

## Intermediate-Scale Ion Exchange Removal of Cesium and Technetium from Hanford Tank 241-AN-102

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Tank 241-AN-102

**Intermediate-Scale Ion Exchange Removal of Cesium and Technetium from  
Hanford Tank 241-AN-102**

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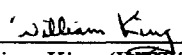


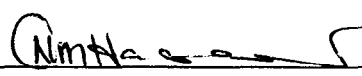
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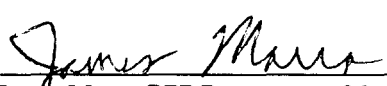
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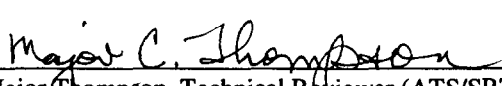
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## SUMMARY

Ion exchange tests have been completed at the Savannah River Technology Center for British Nuclear Fuels Limited, Inc. as part of the Hanford River Protection Project. Radioactive cesium and technetium (pertechnetate form only) were removed by ion exchange from a sample of Envelope C salt solution from Hanford Tank 241-AN-102 (sample volume: ~18 L at 4.8 M Na<sup>+</sup>). The original sample was diluted and subjected to strontium/transuranics (Sr/TRU) precipitation and filtration processes before ion exchange processing was performed. Batch contact and column tests for the ion exchange removal of cesium and technetium were then completed on the Sr/TRU-decontaminated product. Previous ion exchange tests were conducted on a smaller portion (0.5 L) of the Tank 241-AN-102 supernate sample, which had been similarly pretreated, and the results were reported in a separate document.

Poor cesium removal was observed for the large Envelope C sample relative to the small sample. The cesium distribution coefficient ( $K_d$ ) measured by batch contact for the large Envelope C sample of 298.4 mL/g was 39% of the  $K_d$  measured for the small sample ( $K_d$  = 758.9 mL/g). Ion exchange columns containing two 107 mL samples of SuperLig<sup>®</sup> 644 ion exchange resin (lead/guard column configuration; column ID: 2.7 cm) were used for Cs removal on the large sample. 50% Cs breakthrough was observed from the lead column after 130 bed volumes (BV) of solution had been processed (total volume processed: 170 BV). This performance meets the plant design target for column regeneration, which requires a minimum of 100 BV of feed to be processed prior to lead column regeneration. However, the cesium breakthrough profile was linear, which resulted in high cesium concentrations in the effluent collected from the guard column. Evidence suggests that insoluble solids had precipitated after the Sr/TRU filtration step, and these solids blinded the cesium ion exchange resin. Better results were observed for the small-scale (6.2 mL) cesium column test on the 0.5 L sample, in which 11% Cs breakthrough was observed from the lead column after processing 85 bed volumes of solution. For comparison, 11% Cs breakthrough was observed from the lead column in the intermediate-scale (107 mL) column test after ~40 BV of feed had been processed.

In order to meet the plant design criteria for the guard column effluent of 0.1748  $\mu\text{Ci/mL}$  Cs-137/Na [M], it was necessary to reprocess ~75% of the sample through the cesium ion exchange columns following regeneration of both columns. After reprocessing, the composited effluent solution from the two cesium column experiments easily satisfied the plant design requirements (estimated composition: 1.5E-2  $\mu\text{Ci/mL}$  Cs-137/mol Na). The data indicate that the routine elution step is sufficient to remove the insoluble solids and no permanent damage was done to the resin. Further work is needed to better understand the poor performance of SuperLig<sup>®</sup> 644 resin in the intermediate-scale test and the differences between the performance with the two samples tested.

As expected, only ~30% of the technetium was extractable in ion exchange column tests using SuperLig<sup>®</sup> 639 resin on the Cs-decontaminated Envelope C sample. Poor Tc removal had been previously observed with a small subsample of Envelope C, and was



attributed to the large fraction of nonpertechnetate forms of Tc in Envelope C waste. Ion exchange columns containing two 147 mL samples of SuperLig<sup>®</sup> 639 ion exchange resin (lead/guard column configuration; column ID: 2.7 cm) were used for pertechnetate ( $\text{TcO}_4^-$ ) removal on the large Envelope C sample. Technetium breakthrough from both the lead and guard columns stabilized at ~70% after 20-30 BV of feed had been processed and remained at this level throughout the remainder of the experiment (total volume processed: 114 BV). These results reveal that the lead column removed virtually all of the pertechnetate. This result is better than predicted from the equilibrium capacity measurements. Following the completion of the ion exchange column experiments, the decontaminated product and the concentrated eluates were provided for demonstration of the low- and high-activity vitrification processes, respectively. Evaporation and vitrification of these solutions will be reported separately.

Distribution coefficient ( $K_d$ ) values were also determined for the SuperLig<sup>®</sup> 644 and 639 resins over a range of cesium and technetium equilibrium concentrations, in order to generate equilibrium isotherms for each ion exchange resin. Simulant and radioactive ion exchange experiments conducted with the SuperLig<sup>®</sup> resins have been used to develop ion exchange models at the Savannah River Technology Center for the prediction of column performance in the actual plant operation. Batch contact and column data for the Tank 241-AN-102 sample will be used to further strengthen the model to predict full-scale column performance with Envelope C supernate. Results of the SuperLig<sup>®</sup> column modeling will be reported separately.

## 1.0 INTRODUCTION

Hanford radioactive waste materials have been categorized into four envelopes labeled A through D as specified in the Tank Waste Remediation Contract between British Nuclear Fuels Limited, Inc. (BNFL) and the United States Department of Energy (DOE).<sup>1</sup>

Envelopes A, B and C contain only solubilized species with less than 2 wt. % insoluble solids and are specified as Low-Activity Waste (LAW). Each envelope is defined based on compositional maximums of chemical and radioactive constituents. Envelopes A and B contain low concentrations of organic species and the primary form of technetium is pertechnetate ( $\text{TcO}_4^-$ ). Envelope C contains higher levels of organic species and technetium, which is primarily in the nonpertechnetate form. Envelope D is sludge which has been separated from the supernate and is referred to as High Level Waste.

The pretreatment process for the Hanford River Protection Project contracted to BNFL is to provide decontaminated low activity waste and concentrated eluate streams for vitrification into low- and high-activity waste glasses, respectively. The pretreatment includes precipitation and filtration to remove strontium, transuranics, and entrained solids and ion exchange processes to remove cesium and technetium. The cesium and technetium ion exchange removal is accomplished using SuperLig<sup>®</sup> 644 and 639 resins, respectively, from IBC Advanced Technologies. The resins have been shown to selectively remove cesium and anionic pertechnetate ( $\text{TcO}_4^-$ ) from alkaline salt solutions. The efficiency of ion exchange column loading and elution is a complex function involving feed compositions, the equilibrium and kinetic behavior of the ion exchange process, diffusion, and the ionic strength and pH of the aqueous solution. A previous experimental program completed at the Savannah River Technology Center (SRTC) demonstrated the conceptualized BNFL flow sheet parameters with an Envelope C sample from Hanford Tank 241-AN-107. Those experiments included determination of Cs and Tc batch distribution coefficients with SuperLig<sup>®</sup> 644 and 639 resins, respectively,<sup>2</sup> and demonstration of small-scale column breakthrough and elution<sup>3</sup>. The experimental findings were used in support of the preliminary design basis and pretreatment flow sheet developed by BNFL, Inc.

An earlier report described ion exchange testing conducted on a small portion (0.5 L) of the Tank 241-AN-102 sample received by SRTC.<sup>4</sup> This report describes ion exchange testing conducted on the remaining larger portion (~18 L) of the Tank 241-AN-102 sample. The objectives of this study were as follows:

- (1) Determine the batch distribution coefficients ( $K_d$  values) and percent removal for cesium (Cs) and technetium (Tc) ions on SuperLig<sup>®</sup> 644 and 639 ion exchange resins, respectively, with Envelope C salt solution from Hanford Tank 241-AN-102
- (2) Provide equilibrium isotherm data across a range of Cs and Tc concentrations to determine the parameters for successful modeling of ion exchange column breakthrough and elution

- (3) Demonstrate Cs and Tc column loading and elution profiles for Envelope C using SuperLig<sup>®</sup> 644 and 639 ion exchange resins
- (4) Provide decontaminated product samples for vitrification into low-activity waste glass and concentrated eluate samples for vitrification into high-activity waste glass
- (5) Provide information on the composition of column regenerate solutions and caustic displacement and water wash solutions used between loading and elution cycles

Batch contact experiments were performed on the large portion of the Envelope C sample to determine the equilibrium distribution coefficients ( $K_d$  values) and percent removal for cesium and technetium ions. The  $K_d$  values represent a measure of ion equilibrium distributions between the aqueous phase and the ion exchanger at a specific solid to liquid ratio. Also the  $K_d$  values provide valuable information on selectivity, capacity, and affinity of an ion exchange material to remove ions from complex aqueous solutions. Specific tests are performed by the addition of a small quantity of ion exchange material into a small volume of salt solution containing a known exchangeable ion(s). Factors that effect the  $K_d$  values and percent removal of the ion(s) include the temperature, initial concentration of the ions in solution, and the solid/liquid ratio during the contact. Duration of the contact must be sufficient to achieve equilibrium or a constant comparative value must be used. The loading capacity for the ion exchange columns is determined from batch contact experiments conducted under the same conditions as the columns. The  $K_d$  values are also determined at different equilibrium concentrations to establish a robust ion exchange decontamination process across the broad range of cesium and technetium concentrations that will be encountered as the solution passes through the columns. These data will be used as input to a computer model to determine scale-up parameters. The computer modeling work will be reported separately.<sup>5,6</sup>

The intermediate-scale ion exchange column experiments were performed in the Shielded High Level Cells at SRTC, allowing remote handling of materials. All experiments were conducted at ambient temperature. A pair of columns was used for the cesium column experiments and a second pair was used for the technetium column experiments. Each pair consisted of two columns connected in series, one as the lead column and the other as the guard column. The cesium columns were each packed with 107 mL of SuperLig<sup>®</sup> 644 resin and the technetium columns each contained 147 mL of SuperLig<sup>®</sup> 639 resin. In each experiment, the bulk feed sample was allowed to pass through the lead column and was collected from the guard column in fractions. The lead and guard column effluents were periodically sampled during column loading to detect early breakthrough and monitor column performance. Once the analytical results were received and the Cs- and Tc-decontaminated effluent was confirmed to meet the LAW acceptance criteria, the fractions were combined and further characterized. Following column loading, the feed solution was displaced from the columns and the lead and guard columns were eluted separately with 0.5 M HNO<sub>3</sub> (Cs) or water (Tc). The lead column eluates were periodically sampled to determine when elution was complete. The portions of the lead column eluates containing the bulk of the radioactivity were later combined to give

separate Cs and Tc eluate composite samples which were analyzed and provided for evaporation and vitrification into high-activity waste glass (reported separately<sup>7</sup>).

This revised report was generated from the original report describing this work (WSRC-TR-2000-00420, Rev. 0) by incorporating the following modifications to correct errors identified after the original report was issued.

- 1) In the Summary and the Introduction it was stated that the volume of the sample received for ion exchange testing was approximately 17 L. The volume has been changed to ~18 L.
- 2) The calculated values reported for the amount of Cs-137 ( $\mu\text{Ci/mL}/[\text{Na}]$  and total) in the composited guard column effluent and the overall Cs-137 decontamination factor (DF) and % removal were incorrect. The corrected values for this data were input in the first paragraph of Section 3.2.
- 3) In Attachment 4, the original units for the ICP-MS analysis (mg/L) were incorrect and have been changed to  $\mu\text{g/L}$ . In addition, one of the reported values for arsenic (0.235 mg/L) in this attachment was incorrect and has been changed to 2.35 mg/L.

## 2.0 EXPERIMENTAL

### 2.1 Materials

Some physical data measured for the SuperLig<sup>®</sup> 644 and 639 resins which was reported in an earlier document is provided in Table 1.<sup>8</sup> The SuperLig<sup>®</sup> 644 resin was composed of dark brownish-red granules. The SuperLig<sup>®</sup> 639 resin was composed of tan, spherical beads. Batch contact and column tests for Cs removal were conducted with SuperLig<sup>®</sup> 644 batch #981020MB48-563. Tc distribution coefficients were measured for SuperLig<sup>®</sup> 639 batch #981015DHC720011. Tc column experiments were conducted using a 50:50 mixture (by as-received weight) of SuperLig<sup>®</sup> 639 batches 981015DHC720011 and 990420DHC720067. The same mixture of SuperLig<sup>®</sup> 639 batches was used for simulant intermediate-<sup>16</sup> and pilot-scale<sup>17</sup> column tests at SRTC and for radioactive testing with the SRS Tank 44 F Supernate Sample.<sup>18</sup>

Table 1. Physical Characteristics of “As-Received” SuperLig<sup>®</sup> Resins.<sup>8</sup>

SuperLig <sup>®</sup> Resin	Batch #	Particle Density (g/mL)	Bulk Dry Density (g/mL)	As-received Water Content (%)
644	981020MB48-563	1.549	0.758	9.6
639	981015DHC720011	1.147	0.468	1.3
639	990420DHC720067	-----	0.479	-----

Prior to the cesium and technetium ion exchange tests, the large portion of the “as-received” Tank 241-AN-102 sample was diluted to 6.5 M Na<sup>+</sup>, characterized, and treated for Sr/TRU removal by the addition of sodium hydroxide, strontium nitrate and sodium permanganate solutions. The product was then filtered through a cross-flow filter to remove Sr/TRU precipitates. Filter wash solutions were combined with the filtrate just prior to the initiation of the ion exchange experiments. Other reports describe the dilution and characterization<sup>9,10</sup> and the Sr/TRU removal<sup>10</sup> performed for the Tank 241-AN-102 sample. The Sr/TRU-decontaminated and filtered Tank 241-AN-102 sample composition is given in Attachment 1. The primary waste composition based on analysis is: 4.8 M Na<sup>+</sup>, 1.3 M NO<sub>3</sub><sup>-</sup>, 0.66 M NO<sub>2</sub><sup>-</sup>, 1.3 M Free OH<sup>-</sup>, ~1 M CO<sub>3</sub><sup>2-</sup>, and 0.21 M AlO<sub>2</sub><sup>-</sup>. The [Cs] and [Tc] were 172 µCi/mL and 4.31 mg/L, respectively. (The Cs and Tc concentrations are average values calculated from analysis results for the column experiments and the batch contact tests.) The [Na<sup>+</sup>] of the sample decreased from 6.5 to 4.8 M after treatment for Sr/TRU removal. The measured density of the solution was 1.23-1.24 g/mL. 1.0 and 0.25 M NaOH solutions were used for column pretreatment and 0.1 M NaOH solution was used for column washing. 0.5 M HNO<sub>3</sub> was used for SuperLig<sup>®</sup> 644 column elution. These solutions were prepared using ACS certified, high-purity reagents from Fisher Scientific, Inc.

