi total				

Table A.13. ICP-AES Results for Actual 101-SY Elute/Regenerate Samples

Sample Number	Elemental ICP-AES Results ($\mu\text{g/mL}$)											
	Si	Al	Cu	Ni	Ca	Cr	P	S	Mo	B	K	Na
1SYFEED1	132	14800	4.6	64	57	50	1170	720	43	30	1390	121000
1SYELU01	48	19	nd	0.4	nd	nd	nd	nd	nd	7.5	nd	2440
1SYELU02	45	14	nd	nd	nd	nd	nd	nd	nd	7.3	nd	3800
1SYELU03	26	9	nd	nd	nd	nd	nd	nd	nd	5.0	nd	8260
1SYELU04	16	21	nd	4.7	nd	0.6	nd	nd	nd	3.0	9.2	8990
1SYELU05	4	99	0.9	273	8	15	nd	nd	nd	24	13	7733
1SYELU06	4	105	0.8	285	8	27	nd	nd	nd	20	12	5020
1SYELU08	8	12	0.08	5.9	0.4	8.1	0.8	0.3	nd	6.8	nd	61
1SYELU010	13	10	nd	3.7	nd	6.9	nd	nd	nd	7.1	nd	35
1SYELU012	8	6	0.04	1.8	0.4	4.1	0.4	nd	nd	4.7	nd	22
1SYELU014	7	5	0.02	1.0	0.4	2.7	nd	nd	nd	3.8	nd	18
1SYCLEAN	5	3	0.04	0.6	0.5	1.6	0.3	0.1	nd	3.3	0.5	36
1SYFINAL	178	9	nd	nd	nd	2.5	nd	nd	nd	16	nd	18000

Table A.14. TOC and Radionuclide Analyses for Actual 103-SY Load/Wash/Rinse Samples

Sample Number	Vol (mL)	TOC (g/L)	^{137}Cs ($\mu\text{Ci/mL}$)	AT ($\mu\text{Ci/mL}$)	^{90}Sr ($\mu\text{Ci/mL}$)	^{99}Tc ($\mu\text{Ci/mL}$)
3SYFEED	--	2.84	161	0.0008	0.83	0.076
3SYLOD01	39.4	--	nd	--	--	--
3SYLOD02	39.5	--	0.372	--	0.77	--
3SYLOD03	40.1	--	2.93	3.5E-4	--	--
3SYLOD04	39.3	2.52	9.19	--	0.77	0.067
3SYLOD05	40.5	--	17.5	--	--	--
3SYLOD06	40.0	--	28.3	--	0.79	--
3SYLOD07	39.1	--	39.5	< 1.3E-3	--	--
3SYLOD08	39.2	--	52.0	--	0.80	--
3SYLOD09	39.5	--	61.3	--	--	--
3SYLOD10	39.0	3.09	78.0	--	0.78	0.052
3SYLOD11	37.9	--	78.8	< 2.6E-3	--	--
3SYLOD12	6.6	--	73.7	--	0.78	--
3SYWASH1	18.9	2.32	57	< 6.6E-4	0.38	0.045
3SYWASH2	19.8	1.40	25	< 1.3E-3	0.0084	0.0052
3SYRINS1	20.1	0.74	17	< 6.6E-4	0.0056	0.0013
3SYRINS2	19.5	< 0.06	4.9	< 3.3E-4	0.0010	0.0023

Table A.15. ICP-AES Results for Actual 103-SY Load/Wash/Rinse Samples

Sample Number	Elemental ICP-AES Results ($\mu\text{g/mL}$)											
	Si	Al	Cu	Ni	Ca	Cr	P	S	Mo	B	K	Na
3SYFEED1	72	16800	nd	19	nd	43	1520	830	54	40	1600	138000
3SYLOAD1	87	9160	nd	6	nd	22	816	508	30	3	663	90600
3SYLOD02	76	14100	nd	10	nd	36	1230	774	46	36	1320	116000
3SYLOD04	72	13800	nd	8	nd	34	1160	738	43	35	1300	115000
3SYLOD10	70	15200	nd	12	nd	39	1320	826	49	35	1440	125000
3SYWASH1	642	9260	nd	nd	nd	18	802	505	28	25	1070	104000
2SYWASH2	127	286	nd	nd	nd	nd	nd	nd	nd	nd	247	47300
3SYRINS1	132	60	nd	nd	nd	nd	nd	nd	nd	13	nd	30400
3SYRINS2	43	15	nd	nd	nd	nd	nd	25	nd	5	nd	3670

Table A.16. TOC and Radionuclide Analyses for Actual 103-SY Elute/Regenerate Samples