## 2.2 Equipment and Procedures

### 2.2.1 Batch Contact Experiments

Duplicate batch contact tests were performed at ambient temperature using an orbital shaker in the Intermediate Level Cells Facility at SRTC. “As-received” ion exchange resin (0.08-0.18 g) and Tank 241-AN-102 solution (8-18 mL) were transferred to a 20 mL polyethylene vial and placed on the shaker. The ratio of solution volume to exchanger mass was 100±10. The temperature was measured at the beginning and the end of the experiment (observed range: 25-27 °C). After 24±1 hours, the samples were removed from the shaker. The solution was then filtered through a 0.45 micron Nalgene<sup>®</sup> nylon filter using vacuum. Control samples (~18 mL of salt solution) were treated in the same way as those of duplicate test samples without the addition of the resin. The concentrations of Cs and Tc in control samples were used as the starting initial concentrations to determine the K<sub>d</sub> values and the percentage of analyte removed by the resin. A standard batch contact test was conducted using the original ion exchange column feed solution, followed by two sequential recontacts of the filtrates with fresh resin. In addition, independent cesium- and technetium-spiked batch contact tests were conducted. At the conclusion of each test, a 1 mL sub-sample of the filtrate was removed from the cell and analyzed for cesium (gamma scan) or technetium (ICP-MS) by the Analytical Development Section (ADS) at SRTC. Sub-samples were often diluted (10:1) with de-ionized water to reduce the radiation dose rate before the samples were transferred from the cell to the analytical laboratories. All dilutions and measurements were performed based on mass, and corrections were applied for the density of the solution to ensure accuracy. Data for the batch contact tests are provided in Attachments

2 and 5. The Cs and Tc distribution coefficients ( $K_d$ ) and % removal were calculated using the formulas shown in Eqs. 1 and 2, respectively.

$$K_d = [(C_i/C_f) - 1][V/(M \cdot F)] \quad (1)$$

$$\% \text{ removal} = \frac{(100) \cdot (C_i - C_f)}{C_i} \quad (2)$$

$C_i$  = initial [Cs] or [Tc] in feed ( $\mu\text{Ci/mL}$  or  $\text{mg/L}$ )

$C_f$  = final [Cs] or [Tc] after contact ( $\mu\text{Ci/mL}$  or  $\text{mg/L}$ )

$V$  = volume of solution used ( $\text{mL}$ )

$M$  = mass of “as-received” resin ( $\text{g}$ )

$F$  = resin dry weight correction factor

F-factors of 0.904 and 0.987 were used to calculate the Cs and Tc  $K_d$  values, respectively, based on the data reported for the resin batches in Table 1.

Following the sequential batch contact tests, small quantities of cesium nitrate (non-radioactive) or sodium pertechnetate (in  $\sim 1 \text{ M HNO}_3$ ) were spiked into a known volume of the original ion exchange column feed solution. This was done to increase the initial concentration of Cs or Tc in test solutions (total [Cs] increased to  $>30$  times feed; [Tc] was increased by  $\sim 5$ ). Because of the size of the spike sample and the density difference of the spike and the test solution, it was necessary to add the test solution into a polyethylene bottle containing the spike. The spiked solution was then shaken manually in the cell to provide adequate mixing before contacting the solution with the resin. The spiked test solution ( $\sim 10 \text{ mL}$ ) was gently shaken with fresh ion exchange resin ( $\sim 0.1 \text{ g}$ ) for  $24 \pm 1$  hours. A control spike sample was treated in the same manner. After the contact period, the solution was separated from the resin by filtration under vacuum using a  $0.45 \mu\text{m}$  Nalgene<sup>®</sup> nylon filter. A  $1 \text{ mL}$  sub-sample of the filtrate was removed from the shielded cells and analyzed for cesium (gamma) or technetium (ICP-MS). Data for the Cs and Tc batch contact tests are provided in Attachments 2 and 5, respectively.

### 2.2.2 Column Experiments

Figure 1 shows the ion exchange column design. A photograph of the actual ion exchange column apparatus used for Cs removal is shown in Figure 2. A pair of ion exchange columns was used which were constructed from  $2.7 \text{ cm ID}$  sodium borosilicate glass tubing. Decals were affixed to the outer walls of the columns with  $1 \text{ mm}$  graduations to measure the resin bed height. The outsides of the columns were coated with a layer of polyvinylchloride to reduce hazards associated with potentially pressurizing the apparatus. 3-way, Whitey<sup>®</sup> 40 Series stainless steel ball valves (#1 and #7) were attached to the bottoms of the columns. The column heads were attached to the columns using Rudivis<sup>®</sup> ground-glass joints. Stainless steel ball valves (#2 and 4; #8 and

10) were attached on opposite sides of each column head to serve as feed ports. Each column head also contained a vent (valves: #3 and #9), a pressure gauge, a pressure relief valve, and a fill reservoir. The valves were connected to the columns by high-density polyethylene bushings with Viton® O-rings. Swage-lock® fittings were used to connect the valves to low-density polyethylene tubing (11/64" ID) which served as column feed and effluent lines. A 3-way valve (#6) was used to collect samples of the lead column effluent. Prior to each sample collection from valve #6, the tubing was purged to remove liquid retained from previous sample collections. Stainless steel wire screens (200 mesh) were inserted into the columns to support the ion exchange resin. All solutions were passed through the columns in the downflow direction using Fluid Metering Incorporated QG150 positive displacement pumps with 1/4" and 3/8" piston sizes. Guard column samples were collected at the end of the guard column effluent collection line, rather than with the manual sample port located at the bottom of the column. The manual sample ports were used to drain the liquid head from the columns at specific times during column operation as specified in the technical task plan.<sup>11</sup>

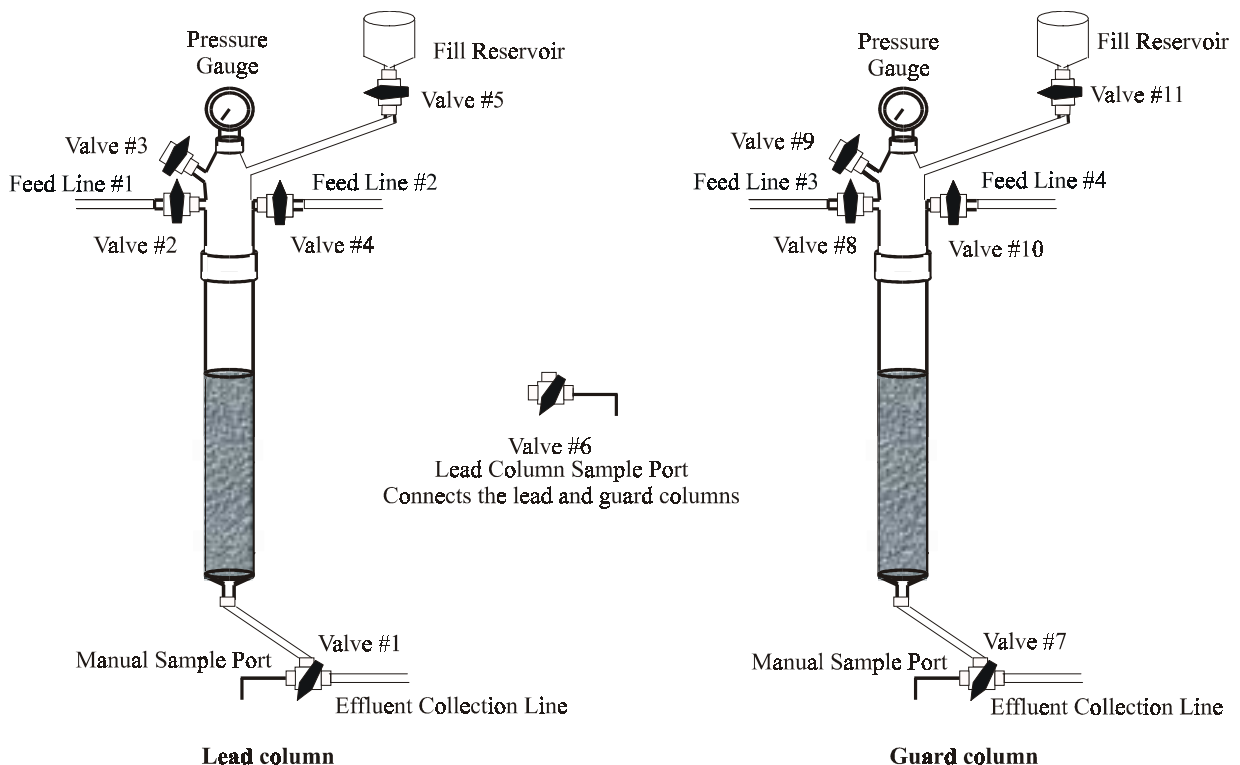


Figure 1. Ion Exchange Column Design



Figure 2. Tank 241-AN-102 Cesium Ion Exchange Column Apparatus (Note: The photograph was taken just prior to initiating the column experiments with the Envelope B sample.)



For the cesium ion exchange column experiment, samples of SuperLig<sup>®</sup> 644 resin from batch #981020MB48-563 were used for the lead and guard columns. The columns used for the Envelope C cesium removal experiment were the same columns used for the Cs ion exchange tests on the Hanford Tank 241-AZ-102 (Envelope B) sample. For the Envelope B Cs ion exchange test, the lead and guard columns each contained ~30 mL of SuperLig<sup>®</sup> 644 resin. Results of this experiment are reported in a separate document.<sup>12</sup> Both the lead and guard columns from the Envelope B Cs ion exchange tests were fully eluted with 0.5 M HNO<sub>3</sub> and washed with deionized water. Additional SuperLig<sup>®</sup> 644 resin was prepared by soaking a portion of the “as-received” sample in 1 M NaOH overnight. The tops to both ion exchange columns were removed remotely in the shielded cell and additional SuperLig<sup>®</sup> 644 resin was added as a suspension. For the technetium ion exchange column experiment, a different, but identical set of ion exchange columns (lead/guard configuration) was used containing a 50:50 mixture (by as-received weight) of SuperLig<sup>®</sup> 639 batches 981015DHC720011 and 990420DHC720067. The resin bed volume was monitored throughout the Cs and Tc column experiments by periodically measuring the height of the bed. The resin bed volume was determined by multiplying the measured height of the resin bed by the column cross-sectional area. The resin volume in the pretreatment solution (SuperLig<sup>®</sup> 644: 0.25 M NaOH; SuperLig<sup>®</sup> 639: 1.0 M NaOH) determined the size of one bed volume (BV). Based on this criteria, the size of one BV for the cesium and technetium column experiments was 107 mL and 147 mL, respectively. The temperature during the column experiments ranged from 22-25 °C.