Sample Number	Vol. (mL)	TOC (g/L)	^{137}Cs ($\mu\text{Ci/mL}$)	AT ($\mu\text{Ci/mL}$)	^{90}Sr ($\mu\text{Ci/mL}$)	^{99}Tc ($\mu\text{Ci/mL}$)
3SYFEED1	--	2.84	161	0.0008	0.83	0.076
3SYELU01	10.8	--	4.0	< 2.2E-4	0.0016	--
3SYELU02	9.0	< 0.06	15	< 6.6E-4	0.0012	8.2E-5
3SYELU03	8.6	--	844	< 2.3E-3	0.122	--
3SYELU04	8.9	1.05	4500	< 6.7E-2	0.572	1.1E-3
3SYELU05	6.6	--	143	< 5.0E-4	0.111	--
3SYELU06	6.8	--	94	< 4.5E-3	0.040	--
3SYELU07	7.2	--	--	--	--	--
3SYELU08	7.4	--	1.03	< 1.0E-4	0.0020	--
3SYELU09	7.3	--	--	--	--	--
3SYELU10	8.1	0.06	0.26	4.9E-5	0.0013	8.2E-5
3SYELU11	7.2	--	--	--	--	--
3SYELU12	7.4	--	0.11	1.7E-5	0.0031	--
3SYCLEAN	37.4	< 0.06	0.096	9.6E-6	0.0008	7.8E-5
3SYFINAL	38.1	< 0.06	0.036	< 2.2E-6	0.00024	5.4E-5
3SYRESIN			$^{137}\text{Cs} = 6.15 \mu\text{Ci}$			

Table A.17. ICP-AES Results for Actual 103-SY Elute/Regenerate Samples

Sample Number	Elemental ICP-AES Results ($\mu\text{g/mL}$)											
	Si	Al	Cu	Ni	Ca	Cr	P	S	Mo	B	K	Na
3SYFEED1	72	16800	nd	19	nd	43	1520	830	54	40	1600	138000
3SYELU01	30	22	nd	0.4	nd	0.4	6	3.5	nd	4	nd	2900
3SYELU02	13	20	nd	nd	nd	nd	nd	nd	nd	nd	nd	8280
3SYELU03	nd	55	7	23	nd	nd	nd	nd	nd	nd	16	9180
3SYELU04	5	129	40	177	19	8	nd	4.0	nd	19	17	4220
3SYELU05	6	24	4	9	1	4	1.0	0.6	nd	7	0.7	105
3SYELU06	10	12	2	4	1	2	0.7	0.5	nd	7	nd	43
3SYELU08	5	7	1.1	1.7	0.4	1.3	0.6	0.3	nd	4.6	nd	24
3SYELU010	5	5	0.8	1.1	0.5	0.9	0.4	0.25	nd	5.0	nd	19
3SYELU012	5	4	0.6	0.9	nd	0.8	nd	nd	nd	4.6	nd	14
3SYCLEAN	2	5	0.1	0.2	0.4	0.5	0.4	0.2	nd	1.1	0.6	41
3SYFINAL	88	13	nd	nd	nd	nd	nd	nd	nd	9	nd	21500

Table A.18. Analytical Results for Blank Samples

Analyte	Units	Blank 1	Blank 2	Blank 3	Blank 4	Blank 5	Blank 6
		2M NaOH Backflush	H ₂ O Hotcell	2M NaOH Hotcell	0.5M HNO ₃ Hotcell	3M HNO ₃ Hotcell	2M NaOH Backflush
¹³⁷ Cs	$\mu\text{Ci/mL}$	<7.4E-2	<7.4E-5	<6.1E-5	<7.2E-5	1.4E-4	nd
AT	$\mu\text{Ci/mL}$	<5.0E-4	<1.1E-6	<4.5E-7	<4.5E-7	<5.2E-7	<7.6E-7
⁹⁰ Sr	$\mu\text{Ci/mL}$	2.0E-3	9.5E-6	1.3E-4	2.3E-5	4.2E-5	6.8E-3
⁹⁹ Tc	$\mu\text{Ci/mL}$	<1.1E-5	<3.3E-5	<3.9E-5	<3.7E-5	<1.7E-5	2.8E-4
Si	$\mu\text{g/mL}$	nd	7	154	2.6	2.5	nd
Al	$\mu\text{g/mL}$	nd	nd	nd	0.18	0.24	nd
Ca	$\mu\text{g/mL}$	nd	nd	nd	0.30	0.25	nd
B	$\mu\text{g/mL}$	nd	3.5	nd	1.9	2.4	nd
K	$\mu\text{g/mL}$	nd	nd	165	nd	nd	nd
Na	$\mu\text{g/mL}$	44400	22	43300	3.5	3.3	43700
H ⁺	M	--	--	--	--	2.98	--
²⁴¹ Am	$\mu\text{Ci/mL}$	<4.5E-5	--	--	--	--	--
^{239/240} Pu	$\mu\text{Ci/mL}$	<3.6E-6	--	--	--	--	--