After transferring the SuperLig<sup>®</sup> 644 resin into the columns and replacing the tops, the resin bed was treated with the following solutions sequentially (Pretreatment Cycle): 3 BV of fresh deionized water, 6 BV of 0.25 M NaOH, 3 BV of deionized water, 3 BV of 0.5 M HNO<sub>3</sub>, and 3 BV of deionized water. All of these solutions were passed through the columns at a flow rate of 3 BV/hr. The bed was pretreated with 6 BV of 0.25 M NaOH over a 2-hour period just prior to beginning the experiment with Tank 241-AN-102 supernate. The 0.25 M NaOH liquid level in the columns was adjusted so that the volume of liquid above the resin bed was near 100 mL (~1 bed volume) before Tank-241-AN-102 solution was pumped into the column. This experimental arrangement for the test ion exchange column was selected to emulate the design of the full-scale columns in the BNFL Inc. waste treatment plant.<sup>13</sup> ~5 mL samples of effluent were collected from the lead column at ~10 bed volume intervals while processing the Tank 241-AN-102 sample (Feed Cycle). The flow rate during the feed cycle was 2.8 BV/hr (0.83 cm/min; measured range: 2.3-3.3 BV/hr). Following the feed cycle, the liquid in both columns was drained to within 1 cm of the top of the resin bed. Sodium hydroxide solution (0.1 M) was pumped into the lead column until the volume of liquid above the resin bed was approximately 100 mL. The lead column exit line (valve #1) was opened and the liquid level in the guard column was raised to the original height (~1 BV) by pumping 0.1 M NaOH solution into the system through the lead column. 3.5 bed volumes of 0.1 M NaOH were passed through the lead/guard column configuration at a flow rate of 3 BV/hr (NaOH Wash Cycle). The columns were then washed with 3.0 BV of deionized water at the same flow rate (Water Wash Cycle). The guard column effluent

during the NaOH and Water Wash Cycles was collected in ~1 BV fractions and samples were submitted for Cs-137, Na, and OH<sup>-</sup> analysis from each bottle. The lead column was then isolated from the guard column (by closing valve #3) and was eluted with 0.5 M HNO<sub>3</sub> at a flow rate of 1.1 BV/hr (0.33 cm/min; measured range: 0.6-1.4 BV/hr). The eluate collection bottles were changed approximately every 2 hours (Elution Cycle). Samples were collected for analysis from each effluent and eluate collection bottle. Loading and elution data from the SuperLig<sup>®</sup> 644 column experiment is provided in Attachment 3.

The SuperLig<sup>®</sup> 639 ion exchange columns were prepared by soaking the “as-received” resin overnight in deionized water and transferring the resin into the columns as a suspension. The bed was pretreated with 3 bed volumes of 1.0 M NaOH over a 1 hour period just prior to beginning the experiment (Pretreatment Cycle). The NaOH liquid level in both columns was adjusted so that the volume of liquid above the resin bed was near 150 mL (~1 bed volume) before Tank 241-AN-102 supernate was pumped into the columns. ~5 mL samples of effluent were collected from the lead column at ~5 bed volume intervals while processing the Tank 241-AN-102 sample (Feed Cycle). The flow rate during the feed cycle was 2.2 BV/hr (0.98 cm/min; range measured: 1.8-2.6 BV/hr). Samples were collected for analysis from each effluent collection bottle. Following the feed cycle, the liquid in both columns was drained to within 1 cm of the top of the resin bed. Sodium hydroxide solution (0.1 M) was pumped into the lead column until the volume of liquid above the resin bed was approximately 150 mL (~1 BV). The lead column exit line (valve #1) was opened and the liquid level in the guard column was raised to the original height (~1 BV) by pumping 0.1 M NaOH solution into the system through the lead column. The guard column exit line was opened and 2.3 bed volumes of 0.1 M NaOH were passed through the columns at a flow rate of 2.5 BV/hr (NaOH Wash Cycle). The lead column was then isolated from the guard column and eluted with deionized water at a flow rate of 0.78 BV/hr (0.35 cm/min; range measured: 0.4-0.9 BV/hr). The eluate collection bottles were changed and 3-5 mL grab samples of the eluate were collected approximately every 4 hours (Elution Cycle). Loading and elution data from the SuperLig<sup>®</sup> 639 column experiment is provided in Attachment 6.

The flow rate was monitored during the column experiments by periodically measuring the time required to collect 5-10 mL samples of effluent. The effluent solution during the feed cycle was collected in 1 L bottles. The weight of effluent solution in each bottle was measured and the volume was calculated using the solution density and corrected for the volume of samples collected. The flow rate was calculated from the time of collection for each bottle. The overall flow rate was taken to be the volume-weighted average of the flow rates calculated for each collection bottle. The flow rates for the elution cycles were calculated similarly. The number of bed volumes of solution processed for each sample was calculated from the time of collection and the flow rate. (**Note:** The column operating procedure was designed to emulate the actual plant operation, where the column is half full of resin and half full of liquid. According to the procedure, the initial simulant that was fed into the ion exchange column during the loading cycle was to be diluted by the 1 BV of NaOH pretreatment solution which remained above the resin bed.

Likewise, the post-feed water wash and the eluting solutions were allowed to mix with the liquid head left above the resin from the previous cycle. No attempt was made to correct for mixing of solutions in the column head-space when calculating the number of bed volumes of feed, wash, or eluate processed. Each processing cycle was considered to start at the moment that the next processing liquid entered the lead column.)

### 3.0 RESULTS AND DISCUSSION

#### 3.1 SuperLig<sup>®</sup> 644 Batch Contact Experiments

Cesium distribution coefficients and % removal measured for SuperLig<sup>®</sup> 644 with the small and large portions of the Tank 241-AN-102 sample are given in Table 2. A plot of the Cs-137  $K_d$  data versus final [Cs] is shown in Figure 3. Considerably higher  $K_d$  values were observed with the small portion of the Env. C sample than for the large Env.

Table 2. Cesium Distribution Coefficients ( $K_d$ ) and % Removal Measured for SuperLig<sup>®</sup> 644 (Batch #981020MB48-563) and the Tank 241-AN-102 Sample

##### Small Tank 241-AN-102 Sample<sup>4</sup>

Cs initial (μg/L)	Cs final (μg/L)	Cs $K_d$ (mL/g)	% Cs Removal
204860.0*	56247.5	301.5	73.0
Cs-137 initial (μCi/mL)	Cs-137 final (μCi/mL)	Cs-137 $K_d$ (mL/g)	% Cs-137 Removal
190.3	24.0	758.9	88.0
24.0**	1.6	1380.0	94.0
1.6**	0.1	1366.0	93.0

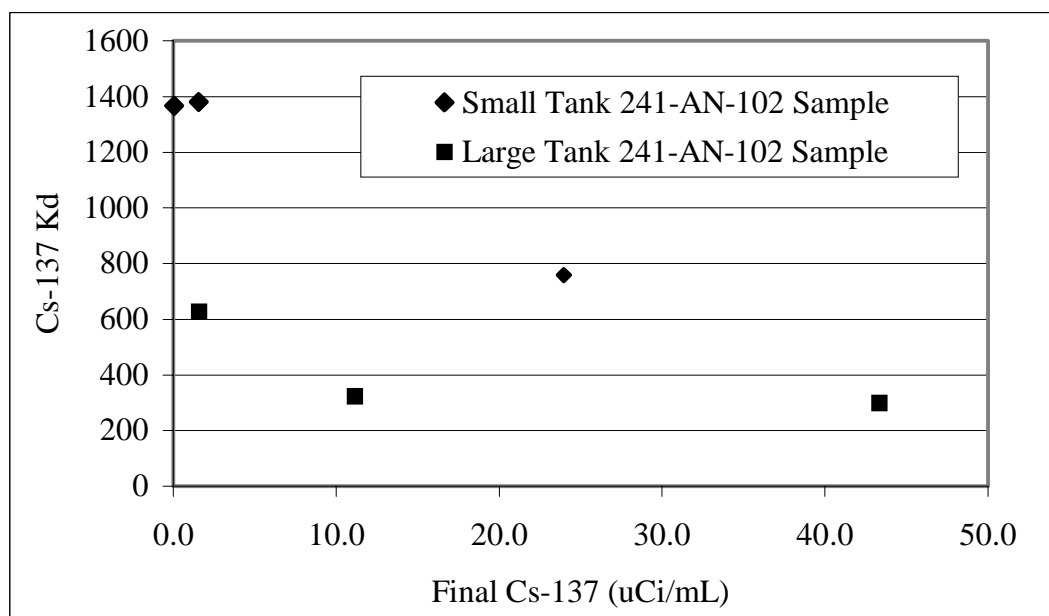
##### Large Tank 241-AN-102 Sample

Cs initial (μg/L)	Cs final (μg/L)	Cs $K_d$ (mL/g)	% Cs Removal
202522.0*	76980.0	175.0	62.0
Cs-137 initial (μCi/mL)	Cs-137 final (μCi/mL)	Cs-137 $K_d$ (mL/g)	% Cs-137 Removal
164.0	43.3	298.4	73.5
43.3**	11.2	323.3	74.1
11.2**	1.6	627.1	85.6

\*Indicates samples which had been spiked with non-radioactive cesium (Note: Total [Cs] was measured for the spiked sample rather than [Cs-137].)

\*\* Indicates recontact tests in which the feed solution was filtrate isolated from previous batch contacts

Figure 3. Cs-137 Distribution Coefficients ( $K_d$ ) versus Final [Cs] for the Tank 241-AN-102 Sample with SuperLig<sup>®</sup> 644 Resin (Batch #981020MB48-563)



C sample. At a final Cs-137 concentration of 1.6  $\mu\text{Ci/mL}$ , the measured  $K_d$  value for the large sample (627.1 mL/g) was 46% of the value observed for the small sample (1366.0 mL/g). This observation was surprising since the small and large portions of the Tank 241-AN-102 sample were treated similarly. The  $K_d$  values decreased considerably for both samples when the solution was spiked with Cs to over 200 mg/L. Based on the  $K_d$  data, cesium breakthrough was expected to occur earlier for the large portion of the Tank 241-AN-102 sample than was observed for the small sample. The theoretical 50% breakthrough point ( $\lambda$ ) for the large portion of the Env. C sample can be predicted from  $K_d$  data and the swollen bulk resin density (0.4 g/mL) using Eqn. 3. The  $K_d$  measured for the original column feed solution was ~300 mL/g (final [Cs-137]: 43  $\mu\text{Ci/mL}$ ; ~25% of the feed Cs-137 concentration).

$$\lambda = (K_d) \times (\text{bulk resin density}) = (300) \times (0.4) = 120 \quad (3)$$

Based on this simple calculation, 50% Cs breakthrough is expected after 120 BV of feed have been processed.

### 3.2 SuperLig<sup>®</sup> 644 Column Experiments

The cesium breakthrough profile observed for the lead SuperLig<sup>®</sup> 644 column during column loading with the large portion of the Envelope C sample is shown in Figure 4. 50% Cs breakthrough was observed for the lead column after ~130 BV of feed had been processed. The observed breakthrough was very near the value predicted based on the  $K_d$

data (120 BV). Previous SuperLig<sup>®</sup> 644 columns have exhibited earlier cesium breakthrough with Hanford waste samples than was predicted by the  $\lambda$  approximation. 50% Cs breakthrough was observed in column tests with SuperLig<sup>®</sup> 644 and Hanford Tank 241-AN-107 (Envelope C) at 37% of the  $\lambda$  value.<sup>3</sup> The observed breakthrough satisfies the current plant design column regeneration target which requires 100 BV of feed to be processed prior to lead column regeneration. However, as is shown in Figure 3, the Cs breakthrough profile for the lead column was nearly linear, which resulted in higher cesium concentrations in the guard column effluent than would be expected if the shape of the profile had been sigmoidal. The estimated Cs-137 concentration for the composited guard column effluent of 1.14  $\mu\text{Ci/mL}$  Cs-137/Na [M] far exceeded the plant design criteria for the effluent (0.1748  $\mu\text{Ci/mL}$  Cs-137/Na [M]). (Note: The Cs-137 concentration in the composited effluent was estimated from the Cs-137 activity and the volume of the effluent fractions which were collected in 1 L bottles. The guard column effluent fractions were never actually combined following the SuperLig<sup>®</sup> 644 column experiment.) Figure 5 shows the Cs-137 activity in each guard column effluent collection bottle. Interestingly, the Cs breakthrough profile for the guard column does not appear to be linear. The calculated total Cs-137 in the guard column effluent was 9.97E4  $\mu\text{Ci}$ . This corresponds to 96.8% Cs-137 removal from the Tank 241-AN-102 sample and a decontamination factor (DF) of 31.6 for the lead/guard column configuration (Total Cs-137 in feed: 3.15E6  $\mu\text{Ci}$ ).

The poor lead column performance with the Tank 241-AN-102 sample may be due to the presence of solids in the column feed solution. Some dark solids were observed in the 1 L bottles which contained the feed during processing of the sample through the SuperLig<sup>®</sup> 644 columns. The amount of visible solids was not large and efforts were undertaken to minimize the amount of solids that were actually transferred into the columns. Solids have also been observed in Tank 241-AN-107 Env. C simulant solutions after the completion of similar Sr/TRU precipitation and cross-flow filtration treatments.<sup>14</sup> Dark solids were also observed to form in the actual Tank 241-AN-102 filtrate several weeks after cross-flow filtration had been completed.<sup>15</sup> However, this filtrate sample had not been diluted with the NaOH filter wash solutions, as was the case with the actual ion exchange column feed solution. Analysis of the filtered solids after dissolution in 0.5 M  $\text{HNO}_3$  revealed that the general composition was consistent with that of the filtrate solution, except that the relative Mn concentration was high. This indicates that the solids were primarily Mn salts and the remaining components reported were the result of insufficient rinsing of the solids to remove residual filtrate solution. The fact that the guard column appeared to perform better than the lead column is consistent with either the removal of insoluble solids or the absorption of specific interfering ions by the lead column. Removal of interfering ions by the lead column could have resulted in decreased cesium removal by the lead column, but may have also yielded a guard column feed which was more suitable for cesium removal by SuperLig<sup>®</sup> 644 resin. Further work is needed to better understand the poor performance of SuperLig<sup>®</sup> 644 resin in the intermediate-scale column test and the differences between the resin performance with the small and large portions of the Tank 241-AN-102 sample.