Table A.19. Correspondence Between Actual Waste Loading Sample Names

<u>Solution Name</u>	<u>Sample Number</u>	<u>Analytical Serial #^(a)</u>	<u>Sample Number</u>	<u>Analytical Serial #</u>
Feed 1	1SYFEED	1J2515	3SYFEED1	J2566
Feed 2	1SYFEED2	J2516	3SYFEED2	J2567
Load 1	1SYLOD01	J2491	3SYLOD01	J2552
Load 2	1SYLOD02	J2492	3SYLOD02	J2553
Load 3	1SYLOD03	J2493	3SYLOD03	J2554
Load 4	1SYLOD04	J2494	3SYLOD04	J2555
Load 5	1SYLOD05	J2495	3SYLOD05	J2556
Load 6	1SYLOD06	J2496	3SYLOD06	J2557
Load 7	1SYLOD07	J2497	3SYLOD07	J2559
Load 8	1SYLOD08	J2498	3SYLOD08	J2560
Load 9	1SYLOD09	J2500	3SYLOD09	J2561
Load 10	1SYLOD10	J2501	3SYLOD10	J2562
Load 11	1SYLOD11	J2502	3SYLOD11	J2563
Load 12	1SYLOD12	J2503	3SYLOD12	J2564
Load 13	1SYLOD13	J2504	--	--
Load 14	1SYLOD14	J2505	--	--
Load 15	1SYLOD15	J2506	--	--
Load 16	1SYLOD16	J2507	--	--
Load 17	1SYLOD17	J2508	--	--
Load 18	1SYLOD18	J2510	--	--
Load 19	1SYLOD19	J2511	--	--
WASH 1	1SYWASH1	J2527	3SYWASH1	J2571
WASH 2	1SYWASH2	J2528	3SYWASH2	J2572
RINSE 1	1SYRINS1	J2529	3SYRINS1	J2573
RINSE 2	1SYRINS2	J2530	3SYRINS2	J2574

(a) Analytical results for all WHC 222-S samples are archived on the Laboratory Customer Communication System Computer. Samples are tracked on LCCS by their serial numbers, which are different from the customer identification (ID) numbers.

Table A.20. Correspondence Between Actual Waste Elution Sample Names

<u>Solution Name^(a)</u>	<u>Sample Number</u>	<u>Analytical Serial #</u>	<u>Sample Number</u>	<u>Analytical Serial #</u>
ELUTE 1	1SYELU01	J2531	3SYELU01	J2575
ELUTE 2	1SYELU02	J2532	3SYELU02	J2576
ELUTE 3	1SYELU03	J2533	3SYELU03	J2578
ELUTE 4	1SYELU04	J2534	3SYELU04	J2579 ^(b)
ELUTE 5	1SYELU05	J2535	3SYELU05	J2580 ^(c)
ELUTE 5			3SYELU05A	J2604 ^(d)
ELUTE 6	1SYELU06	J2536	3SYELU06	J2581 ^(e)
ELUTE 6			3SYELU6A	J2605 ^(f)
ELUTE 7	1SYELU07	J2537	--	--
ELUTE 8	1SYELU08	J2538	3SYELU08	J2582
ELUTE 9	1SYELU09	J2539	--	--
ELUTE 10	1SYELU10	J2540	3SYELU10	J2583
ELUTE 11	1SYELU11	J2541	--	--
ELUTE 12	1SYELU12	J2542	3SYELU12	J2585
ELUTE 13	1SYELU13	J2543	--	--
ELUTE 14	1SYELU14	J2544	--	--
STRIP	1SYSTRP1	J2545	--	--
CLEAN	1SYCLEAN	J2546	3SYCLEAN	J2587
FINAL	1SYFINAL	J2547	3SYFINAL	J2588
BLANK 1	1SYBLK01	J2490	3SYBLK01	J2593
BLANK 2	1SYBLK02	J2524	--	--
BLANK 3	1SYBLK03	J2525	--	--
BLANK 4	1SYBLK04	J2526	--	--
BLANK 5	1SYBLK05	J2519	--	--
RESIN	RES101	J2607	RES103	J2606

(a) Descriptive sample name used in this report.

(b) Diluted sample; analytical results must be multiplied by appropriate dilution factor.

(c) Diluted sample; analytical results must be multiplied by appropriate dilution factor.

(d) Corresponding undiluted sample submitted independently.

(e) Diluted sample; analytical results must be multiplied by appropriate dilution factor.

(f) Corresponding undiluted sample submitted independently.