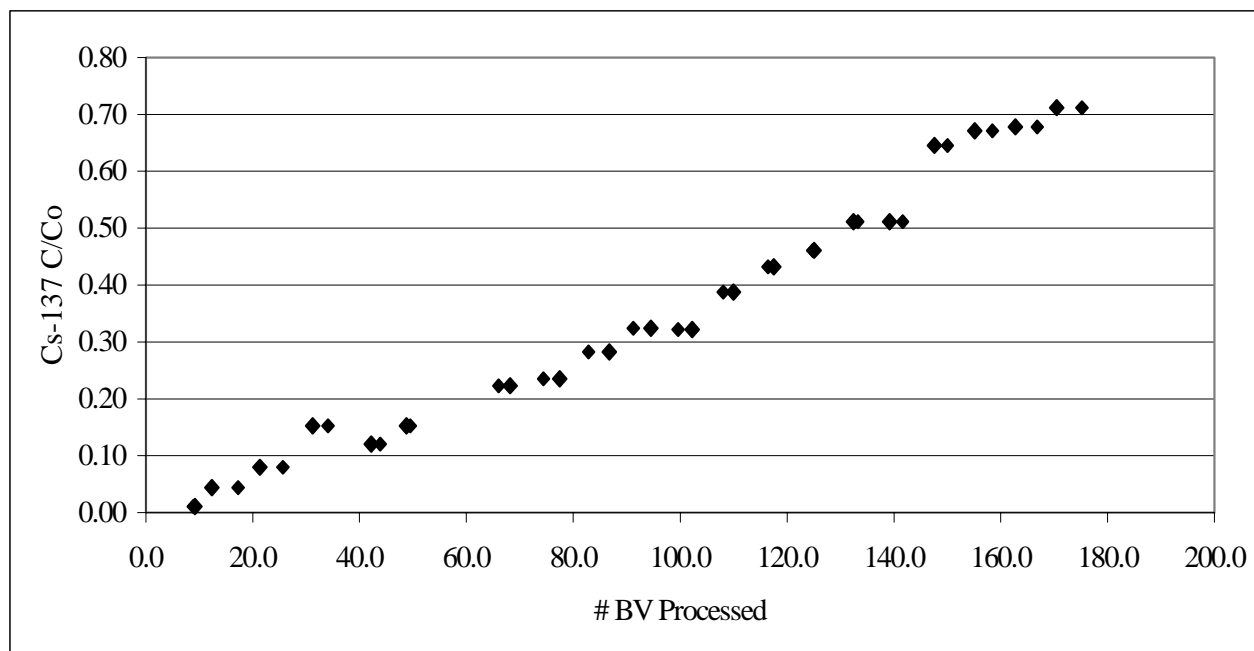


Figure 4. SuperLig® 644 Lead Column Cs-137 Breakthrough Profile with the Large Env. C Sample (Column ID: 2.7 cm, Flow Rate: 2.8 BV/hr, Resin Batch #981020MB48-563, Resin Bed Volume: 107 mL)

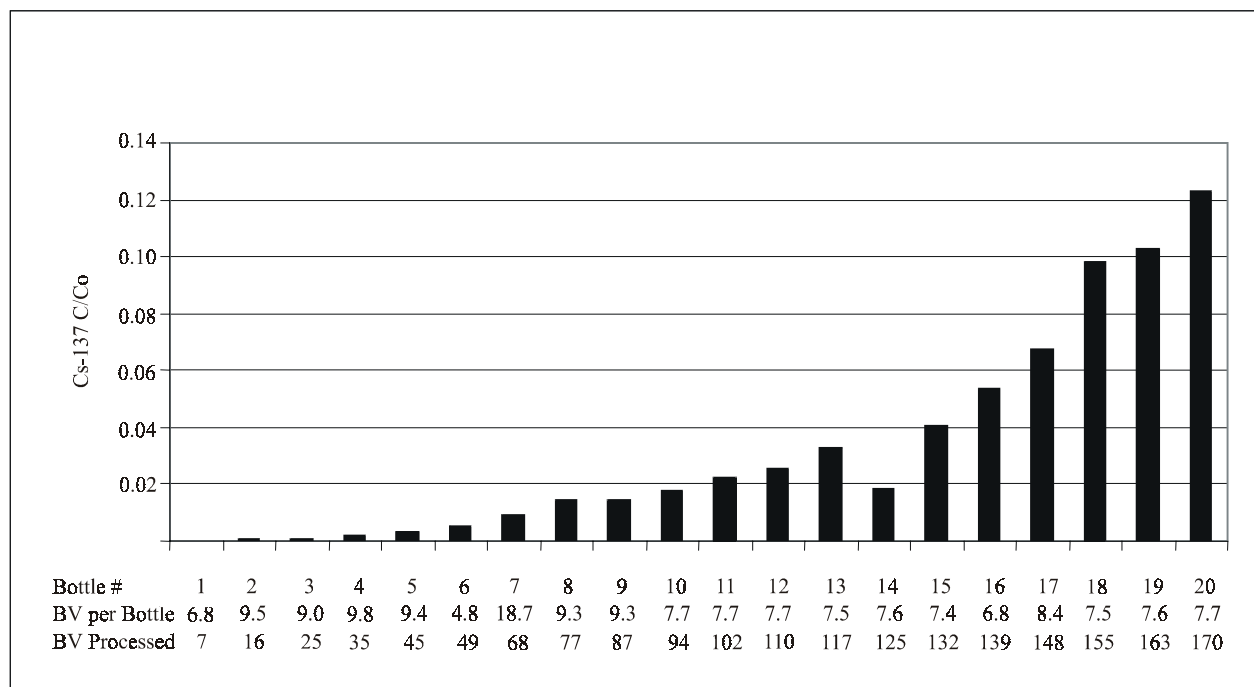


Figure 5. SuperLig® 644 Guard Column Effluent Fractions Cs-137 Breakthrough Profile with the Large Envelope C Sample (Column ID: 2.7 cm, Flow Rate: 2.8 BV/hr, Resin Batch #981020MB48-563, Resin Bed Volume: 107 mL)

During the NaOH and Water Wash Cycles, the effluent was collected from the guard column in ~1 BV fractions. Analysis results received for the wash fractions are shown in Table 3. The Cs-137 concentration in the wash collection bottles decreased during the wash cycle from 20.4  $\mu\text{Ci/mL}$  (Fraction #1) to 1.45  $\mu\text{Ci/mL}$  (Fraction #5), which is <1% of the feed [Cs-137]. The wash solutions contained a total of 5.36E3  $\mu\text{Ci}$  of Cs-137 (0.17% of the feed Cs-137).  $\text{Na}^+$  and  $\text{OH}^-$  concentrations decreased as expected in each successive wash fraction.

Following the wash cycle, the lead column was eluted with 0.5 M  $\text{HNO}_3$ . The eluate was collected in ~2 BV fractions and samples from each bottle were submitted for Cs-137 analysis. The lead column Cs-137 elution profile is shown in Figure 6. Analytical results for the lead column eluate fractions are provided in Attachment 3. The Cs-137 peak concentration was observed after 5.0-7.6 BV of eluate had been processed. The delay in the observed peak concentration in the eluate fractions was partially due to the liquid head atop the resin bed. The Cs-137 concentration reached <1% of the feed (1.79E3  $\mu\text{Ci/mL}$ ) after 10.9 BV (6 bottles) of eluate had been processed. A total of 22.5 BV of acid was passed through the lead column during the elution cycle. The first 6 bottles of eluate (total volume: 1,166 mL) were composited for further characterization and vitrification. Analysis results received for the composited eluate are provided in Attachment 4. The average Cs-137 concentration in the eluate was 1.81E3  $\mu\text{Ci/mL}$ , which corresponds to 2.11E6  $\mu\text{Ci}$  of Cs-137 (67.0% of the feed [Cs-137]). Integration of the breakthrough profile indicated that 2.16E6  $\mu\text{Ci}$  Cs-137 (68.4% of the feed Cs-137) was adsorbed by the lead column. Based on this data 97.9%, of the Cs-137 that was adsorbed on the lead column was recovered in the composited eluate. The remaining 6 bottles of eluate (bottles number 7 through 12) contained ~106.1  $\mu\text{Ci}$  of Cs-137, representing ~0.003% of the total Cs-137 in the feed. Relatively high concentrations of Ca, Cu, Cr, Ni, Pb and P were present in the cesium eluate. The P and Pb concentrations

Table 3. SuperLig® 644 Column Post-Feed 0.1 M NaOH Column Wash and Regenerate Solution Characterization Data

Sample	Cumulative BV	Cs-137 ( $\mu\text{Ci/mL}$ )	Cs-137 C/Co	Na (M)	Free $\text{OH}^-$ (M)
Fraction 1	1.5	2.04E+01	1.14E-01	3.60	1.015
Fraction 2	2.5	1.03E+01	5.74E-02	0.442	0.3055
Fraction 3	3.5	4.96E+00	2.77E-02	0.200	0.2275
Fraction 4	5.0	2.11E+00	1.18E-02	0.124	0.1723
Fraction 5	6.5	1.45E+00	8.08E-03	0.0971	0.1452
Regenerate	6.0	1.06E-01	5.94E-04	0.0700	0.1008

were within twice the minimum detection limits for these analytes (see footnote Attachment 4) and may not be real values. Comparably high concentrations of Ca, Cu, Cr, and Ni were observed in the cesium ion exchange eluate isolated during treatment of the small portion of the AN-102 sample.<sup>4</sup> Apparently, these species either absorb to specific resin sites or precipitate from the solution during ion exchange processing of Envelope C solution.

In order to sufficiently decontaminate the Envelope C sample to meet the vitrified LAW specifications, it was necessary to reprocess a portion of the Envelope C sample. The SuperLig<sup>®</sup> 644 guard column was fully eluted with 21 BV of 0.5 M HNO<sub>3</sub>. Analysis at the conclusion of the elution confirmed that the Cs-137 concentration was <1% of the feed concentration. The columns were then washed in series with deionized water and regenerated with 6 BV of 0.25 M NaOH (flow rate: 3 BV/hr). The water/NaOH regenerate solution was collected and analyzed for Cs-137, Na<sup>+</sup> and OH<sup>-</sup> (Table 3). The regenerate solution contained approximately 68 µCi of Cs-137 (total volume collected: ~640 mL) which corresponds to 0.002% of the feed [Cs-137]. The [Na<sup>+</sup>] and [OH<sup>-</sup>] reported for the regenerate were considerably lower than 0.25 M because at least half of the regenerate was water (much of the 0.25 M NaOH remained in the columns) and exchange with the resin also reduces the initial NaOH concentration of the wash solutions.

Effluent composite bottles 7-20 from the first cesium column experiment were reprocessed through the regenerated SuperLig<sup>®</sup> 644 columns in the order that they were collected during the first experiment. Less than 5% Cs breakthrough (based on the [Cs-137] in the original feed) was observed from the guard column during reprocessing. The estimated Cs-137 concentration in the total sample (including the reprocessed portion and the remaining effluent bottles from the first cesium column experiment) was estimated to be 0.0152 µCi/mL Cs-137/Na [M] (1.29E3 µCi total Cs-137) which corresponds to an overall Cs-137 DF of 2451 (99.94% Cs removal). The decontaminated AN-102 solution was sampled and analyzed following the Tc ion exchange column test to confirm the Cs-137 concentration was below the maximum allowable amount for vitrifying this waste sample.

### 3.3 SuperLig<sup>®</sup> 639 Batch Contact Experiments

Technetium distribution coefficients and % removal measured for SuperLig<sup>®</sup> 639 with the small and large portions of the Tank 241-AN-102 sample are provided in Table 4. The total Tc concentration measured for the small and large Env. C samples was 4,582 and 4,769 µg/L, respectively. The Tc K<sub>d</sub>'s measured for the two samples were 15.5 mL/g for the small sample and 42.1 mL/g for the large sample. The concentration of nonpertechnetate forms of Tc was calculated for each sample based on the amount of extractable Tc in subsequent recontact tests. These calculations indicate that 37% of the Tc is TcO<sub>4</sub><sup>-</sup> in the large Envelope C sample, while only 22% of the technetium in the small sample was in the extractable TcO<sub>4</sub><sup>-</sup> form. This observation was surprising since the small and large portions of the Tank 241-AN-102 sample were treated similarly.



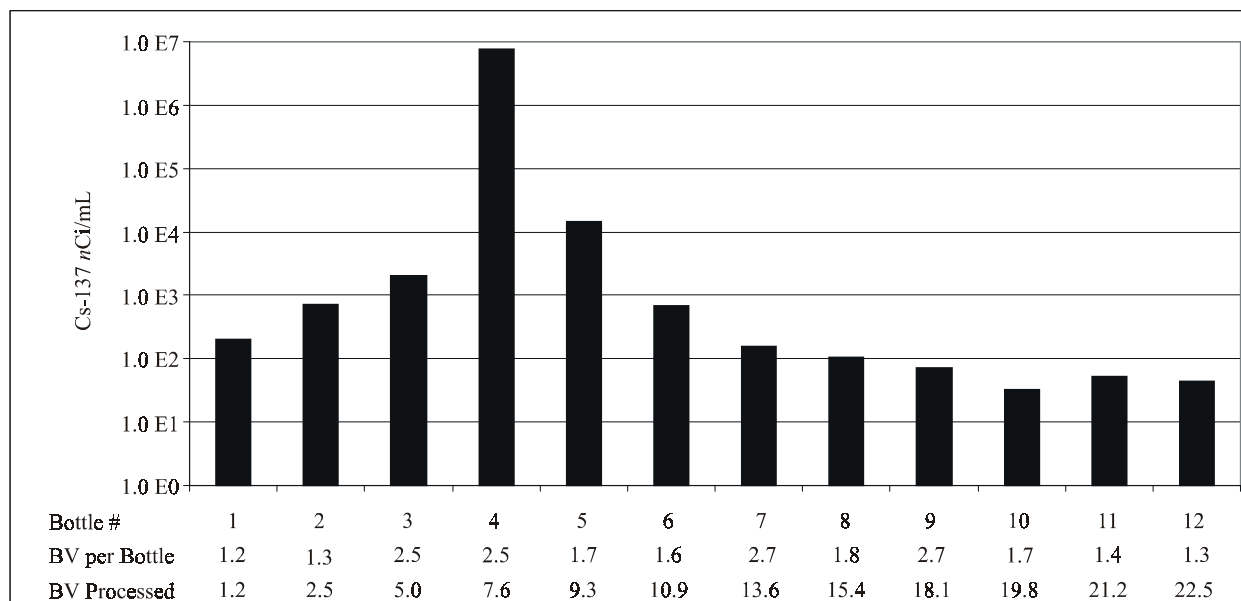


Figure 6. SuperLig<sup>®</sup> 644 Lead Column Eluate Fractions Cs-137 Profile with the Large Envelope C Sample (Column ID: 2.7 cm, Flow Rate: 1.1 BV/hr, Resin Batch #981020MB48-563, Resin Bed Volume: 107 mL, Eluate: 0.5 M HNO<sub>3</sub>)

Table 4. Technetium Distribution Coefficients (K<sub>d</sub>) and % Removal Measured for SuperLig<sup>®</sup> 639 (Batch #981015DHC720011) and the Tank 241-AN-102 Sample

**Small Tank 241-AN-102 Sample**

Tc initial ( $\mu\text{g/L}$ )	Tc final ( $\mu\text{g/L}$ )	Tc K <sub>d</sub> (mL/g)	% Tc Removal	TcO <sub>4</sub> <sup>-</sup> initial ( $\mu\text{g/L}$ )	TcO <sub>4</sub> <sup>-</sup> final ( $\mu\text{g/L}$ )	TcO <sub>4</sub> <sup>-</sup> K <sub>d</sub> (mL/g)	% TcO <sub>4</sub> <sup>-</sup> Removal
4582	3991.5	15.53	12.9	1022	431.5	158.4	57.8
3991.5**	3569.5	10.73	9.2	431.5	0	0.0	100.0
3569.5**	3550.5	0	0	0	0	--	0.0

assumed nonpertechnetate conc. 3560  $\mu\text{g/L}$

original % pertechnetate 22.3 %

**Large Tank 241-AN-102 Sample**

Tc initial ( $\mu\text{g/L}$ )	Tc final ( $\mu\text{g/L}$ )	Tc K <sub>d</sub> (mL/g)	% Tc Removal	TcO <sub>4</sub> <sup>-</sup> initial ( $\mu\text{g/L}$ )	TcO <sub>4</sub> <sup>-</sup> final ( $\mu\text{g/L}$ )	TcO <sub>4</sub> <sup>-</sup> K <sub>d</sub> (mL/g)	% TcO <sub>4</sub> <sup>-</sup> Removal
25541.0*	6834.0	265.9	73.2	22540.0	3833.0	386.3	83.0
4769.0	3369.5	42.1	29.3	1768.0	368.5	389.8	79.2
3369.5**	2941.5	14.2	12.7	368.5	0.0	--	100.0
2941.5**	3060.0	0.0	0.0	0.0	0.0	--	0.0

assumed nonpertechnetate conc. 3001  $\mu\text{g/L}$

original % pertechnetate 37.1 %

\* Indicates samples which had been spiked with pertechnetate

\*\* Indicates recontact tests in which the feed solution was filtrate isolated from previous batch contacts

Based on this data,  $\text{TcO}_4^- K_d$ 's calculated for each original sample were 158.4 mL/g (small sample) and 389.8 mL/g (large sample). It should be noted that the calculated concentrations of pertechnetate after the initial contact in samples from both the small and large portions of the Envelope C sample were small ( $<0.5$  mg/L) relative to the total Tc remaining ( $\sim 3.5$  mg/L). This may have resulted in significant error in the calculated pertechnetate concentrations.

The theoretical 50%  $\text{TcO}_4^-$  breakthrough point ( $\lambda$ ) for the large portion of the Env. C sample can be predicted from  $K_d$  data and the swollen bulk resin density using Eqn. 3. The bulk resin density for batch #980624001DC was reported by SRTC to be 0.489 g/mL.<sup>8</sup> The  $\text{TcO}_4^- K_d$  measured for the initial large Env. C sample batch contact was  $\sim 400$  mL/g (final  $[\text{TcO}_4^-]$ : 368.5  $\mu\text{g/L}$ ;  $\sim 21\%$  of the feed  $[\text{TcO}_4^-]$ ).

$$\lambda = (K_d) \times (\text{bulk resin density}) = (400) \times (0.489) = 196 \quad (3)$$

Previous Re batch contact tests with SuperLig<sup>®</sup> 639 indicate that batch #990420DHC720067 has a 31% higher capacity than batch 981015DHC720011 at comparable final Re concentrations.<sup>16</sup> Based on this data the 50%  $\text{TcO}_4^-$  breakthrough would be expected to occur after  $>196$  BV of feed have been processed.

### 3.4 SuperLig<sup>®</sup> 639 Column Experiment

The lead column Tc breakthrough profile observed for the Large Env. C sample is provided in Figure 7. During the cesium ion exchange column tests the guard column effluent was collected in 1 L bottles and was not composited prior to processing the sample through the SuperLig<sup>®</sup> 639 columns for Tc removal. The first effluent bottles collected during the two cesium column experiments which were diluted by the 0.25 M NaOH pretreatment solution were processed through the Tc ion exchange columns first. The Tc breakthrough rapidly increased to  $\sim 70\%$  after 20 BV of solution had been processed and remained nearly constant (range: 68-77% breakthrough) throughout the remainder of the experiment (total volume processed: 114 BV). The effluent from the guard column was collected in  $\sim 5$  BV fractions and samples from each bottle were analyzed for Tc-99. The analysis results for the guard column fractions are provided in Figure 8. The guard column breakthrough profile is very similar to the lead column breakthrough. The Tc-99 breakthrough stabilized near 70% after  $\sim 20$  BV of solution had been processed and remained nearly constant (range: 67-77%) throughout the remainder of the experiment. The lead and guard column breakthrough data indicate that the lead column removed virtually all of the pertechnetate, such that little pertechnetate actually reached the guard column. This is consistent with the  $\lambda$  value predicted from the batch contact data, which indicated that the  $\text{TcO}_4^-$  breakthrough would not be observed until  $>196$  BV of solution had been processed.  $\sim 70\%$  Tc breakthrough was also observed for the small portion of the Envelope C sample using a 5.6 mL column. The similarity

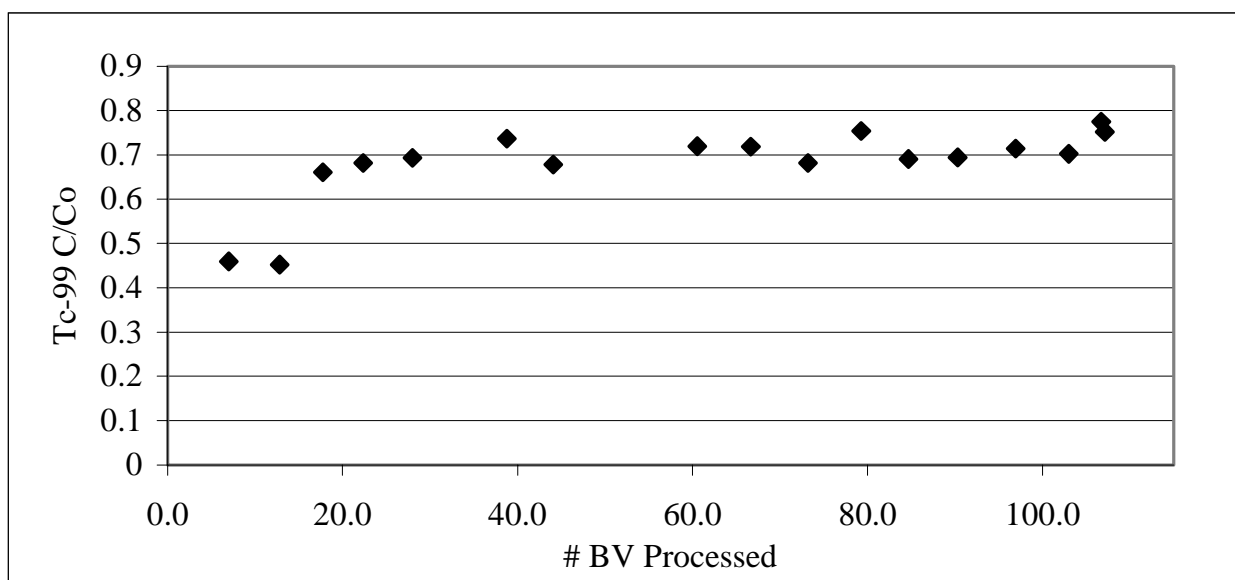


Figure 7. SuperLig® 639 Lead Column Tc-99 Breakthrough Profile with the Large Env. C Sample (Column ID: 2.7 cm, Flow Rate: 2.2 BV/hr, Resin Batches: 50:50 Mixture of 981015DHC720011 and 990420DHC720067, Resin Bed Volume: 147 mL)

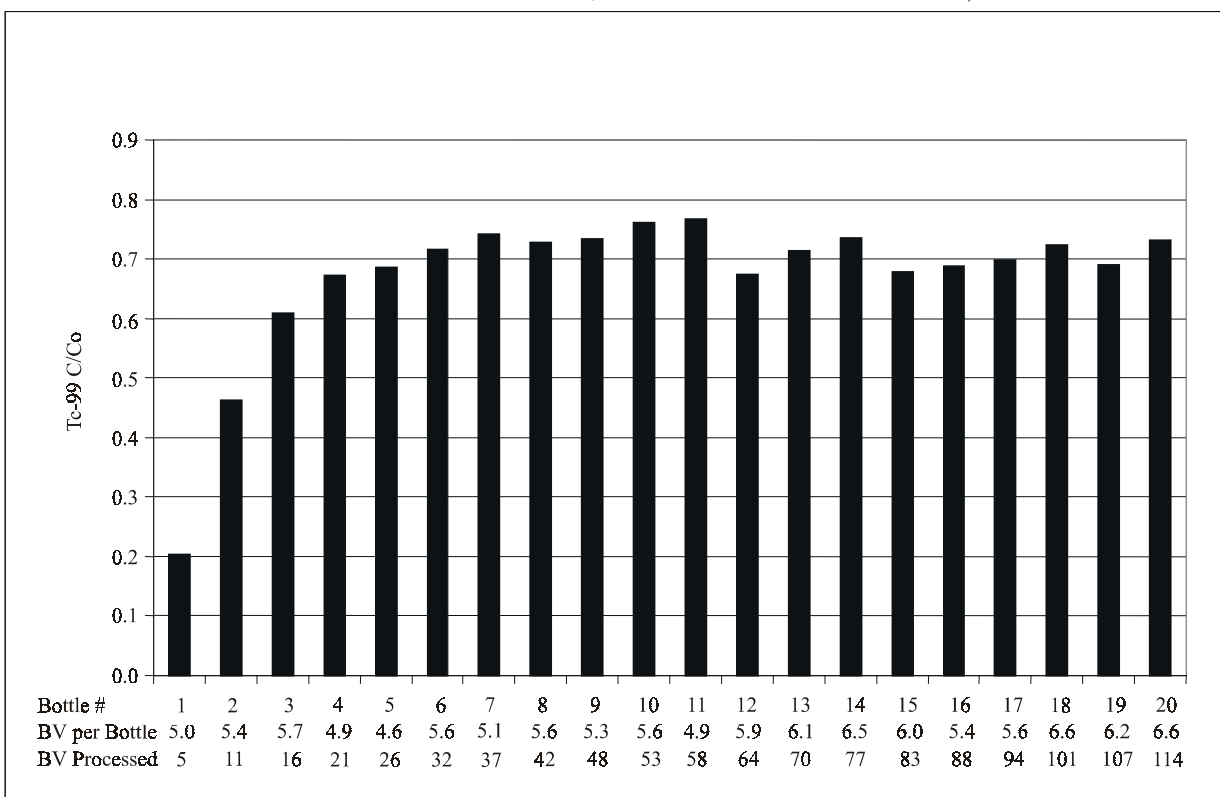


Figure 8. SuperLig® 639 Guard Column Effluent Fractions Tc-99 Breakthrough Profile with the Large Envelope C Sample (Column ID: 2.7 cm, Flow Rate: 2.2 BV/hr Resin Batches: 50:50 Mixture of 981015DHC720011 and 990420DHC720067, Resin Bed Volume: 147 mL)

between the SuperLig<sup>®</sup> 639 column performance (~30% extractable Tc) observed for the small and large Env. C samples contrasts with the batch contact data which indicated that the fractions of pertechnetate in the two samples were different. Following the completion of the SuperLig<sup>®</sup> 639 column experiment, the Cs/Tc-decontaminated effluent was removed from the Shielded High Level Cells and composited in a radiological hood. After allowing sufficient time for mixing, samples of the composite were collected and analyzed (Attachment 8). The composited effluent contained 46.4 mg of Tc-99 (2.78 mg/L, 16.7 L), which corresponds to 65% of the total Tc-99 in the Cs-decontaminated feed (4.305 mg/L Tc-99, 16.7 L, 71.8 mg Tc-99). Integration of the lead column breakthrough profile indicates that 24.6 mg of Tc-99 (34.3% of the feed [Tc-99]) were adsorbed by the column. The amounts of Tc-99 adsorbed to the lead column and contained in the guard column effluent account for 99.2% of the total Tc-99 in the large portion of the Env. C sample. The Cs-137 activity in the decontaminated sample was 0.0109  $\mu\text{Ci/mL}$  Cs-137/Na [M], which is consistent with the value estimated earlier (0.0152  $\mu\text{Ci/mL}$  Cs-137/Na [M]), considering some dilution by the SuperLig<sup>®</sup> 639 column pretreatment solution. Comparison of the  $\text{K}^+$  concentrations in the initial large portion of the Env. C sample (994 mg/L) and the final decontaminated product (951 mg/L), indicates that the final Cs/Tc-decontaminated sample contained ~4.3% column pretreatment solution.