Table A.21. WHC Analytical Results for Tank 101-SY and 103-SY Actual Waste Samples

Analyte	Units	Tank 101-SY		Tank 103-SY	
		Sample	Duplicate	Sample	Duplicate
% H ₂ O	wt %	73.6	73.7	72.5	72.6
Na	μg/mL	119000	123000	144000	133000
Na	M	5.2	5.3	6.3	5.8
Si	μg/mL	133	130	88	74
Al	μg/mL	15400	14200	17700	15900
Cu	μg/mL	5.2	4.0	nd ^(a)	nd
Ni	μg/mL	66	62	21	17
Ca	μg/mL	61	53	nd	nd
Cr	μg/mL	50	49	46	40
P	μg/mL	1200	1140	1620	1430
S	μg/mL	738	694	986	671
Mo	μg/mL	45	41	58	50
B	μg/mL	32	29	42	37
K	μg/mL	1440	1340	1660	1550
Cl ⁻	μg/mL	3510	3500	4140	4120
NO ₂ ⁻	μg/mL	43500	42800	48800	49000
NO ₃ ⁻	μg/mL	57400	54200	69900	70200
PO ₄ ³⁻	μg/mL	2330	3010	3400	3390
SO ₄ ²⁻	μg/mL	1760	1670	2210	2140
Oxalate	μg/mL	540	636	< 1100	< 1100
TOC	g/L	4.27	4.27	3.05	2.62
TIC	g/L	2.69	2.83	2.99	2.86
¹³⁷ Cs	μCi/mL	138	142	161	161
AT	μCi/mL	< 0.0010	< 0.0005	.00091	.00076
⁹⁰ Sr	μCi/mL	1.82	1.84	.82	.84
⁹⁹ Tc	μCi/mL	0.0271	0.0340	.072	.079
²⁴¹ Am	μCi/mL	< 2.9E-4	< 2.7E-4	6.3E-4	6.0E-4
^{239/240} Pu	μCi/mL	4.1E-5	3.6E-5	9.7E-5	< 3.9E-5

(a) Not detected.

Table A.22. PNL Analytical Results for Tank 101-SY and 103-SY Actual Waste Samples

Analyte	Units	Tank 101-SY		Tank 103-SY	
		Sample	Duplicate	Sample	Duplicate
Al	M	4.61E-01	4.63E-01	4.50E-01	na ^(a)
B	M	8.87E-03	7.66E-03	8.58E-03	na
Ca	M	1.25E-03	1.31E-03	8.11E-04	na
Cd	M	5.78E-06	5.69E-06	3.56E-06	na
Co	M	1.04E-05	1.07E-05	7.64E-06	na
Cr	M	8.78E-04	9.49E-04	6.97E-04	na
Cs	M	4.66E-05	4.82E-05	5.40E-05	na
Cu	M	6.67E-05	6.35E-05	2.98E-05	na
Fe	M	2.65E-04	4.94E-04	1.40E-04	na
K	M	2.99E-02	3.01E-02	3.11E-02	na
Mn	M	3.82E-06	4.19E-06	4.00E-06	na
Mo	M	3.72E-04	3.75E-04	4.09E-04	na
Na	M	4.75E+00	4.92E+00	4.86E+00	na
Ni	M	9.92E-04	1.00E-03	2.57E-04	na
P	M	3.30E-02	3.31E-02	3.64E-02	na
Pb	M	1.33E-05	1.34E-05	9.94E-06	na
Rb	M	3.28E-05	3.28E-05	3.51E-05	na
Sb	M	4.63E-05	4.70E-05	4.54E-05	na
Si	M	1.05E-02	9.74E-03	7.52E-03	na
Sr	M	2.02E-05	2.08E-06	nd ^(b)	na
Tl	M	1.07E-04	1.06E-04	1.04E-04	na
W	M	2.66E-04	2.69E-04	3.22E-04	na
Zn	M	8.38E-05	8.40E-05	1.24E-05	na
Zr	M	7.34E-06	7.24E-06	7.13E-06	na
Na/Cs	M/M	1.02E+05	1.02E+05	9.00E+04	na
Na/K	M/M	1.59E+02	1.63E+02	1.56E+02	na
¹³⁷ Cs	% Isotopic	2.35E+01	2.26E+01	2.28E+01	na
⁸⁷ Rb	% Isotopic	6.50E+01	2.90E+01 ^(c)	6.81E+01	na
⁹⁰ Sr	% Isotopic	1.50E+00	2.96E+00	3.03E+00	na

(a) Duplicate analyses not completed.

(b) Not detected.

(c) The determined Rb isotopic ratios are close to those expected for natural Rb, suggesting contamination of the analytical sample.

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