Following the loading cycle, the columns were washed by transferring 3 BV of 0.1 M NaOH through the columns in series at a flow rate of 2.5 BV/hr. The eluate was collected from the guard column in ~1 BV fractions and 2-3 mL grab samples were collected each time that the collection bottles were changed. The grab samples and the wash fractions were analyzed for Tc-99,  $\text{Na}^+$ , and  $\text{OH}^-$ . Analysis results for the wash samples are provided in Table 5. During the wash cycle, the Tc-99 concentration in the effluent decreased from 67% to 6.2% of the feed [Tc]. An earlier SRTC document describing SuperLig<sup>®</sup> 639 ion exchange column tests on a Savannah River Site Tank sample reported increasing Tc-99 concentrations in successive wash samples.<sup>17</sup> However, no guard column was used in this experiment and the  $\text{OH}^-$  concentration of the sample was significantly higher (total  $\text{OH}^-$ : 4.50 M) than the Tank 241-AN-102 sample, which resulted in much higher loading in the column used for the SRS sample. These observations may explain the opposing trends noted in the wash sample compositions for

Table 5. SuperLig<sup>®</sup> 639 Column Post-Feed 0.1 M NaOH Wash Solution Characterization Data

Sample	Cumulative BV	Tc-99 ( $\mu\text{g/L}$ )	Tc C/Co	Na (M)	Free $\text{OH}^-$ (M)	Total $\text{OH}^-$ (M)
Grab 1	0.8	2896.0	0.673	-----	-----	-----
Grab 2	1.9	2568	0.597	-----	-----	-----
Grab 3	2.3	268	0.062	-----	-----	-----
Comp. 1	0.8	2949	0.685	5.24	1.23	1.98
Comp. 2	1.9	2932	0.681	5.30	1.25	1.97
Comp. 3	2.3	957	0.222	2.17	0.50	0.60

Note: Grab = eluate samples collected directly from the guard column eluate collection line; Comp. = samples taken from the eluate collection bottles

these two samples. The  $\text{Na}^+$  and  $\text{OH}^-$  concentrations of successive wash sample fractions collected during the Tank 241-AN-102 column experiment follow the expected trends.

Following the wash cycle, the lead column was eluted with water. The eluate was collected in 3-4 BV fractions and 2-3 mL grab samples were collected for analysis each time that the collection bottle was changed. The lead column Tc-99 elution profile is shown in Figure 9. The Tc-99 peak concentration was observed after 10.4 BV of eluate had been processed. The Tc-99 concentration did not reach <1% of the feed (the current plant design criteria for full elution) until 31 BV of eluate had been processed (Figure 10). The first 31 BV of eluate from the lead column (total volume: 4,526 mL) were composited for further characterization and vitrification. Analysis results for the composited lead column Tc eluate are provided in Attachment 7. The composited eluate contained 20.2 mg Tc-99 based on ICP-MS analysis, which corresponds to 82% of the Tc-99 adsorbed to the lead column (based on integration of the breakthrough curve). Ion selective electrode (ISE) analysis indicated a high concentration of chloride (>15 g/L) in the technetium eluate. In contrast, ion chromatography analysis indicated that the chloride concentration was <1000 mg/l. Observations of high chloride analysis by ISE with this sample type have been reported previously.<sup>12</sup>

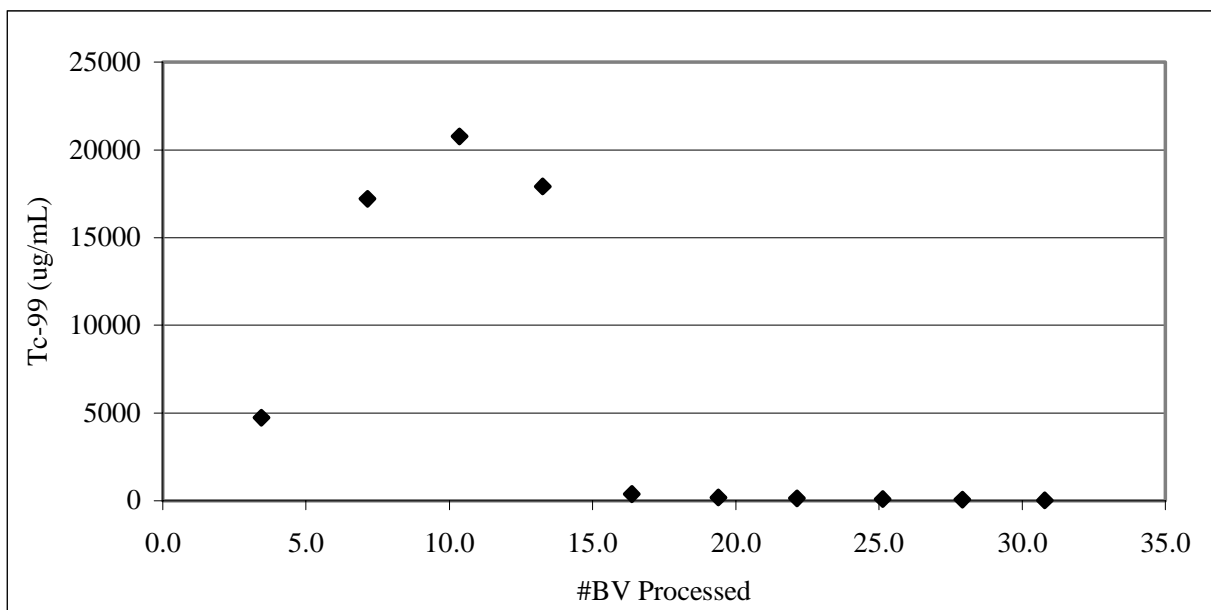


Figure 9. SuperLig® 639 Lead Column Water Elution Profile with the Large Envelope C Sample (Column ID: 2.7 cm, Flow Rate: 0.78 BV/hr, Resin Batches: 50:50 Mixture of 981015DHC720011 and 990420DHC720067, Resin Bed Volume: 147 mL)

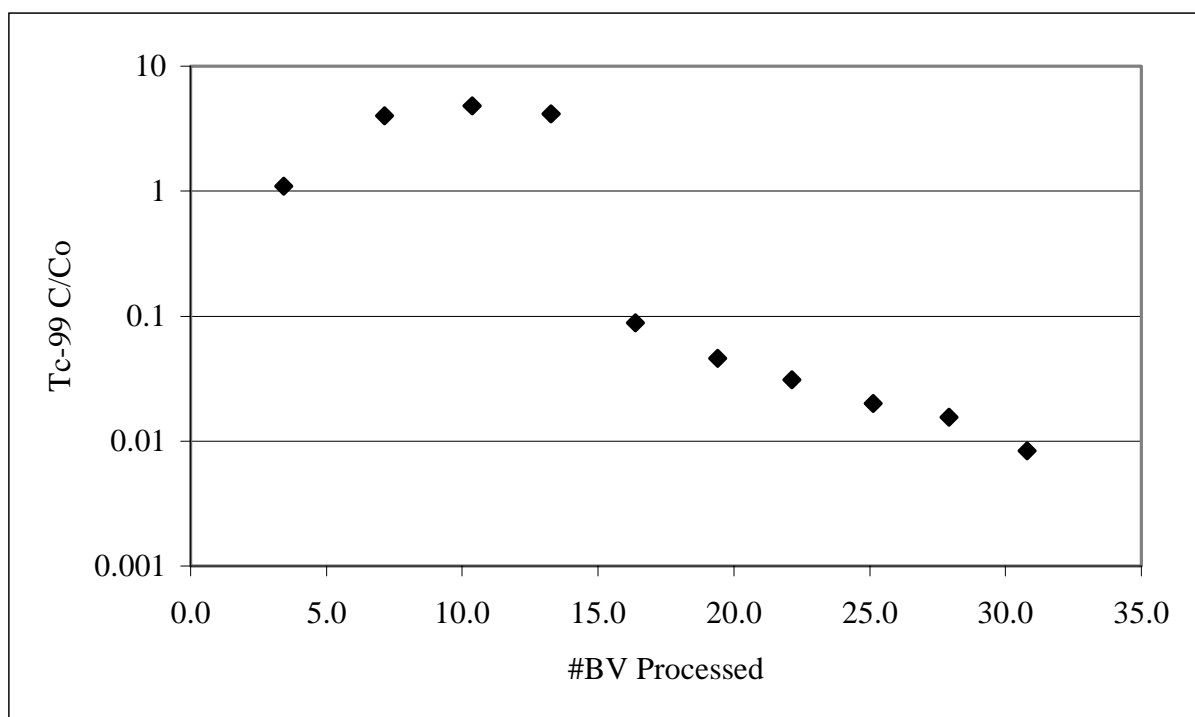


Figure 10. SuperLig® 639 Lead Column Water Elution Profile with the Large Envelope C Sample – SemiLog Plot (Column ID: 2.7 cm, Flow Rate: 0.78 BV/hr, Resin Batches: 50:50 Mixture of 981015DHC720011 and 990420DHC720067, Resin Bed Volume: 147 mL)

## 4.0 CONCLUSIONS

SuperLig<sup>®</sup> ion exchange resins were utilized to produce a final decontaminated Tank 241-AN-102 sample that contained 0.0109  $\mu\text{Ci/mL}$  Cs-137/ Na [M] and 0.574 mg/L Tc-99/Na [M] (final [Na]: 4.85 M). Poor Cs removal was initially observed using SuperLig<sup>®</sup> 644 ion exchange columns, presumably as a result of the presence of insoluble solids or specific ions which competed for resin adsorption sites. Approximately 10.9 bed volumes of 0.5 M nitric acid were needed for thorough elution of the lead SuperLig<sup>®</sup> 644 column. Reprocessing of the sample through the SuperLig<sup>®</sup> 644 columns following column regeneration, resulted in sufficient Cs decontamination to meet the plant vitrified LAW specifications. Further work is needed to understand the poor performance of the SuperLig<sup>®</sup> 644 columns with the large portion of the Tank 241-AN-102 sample.

Essentially complete removal of pertechnetate ( $\text{TcO}_4^-$ ) ion from the Tank 241-AN-102 sample was accomplished with SuperLig<sup>®</sup> 639 ion exchange columns. Approximately 70% of the total Tc-99, which presumably exists as unextractable nonpertechnetate species, remained in the sample. Approximately 31 bed volumes of water at 22 to 25°C were needed for thorough elution of the lead SuperLig<sup>®</sup> 639 column. Following the completion of the column experiments, the final decontaminated product and the concentrated eluates were provided for vitrification into low- and high-activity glass, respectively.

## 5.0 QUALITY ASSURANCE

McCabe, D. J. Intermediate-Scale Radioactive Ion Exchange Task Technical and Quality Assurance Plan, BNF-003-98-00165, Rev. 0.

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18. King, W. D.; McCabe, D. J.; Hassan, N. M.; Walker, D. D. Intermediate-Scale Ion Exchange Removal of Technetium from Savannah River Site Tank 44 F Supernate Solution, BNF-003-98-0230, Rev. 0.

**7.0 APPENDIX**Attachment 1. Filtered Large Envelope C Sample (SuperLig® 644 IX Column Feed)  
Characterization Data

<b>LIMS# 300145153</b>		μCi/mL
Sr-90		9.14E-01
Pu-238		9.28E-04
Pu-239-240		7.39E-04
Am-241		1.04E-02
Cm-244		1.13E-02
Cs-137		1.51E+02
<b>LIMS# 300145154</b>		mg/L
U		12.6
<b>LIMS# 300145155</b>		
<b>ICP-ES</b>		mg/L
Ag	<	0.60
Al		5775.00
B		15.60
Ba	<	0.20
Ca		97.90
Cd		24.10
Co		1.60
Cr		93.00
Cu		9.70
Fe		2.50
La	<	1.10
Li	<	0.20
Mg		0.30
Mn		1.20
Mo		22.90
Na		110780.90
Ni		162.40
P		828.20
Pb		58.10
Si		8.40
Sn		17.50
Sr		147.10
Ti	<	0.20
V	<	0.30
Zn		2.70
Zr		1.00
<b>LIMS# 300145156</b>		mg/L
F (ISE)	<	1000
Cl (ISE)		1810
Free OH (M)		1.25

## Attachment 1. (cont.)

<b>LIMS# 300145157</b>		
<b>IC Anion</b>		mg/L
F		600
Formate		4200
Chloride		2500
Nitrite		30300
Nitrate		82400
Phosphate	<	10000
Sulfate		5000
Oxalate	<	10000
<b>LIMS# 300145158</b>		mg/L
Na (AA)		111912
K (AA)		993.9

Attachment 2. SuperLig<sup>®</sup> 644 Batch Contact Data

Sample ID	Sample wt. (g)	Density (g/mL)	Resin wt. (g)	Phase ratio	[Cs-137] (μCi/mL)	Dilution Factor	[Cs-137] dil. Correct (μCi/mL)	Cs-137 removal (μCi/mL)	K <sub>d</sub> (mL/g)	% R
BNF-LC305-CTR-1	22.253	1.234	na	na	13.67	11.97	164	na		
BNF-LC305-S644-1	21.834	1.234	0.1803	98.1	41.91	na	41.91	121.7	315.2	74.4
BNF-LC305-S644-1D	21.305	1.234	0.1802	95.8	44.75	na	44.75	118.9	281.5	72.6
<b>1<sup>st</sup> Re-contact</b>								avg.	298.4	73.5
BNF-LC305-S644-1R-1	14.863	1.234	0.1208	99.7	12.80	na	12.801	29.11	250.8	69.5
BNF-LC305-S644-1R-1D	14.620	1.234	0.1226	96.6	9.517	na	9.517	35.23	395.8	78.7
								avg.	323.3	74.1
<b>2<sup>nd</sup> Re-contact</b>										
BNF-LC305-S644-2R-1	9.756	1.234	0.0836	94.6	1.802	na	1.802	11.00	638.5	85.9
BNF-LC305-S644-2R-1D	9.792	1.234	0.0828	95.8	1.398	na	1.398	8.119	615.7	85.3
								avg.	627.1	85.6
<b>non-rad Cs Spike</b>					Cs (μg/L)		Cs (μg/L)			
BNF-LC305-CSspk-1	10.660	1.234	na	na	17472	11.6	202522	na	na	na
BNF-LC305-644-spk-1	12.229	1.234	0.1043	95.0	77356	na	77356	125166	170.1	61.8
BNF-LC305-644-spk-1D	12.474	1.234	0.1022	98.9	76604	na	76604	125918	179.8	62.2
								avg.	175.0	62.0

Attachment 3. SuperLig<sup>®</sup> 644 Column Loading/Elution DataLead Column Feed Cycle Grab Samples

ADS#	Sample ID	Cs-137 (μCi/mL)	# BV	C/Co
3-136124	BNF-5-LC-FS-1	1.92E+00	9.2	0.01
3-136125	BNF-5-LC-FS-2	7.85E+00	12.4	0.04
3-136126	BNF-5-LC-FS-3	1.43E+01	21.4	0.08
3-136127	BNF-5-LC-FS-4	2.72E+01	31.2	0.15
3-136128	BNF-5-LC-FS-5	2.16E+01	42.2	0.12
3-136129	BNF-5-LC-FS-6	2.72E+01	48.8	0.15
3-136130	BNF-5-LC-FS-7	3.99E+01	68.2	0.22
3-136131	BNF-5-LC-FS-8	4.21E+01	77.4	0.24
3-136132	BNF-5-LC-FS-9	5.05E+01	86.8	0.28
3-136133	BNF-5-LC-FS-10	5.80E+01	94.5	0.32
3-136134	BNF-5-LC-FS-11	5.77E+01	102.3	0.32
3-136135	BNF-5-LC-FS-12	6.94E+01	110.0	0.39
3-136136	BNF-5-LC-FS-13	7.73E+01	117.5	0.43
3-136137	BNF-5-LC-FS-14	8.25E+01	125.1	0.46
3-136138	BNF-5-LC-FS-15	9.15E+01	132.4	0.51
3-136139	BNF-5-LC-FS-16	9.15E+01	139.2	0.51
3-136140	BNF-5-LC-FS-17	1.15E+02	147.6	0.65
3-136141	BNF-5-LC-FS-18	1.20E+02	155.1	0.67
3-136142	BNF-5-LC-FS-19	1.21E+02	162.7	0.68
3-136143	BNF-5-LC-FS-20	1.27E+02	170.4	0.71
3-136956	BNF-5-BK-FD	1.79E+02	NA	NA

Guard Column Effluent Composite Bottles

ADS #	Bottle #	# BV per Bottle	Cumulative # BV	Cs-137 (μCi/mL)
3-136096	1	6.8	6.8	4.83E-02
3-136107	2	9.5	16.3	1.08E-01
3-136098	3	9.0	25.3	7.31E-02
3-136099	4	9.8	35.2	3.00E-01
3-136100	5	9.4	44.6	5.68E-01
3-136101	6	4.8	49.3	8.85E-01
3-136102	7	18.7	68.0	1.67E+00
3-136103	8	9.3	77.3	2.58E+00
3-136104	9	9.3	86.6	2.58E+00
3-136105	10	7.7	94.4	3.18E+00
3-136106	11	7.7	102.1	3.98E+00
3-136097	12	7.7	109.9	4.51E+00
3-136108	13	7.5	117.4	5.84E+00
3-136109	14	7.6	125.0	3.32E+00
3-136110	15	7.4	132.3	7.32E+00
3-136111	16	6.8	139.1	9.65E+00
3-136112	17	8.4	147.5	1.21E+01
3-136113	18	7.5	155.0	1.76E+01
3-136114	19	7.6	162.6	1.83E+01
3-136115	20	7.7	170.4	2.20E+01

## Attachment 3. (cont.)

Lead Column Eluate Composite Bottles

<b>LIMS</b>	<b>Sample ID</b>	<b>Cs-137 (<math>\mu</math>Ci/mL)</b>	<b>Cumulative #BV</b>	<b>Cs-137 C/Co</b>
3-136154	BNF-5-LC-EC-1	0.200	1.2	0.00112
3-136155	BNF-5-LC-EC-2	0.706	2.5	0.00394
3-136156	BNF-5-LC-EC-3	2.079	5.0	0.01162
3-136157	BNF-5-LC-EC-4	7831.4	7.6	43.7508
3-136158	BNF-5-LC-EC-5	14.578	9.3	0.08144
3-136159	BNF-5-LC-EC-6	0.688	10.9	0.00385
3-136160	BNF-5-LC-EC-7	0.154	13.6	0.00086
3-136161	BNF-5-LC-EC-8	0.108	15.4	0.0006
3-136162	BNF-5-LC-EC-9	0.072	18.1	0.0004
3-136163	BNF-5-LC-EC-10	0.033	19.8	0.00018
3-136164	BNF-5-LC-EC-11	0.052	21.1	0.00029
3-136165	BNF-5-LC-EC-12	0.045	22.5	0.00025

## Attachment 4. Composited Cs Eluate Characterization Data

<b>ICP-ES (mg/L)</b>		<b>140091</b>		<b>140092</b>		<b>Average</b>
Al	<	12.00	<	12.00	<	12.00
B		11.80		9.05		10.43
Ba	<	1.00	<	1.00	<	1.00
Ca		40.95		39.20		40.08
Cd	<	1.50	<	1.50	<	1.50
Co	<	2.50	<	2.50	<	2.50
Cr		12.80		12.20		12.50
Cu		24.15		23.90		24.03
Fe	<	3.00	<	3.00	<	3.00
La	<	5.50	<	5.50	<	5.50
Li	<	1.00	<	1.00	<	1.00
Mg		0.75	<	0.50	<	0.63
Mn		2.20		1.70		1.95
Mo	<	3.00	<	3.00	<	3.00
Na		2648.60		2649.85		2649.23
Ni		84.95		86.35		85.65
P		26.80		15.35		21.08*
Pb		14.30		18.90		16.60*
Si	<	8.00	<	8.00	<	8.00
Sn	<	7.50	<	7.50	<	7.50
Sr		1.40		1.35		1.38
Ti	<	1.00	<	1.00	<	1.00
V	<	1.50	<	1.50	<	1.50
Zn	<	1.50	<	1.50	<	1.50
Zr	<	2.00	<	2.00	<	2.00
<b>ICP-MS (µg/L)</b>						
Mass 99		167.26		139.54		153.40
Mass 235 (U)		490.34		494.62		492.48
Mass 238 (Pu + U)		52846.04		53299.58		53072.81
<b>Ion Selective Electrode (mg/L)</b>						
F <sup>-</sup>	<	500	<	500	<	500
<b>Radiocounting (µCi/mL)</b>						
Pu-239/240		2.41E-03		1.93E-03		2.17E-03
Pu-238		1.99E-03		1.31E-03		1.65E-03
Am-241		3.96E-03		5.25E-03		4.61E-03
Cm-244		2.52E-02		4.10E-02		3.31E-02
Cs-137		1.81E+03		1.81E+03		1.81E+03
Sr-90		2.34E-02		2.24E-02		2.29E-02
Tc-99		1.07E-02		1.71E-02		1.39E-02

\* Dilution-corrected minimum detection limits: P – 13.0 mg/L, Pb – 14.0 mg/L

## Attachment 4. (Cont.)

AA (mg/L)		140087		140088		Average
Na		2.63E+03		2.50E+03		2.57E+03
Hg		8.25E-01		4.95E-01		6.60E-01
As		2.47E+00		2.35E+00		2.41E+00
K		1.28E+02		1.25E+02		1.26E+02
Cs		7.70E+01		7.73E+01		7.71E+01
IC Anion (mg/L)						
F <sup>-</sup>	<	1.00E+02	<	1.00E+02	<	1.00E+02
Cl <sup>-</sup>	<	1.00E+02	<	1.00E+02	<	1.00E+02
NO <sub>2</sub> <sup>-</sup>	<	5.00E+02	<	5.00E+02	<	5.00E+02
NO <sub>3</sub> <sup>-</sup>		1.80E+04		1.80E+04		1.80E+04
PO <sub>4</sub> <sup>3-</sup>	<	5.00E+02	<	5.00E+02	<	5.00E+02
SO <sub>4</sub> <sup>2-</sup>	<	2.50E+02	<	2.50E+02	<	2.50E+02
oxalate	<	5.00E+02	<	5.00E+02	<	5.00E+02
formate	<	5.00E+02	<	5.00E+02	<	5.00E+02



Attachment 5. SuperLig<sup>®</sup> 639 Batch Contact Data

Sample ID	Sample wt. (g)	Density (g/mL)	Resin wt. (g)	Phase ratio	Tc-99 (µg/L)	Dilution factor	[Tc-99] Dil. Correct (µg/L)	[Tc-99] removal (µg/L)	K <sub>d</sub> (mL/g)	% R
BNF-LC305-CTR-1	22.253	1.234	na	na	397.2	12.0	4769	na	na	na
BNF-LC305-S639-1	22.026	1.234	0.1805	98.9	304.2	11.2	3407	1362	40.06	28.6
BNF-LC305-S639-1D	22.512	1.234	0.1809	101	291.4	11.4	3332	1437	44.08	30.1
								avg.	42.07	29.35
<b>1<sup>st</sup> Re-contact</b>										
BNF-LC305-S639-1R-1	13.955	1.234	0.1223	92.5	238.2	12.5	2982	425.3	13.36	12.5
BNF-LC305-S639-1R-1D	14.742	1.234	0.1206	99.1	250.1	11.6	2901	431.5	14.93	13.0
<b>2<sup>nd</sup> Re-contact</b>							2941	avg.	14.15	12.72
BNF-LC305-S639-2R-1	9.611	1.234	0.0813	95.8	262.0	11.8	3081	na	na	na
BNF-LC305-S639-2R-1D	9.900	1.234	0.081	99.0	246.0	12.4	3039	na	na	na
<b>Tc Spike</b>										
BNF-LC305-CSspk-1	12.506	1.234	na	na	1931	13.2	25541	na	na	na
BNF-LC305-639-spk-1	12.614	1.234	0.1012	101	528.0	13.7	7258	18283	257.8	71.6
BNF-LC305-639-spk-1D	11.596	1.234	0.1037	90.6	495.1	12.9	6410	19131	274.0	74.9
								avg.	265.9	73.24

## Attachment 6. SuperLig® 639 Column Loading/Elution Data

Lead Column Feed Cycle Grab Samples

ADS #	Sample ID	Cumulative #BV	Tc-99 (ug/L)	C/Co
3-138786	BNF-7-LC315-FD-2	NA	4115	NA
3-137857	BNF-7-LC315-FD-3	NA	4495	NA
		average	4305	NA
3-137858	BNF-7-LC315-LC-FS-1	7.0	1978	0.459
3-137859	BNF-7-LC315-LC-FS-2	12.8	1945	0.452
3-137860	BNF-7-LC315-LC-FS-3	17.8	2844	0.661
3-137861	BNF-7-LC315-LC-FS-4	22.4	2933	0.681
3-137862	BNF-7-LC315-LC-FS-5	28.0	2984	0.693
3-137864	BNF-7-LC315-LC-FS-7	38.8	3170	0.736
3-137865	BNF-7-LC315-LC-FS-8	44.1	2918	0.678
3-137868	BNF-7-LC315-LC-FS-11	60.5	3097	0.719
3-137869	BNF-7-LC315-LC-FS-12	66.7	3093	0.718
3-137870	BNF-7-LC315-LC-FS-13	73.2	2932	0.681
3-137872	BNF-7-LC315-LC-FS-15	79.3	3244	0.754
3-137873	BNF-7-LC315-LC-FS-16	84.7	2971	0.690
3-138496	BNF-7-LC315-LC-FS-17	90.3	2987	0.694
3-138497	BNF-7-LC315-LC-FS-18	96.9	3075	0.714
3-138498	BNF-7-LC315-LC-FS-19	103.0	3024	0.702
3-138499	BNF-7-LC315-LC-FS-20	106.7	3336	0.775
3-138500	BNF-7-LC315-LC-FS-21	107.1	3236	0.752

Guard Column Effluent Composite Bottles

ADS #	Sample ID	Volume per Bottle (mL)	Cum. # BV	Tc-99 (ug/L)	C/Co
3-137874	BNF-7-LC315-GC-FC-1	740.0	5.0	873	0.203
3-137875	BNF-7-LC315-GC-FC-2	800.5	10.5	1993	0.463
3-138648	BNF-6-LC315-GC-FC-2	734.2	16.3	2620	0.609
3-137876	BNF-7-LC315-GC-FC-3	731.4	21.2	2894	0.672
3-137877	BNF-7-LC315-GC-FC-4	676.6	25.8	2951	0.685
3-137878	BNF-7-LC315-GC-FC-5	828.2	31.5	3083	0.716
3-137879	BNF-7-LC315-GC-FC-6	751.1	36.6	3196	0.742
3-137880	BNF-7-LC315-GC-FC-7	833.7	42.3	3133	0.728
3-137881	BNF-7-LC315-GC-FC-8	778.1	47.6	3161	0.734
3-137882	BNF-7-LC315-GC-FC-9	833.4	53.2	3280	0.762
3-137883	BNF-7-LC315-GC-FC-10	725.0	58.2	3306	0.768
3-137884	BNF-7-LC315-GC-FC-11	867.8	64.1	2908	0.675
3-137885	BNF-7-LC315-GC-FC-12	903.5	70.2	3073	0.714
3-137886	BNF-7-LC315-GC-FC-13	957.8	76.7	3168	0.736
3-137887	BNF-7-LC315-GC-FC-14	892.4	82.8	2918	0.678
3-137888	BNF-7-LC315-GC-FC-15	795.9	88.2	2965	0.689
3-137889	BNF-7-LC315-GC-FC-16	825.5	93.8	3004	0.698
3-138501	BNF-7-LC315-GC-FC-17	981.5	100.5	3116	0.724
3-138502	BNF-7-LC315-GC-FC-18	923.7	106.8	2973	0.691
3-138503	BNF-7-LC315-GC-FC-19	983.8	113.5	3148	0.731

## Attachment 6. (cont.)

Lead Column Eluate Grab Samples

<b>LIMS #</b>	<b>Sample ID</b>	<b># BV</b>	<b>Tc-99 (ug/L)</b>	<b>Tc-99 C/Co</b>
3-137897	BNF-7-LC315-LC-ES-1	3.4	17221.6	1.102
3-137898	BNF-7-LC315-LC-ES-2	7.1	4738.4	4.000
3-137899	BNF-7-LC315-LC-ES-3	10.4	20757.6	4.822
3-137900	BNF-7-LC315-LC-ES-4	13.3	17911.4	4.161
3-137901	BNF-7-LC315-LC-ES-5	16.4	379.94	0.088
3-137902	BNF-7-LC315-LC-ES-6	19.4	197.79	0.046
3-137903	BNF-7-LC315-LC-ES-7	22.1	133.29	0.031
3-137904	BNF-7-LC315-LC-ES-8	25.1	86.45	0.020
3-137905	BNF-7-LC315-LC-ES-9	27.9	67.04	0.016
3-137906	BNF-7-LC315-LC-ES-10	30.8	36.14	0.008

## Attachment 7. Composited Tc Eluate Characterization Data

ICP-ES (mg/L)		300137947		3001387948		Average
Al		28.10		41.90		35.00
B		2.50		2.50		2.50
Ba		1.90		1.30		1.60
Ca		41.95		52.40		47.18
Cd		2.05		2.15		2.10
Co		5.20		3.80		4.50
Cr		4.45		3.50		3.98
Cu		3.80		3.95		3.88
Fe		4.95		6.00		5.48
La		6.25		5.50		5.88
Li		1.95		1.35		1.65
Mg		2.75		4.45		3.60
Mn		1.00		0.75		0.88
Mo		3.05		3.00		3.03
Na		90.60		95.15		92.88
Ni		5.70		4.00		4.85
P		13.00		13.00		13.00
Pb		23.90		24.15		24.03
Ru		24.50		24.50		24.50
Si		23.65		20.60		22.13
Sn		10.50		9.60		10.05
Sr		1.40		1.30		1.35
Tc		7.20		4.70		5.95
Ti		2.70		2.25		2.48
V		4.45		3.95		4.20
Zn		4.25		5.55		4.90
Zr		3.20		2.00		2.60
<b>ICP-MS (ug/L)</b>						
99 (Tc)		4646.04		4287.24		4466.64
<b>IC Anion (mg/L)</b>						
NO <sub>3</sub> <sup>-</sup>	<	5000	<	5000	<	5000
NO <sub>2</sub> <sup>-</sup>	<	5000	<	5000	<	5000
PO <sub>4</sub> <sup>3-</sup>	<	5000	<	5000	<	5000
SO <sub>4</sub> <sup>2-</sup>	<	2500	<	2500	<	2500
F <sup>-</sup>	<	1000	<	1000	<	1000
Cl <sup>-</sup>	<	1000	<	1000	<	1000
Formate	<	5000	<	5000	<	5000
Oxalate	<	5000	<	5000	<	5000

## Attachment 7. (cont.)

		<b>300137947</b>		<b>300137948</b>		<b>Average</b>
K (mg/L)	<	6.75	<	6.75	<	6.75
Free OH (M)	<	0.10	<	0.10	<	0.10
Alpha Count (μCi/mL)		4.91E-01		2.75E-02		2.59E-01
Total Beta (μCi/mL)		3.85E-01		7.41E-02		2.30E-01
U (mg/L)	<	0.50	<	0.50	<	0.50
F <sup>-</sup> (mg/L by ISE)	<	1000	<	1000	<	1000
Cl <sup>-</sup> (mg/L by ISE)		1.56E+04		1.59E+04		1.58E+04
Cs-137 (μCi/mL)		3.66E-02		3.76E-02		3.71E-02
Sr-90 (μCi/mL)		3.42E-02		3.22E-02		3.32E-02

## Attachment 8. Cs/Tc Decontaminated Large Env. C Characterization

LIMS#		140519		140520		140521		140522		Average
Co-60 (μCi/mL)		3.81E-02		4.10E-02		4.15E-02		3.92E-02		3.99E-02
Cs-137 (μCi/mL)		4.54E-02		4.66E-02		6.17E-02		5.75E-02		5.28E-02
Eu-154 (μCi/mL)		2.93E-02		3.63E-02		3.71E-02		3.41E-02		3.42E-02
Eu-155 (μCi/mL)		2.27E-02		2.38E-02		2.45E-02		2.18E-02		2.32E-02
Am-241 (μCi/mL)		1.61E-02	<	1.38E-02		1.42E-02		2.10E-02	<	1.63E-02
total alpha (μCi/mL)		8.95E-02		6.09E-02		7.59E-02		3.45E-02		6.52E-02
total beta (μCi/mL)		4.38		4.46		4.11		4.27		4.31
Sr-90 (μCi/mL)		1.41		1.21		1.41		1.37		1.35
<b>ICP-MS (mg/L)</b>										
mass 99		2.77E+00		2.78E+00		2.78E+00		2.81E+00		2.78E+00
mass 230	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 231	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 232 (Th)		1.51E+00		1.46E+00		1.46E+00		1.45E+00		1.47E+00
mass 233	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 234 (U)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 235 (U)		8.88E-03		8.68E-03		1.07E-02		1.03E-02		9.63E-03
mass 236 (U)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 237 (Np)		8.78E-02		8.93E-02		8.82E-02		8.79E-02		8.83E-02
mass 238 (Pu & U)		8.64E-01		8.77E-01		9.39E-01		9.35E-01		9.03E-01
mass 239 (Pu)		1.50E-02		1.54E-02		1.35E-02		1.41E-02		1.45E-02
mass 240 (Pu)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 241 (Am & Pu)		7.46E-03		7.83E-03		7.09E-03		7.69E-03		7.52E-03
mass 242 (Pu)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 243 (Am)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 244 (Cm)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 245 (Cm)	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
mass 246	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03	<	3.30E-03
<b>IC Anion (mg/L)</b>										
formate		4.58E+03		4.65E+03		4.60E+03		4.45E+03		4.57E+03
NO <sub>2</sub> <sup>-</sup>		3.65E+04		3.66E+04		3.62E+04		3.56E+04		3.62E+04
NO <sub>3</sub> <sup>-</sup>		8.56E+04		8.69E+04		8.65E+04		8.48E+04		8.59E+04
SO <sub>4</sub> <sup>2-</sup>		5.59E+03		5.51E+03		5.48E+03		5.32E+03		5.47E+03
PO <sub>4</sub> <sup>3-</sup>		2.68E+03		1.73E+03		1.73E+03		2.64E+03		2.20E+03
Oxalate		1.15E+03		1.00E+03		9.98E+02		1.12E+03		1.07E+03
sp. G. (g/mL)		1.2255		1.2283		1.229		1.2323		1.23E+00
insol solids (wt%)	<	0.002	<	0.002	<	0.002	<	0.002	<	2.00E-03
sol solids (wt%)		30.98		30.58		32.04		32.43		3.15E+01

## Attachment 8. (cont.)

ICP-ES (mg/L)		140515		140516		140517		140518		Average
Al		5.39E+03		4.51E+03		5.70E+03		5.84E+03		5.36E+03
B		1.37E+01		1.14E+01		1.39E+01		1.44E+01		1.34E+01
Ba	<	1.82E-01		4.69E-01	<	1.82E-01	<	1.82E-01	<	2.54E-01
Ca		1.12E+02		9.40E+01		1.18E+02		1.20E+02		1.11E+02
Cd		1.99E+01		1.67E+01		2.12E+01		2.19E+01		1.99E+01
Co		1.57E+00		1.95E+00		1.46E+00		1.24E+00		1.56E+00
Cr		7.07E+01		5.93E+01		7.46E+01		7.64E+01		7.03E+01
Cu		3.58E+00		3.39E+00		3.61E+00		3.76E+00		3.58E+00
Fe		2.14E+00		2.15E+00		1.70E+00		1.73E+00		1.93E+00
La		1.25E+00		2.16E+00	<	1.00E+00	<	1.00E+00	<	1.35E+00
Li		1.83E-01		5.34E-01	<	1.82E-01	<	1.82E-01	<	2.70E-01
Mg	<	9.10E-02		9.70E-02	<	9.10E-02	<	9.10E-02	<	9.25E-02
Mn		8.16E-01		7.83E-01		7.91E-01		8.00E-01		7.98E-01
Mo		2.25E+01		1.81E+01		2.21E+01		2.32E+01		2.15E+01
Na		1.10E+05		1.07E+05		1.16E+05		1.13E+05		1.12E+05
Ni		1.23E+02		1.03E+02		1.33E+02		1.36E+02		1.24E+02
P		7.05E+02		5.71E+02		7.30E+02		7.42E+02		6.87E+02
Pb		4.40E+01		3.92E+01		4.73E+01		4.71E+01		4.44E+01
Si		4.35E+01		1.21E+01		3.91E+01		4.09E+01		3.39E+01
Sn		1.19E+01		9.82E+00		1.10E+01		1.12E+01		1.10E+01
Sr		1.27E+02		1.02E+02		1.37E+02		1.40E+02		1.26E+02
Tc		2.83E+00		2.79E+00		2.41E+00		2.05E+00		2.52E+00
Ti		3.05E-01		7.63E-01	<	1.82E-01	<	1.82E-01	<	3.58E-01
V		6.07E-01		1.29E+00	<	2.73E-01	<	2.73E-01	<	6.10E-01
Zn		1.40E+00		1.25E+00		1.30E+00		1.22E+00		1.29E+00
Zr	<	3.64E-01	<	3.64E-01		5.05E-01	<	3.64E-01	<	3.99E-01
Cl		1.60E+03		1.56E+03		1.56E+03		1.59E+03		1.57E+03
F		7.69E+02		7.71E+02		7.77E+02		7.46E+02		7.66E+02
K (mg/L)		1.06E+03		9.39E+02		8.98E+02		9.11E+02		9.51E+02
As (mg/L)		2.84E-01		2.36E-01		2.14E-01		2.85E-01		2.55E-01
Se (mg/L)		2.19E-01		2.65E-02		9.65E-02		2.25E-01		1.42E-01
Hg (mg/L)	<	2.40E-02	<	1.98E-02	<	1.76E-02	<	1.65E-02	<	1.95E-02
U (mg/L)	<	1.00E+00	<	1.00E+00	<	1.00E+00	<	1.00E+00	<	1.00E+00
Cl (mg/L by ISE)		179.6		188.5		213.2		218.34		2.00E+02
F (mg/L by ISE)		119.4		105.85		93.78	<	10	<	8.23E+01
TIC (mg/L)		5782		5618		6294		5656		5.84E+03
TOC (mg/L)		11432		11617		11814		10732		1.14E+04
Free OH (M)		1.1531		1.207		1.2879		1.3229		1.24E+00