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Cesium Removal from Simulated SRS High-Level Waste Using
Crystalline Silicotitanate

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CONTENTS

CESIUM REMOVAL FROM SIMULATED SRS HIGH-LEVEL WASTE USING CRYSTALLINE SILICOTITANATE

By D. D. WALKER

SUMMARY

This study measured the adsorption of cesium from simulated Savannah River Site liquid waste onto crystalline silicotitanate (CST) in equilibrium (K_d) and ion exchange column tests. The tests used IONSIV® IE-911 (UOP LLC, Molecular Sieves Division, Des Planes, IL), the engineered form of CST. The results of the tests show good agreement with predictions from models used by Professor R. G. Anthony of Texas A&M University. The following summarize the results.

- Cesium adsorption in static (K_d) tests matched predictions from a model developed at Texas A&M for the powder form of CST.
  - Measured K_ds averaged about 10% below predicted values.
  - At 23 °C, K_ds varied between 1560 and 2840 mL/g depending on the composition of the salt solution.
  - Measured trends in K_ds due to changes in solution composition agreed with trends predicted by the model.
  - Measured K_ds in low sodium salt solution (4 M) and at elevated temperature (45 °C) showed less agreement with the model.
  - Differences between production batches were small (<15%).
  - The K_d for the developmental sample (38B) used in previous testing was significantly lower (-27%) than the average for production batches. This difference accounts for the "dilution factor" derived from previous tests.

- Cesium removal in two column tests at rapid feed rates (i.e., superficial velocities of 0.98 and 4.1 cm/min) in the range of proposed process conditions closely matched predictions from Texas A&M.
  - This result implies that current models will predict process column performance without major adjustments to the models.

- Cesium removal in a slow feed rate (i.e., superficial velocity of 0.27 cm/min) column test demonstrated better cesium removal than predicted by the Texas A&M model.
  - Cesium breakthrough remained less than 0.0025% for 300 column volumes. The model predicted cesium would breakthrough at 103 column volumes.

INTRODUCTION

Non-elutable ion exchange is one of the alternatives for removing cesium from Savannah River Site (SRS) soluble radioactive waste.[1] Crystalline silicotitanate (CST) particles are the reference ion exchange medium for the process. Previous studies measured cesium distribution on CST samples in powder form[2] and in the engineered form[3] in SRS simulated wastes. The distribution coefficients (K_ds) measured previously with the engineered form of CST proved consistently smaller than predicted from a Texas A&M model based on the powdered form of CST.[4-5] This is not unexpected since the engineered form contains an inert binder in addition to the powdered form of CST. No previous column experiments examined using CST in SRS wastes. Therefore, Savannah River Technology Center (SRTC) began this study to determine the performance of the IONSIV® IE-911 production batches in SRS waste and quantify the discrepancy between measured K_ds and predictions from the Texas A&M model. Additional K_d tests started at Oak Ridge National Laboratory (ORNL) to supplement the SRTC work. In addition, column tests with production batches of IONSIV® IE-911 in SRS simulated waste initiated at SRTC.

This work partially fulfills a request from P. L. Rutland, Technical Task Request, "CST Ion Exchange - Salt Team Phase 3 Evaluation," #HLE-TAR-98060, July 14, 1998. The work complies with the following plan: D. D. Walker, "Scoping Tests for Crystalline Silicotitanate Flowsheet," WSRC-RP-98-00732, Rev.0, August 21, 1998.

RESULTS AND DISCUSSION

Researchers investigated both equilibrium and dynamic aspects of cesium adsorption on CST particles. The tests occurred at SRTC and ORNL. Appendix A summarizes the ORNL results. Appendix C contains the experimental details for the SRTC tests.

The equilibrium measurements (K_d tests) measured the performance differences between IONSIV® IE-911 production batches as well as the variance in performance with different waste compositions. Column tests at different feed rates measured kinetic aspects of cesium absorption at superficial velocities in the range proposed for full-scale column operations and demonstrated that large cesium decontamination factors result with CST.

The salt solutions used in these tests simulate the variety of compositions expected in processing the inventory of SRS soluble waste (Table I).[1] The major difference in the three waste varieties involves the ratio of hydroxide to nitrate ions. Average waste represents the hypothetical feed composition assuming dissolution of all of the current inventory of salt cake and blending with the total supernate inventory. High hydroxide waste represents partial blending of supernate with dissolved salt cake and reflects the high hydroxide content of the supernates in the waste tanks. High nitrate waste represents the feed composition of dissolved salt cake without blending of supernates.

TABLE I. Composition of Simulated Waste Solutions

Component	Concentration (molar) Average	High OH^-	High NO_3^-
Na^+	5.6	5.6	5.6
K^+	0.015	0.030	0.0041
Cs^+	0.00014	0.00037	0.00014
OH^-	1.91	3.05	1.17
NO_3^-	2.14	1.10	2.84
NO_2^-	0.52	0.74	0.37
AlO_2^-	0.31	0.27	0.32
CO_3^{2-}	0.16	0.17	0.16
SO_4^{2-}	0.15	0.30	0.22
Cl^-	0.025	0.010	0.040
F^-	0.032	0.010	0.050
PO_4^{3-}	0.010	0.008	0.010
$C_2O_4^{2-}$	0.008	0.008	0.008
SiO_3^{2-}	0.004	0.004	0.004
MoO_4^{2-}	0.0002	0.0002	0.0002

K_d Tests

Consistent and reproducible K_d values prove difficult to obtain and require careful attention to the sample preparation and experimental procedure. Factors that may contribute to the scatter in the measurements include water adsorption by IONSIV® IE-911 during storage and handling, differences in cesium adsorption properties between production batches, CST fines adhering to the larger engineered particles, and differences in laboratory protocols for measuring K_ds. The controlled tests described below investigated these factors.

A typical test shakes a portion of CST in a fixed volume of salt solution until equilibrium occurs. Personnel measure initial (C_o) and final (C) concentrations of cesium and calculate the distribution coefficient (K_d) from the following equation.

$$K_d = [(C_o/C)-1]\ V/m$$

where V is the volume of solution
and m is the mass of CST.

The mass of CST used includes a correction for the water lost on drying as measured in a separate experiment.

Water Content of Production Batches

Water content of a developmental sample (38B) and three production batches varied between 4 and 13 wt %. Table II lists measurements of weight loss on drying at 105 °C for as-received IONSIV® IE-911. Weight loss presumably results from vaporization of water retained in the macropores of the IONSIV® IE-911. The water content of the particles will vary depending on the air humidity and exposure. In some cases, particularly Batch 0002, the water content significantly affects the calculated K_ds. To eliminate this source of variability when calculating K_ds, researchers in both laboratories corrected the weight of IONSIV® IE-911 particles for the measured water content.

Evaluation of IONSIV® IE-911 Production Batches

SRTC and ORNL researchers measured K_ds on three production batches and on the developmental sample in average SRS waste simulant. No sample of production Batch 3 existed in the as-received condition. Table III lists the results. The data indicates good agreement between ORNL and SRTC measurements (i.e., compare the two laboratories for Batch 4) and no significant differences among the three production batches. However, the developmental sample (38B) appears to have a significantly lower K_d compared to the production batches.

The SRTC researchers further investigated the developmental sample to confirm the low K_d. McCabe[3] reported a K_d of 1500 (±170) using a salt solution with composition similar to the high hydroxide simulant listed in Table I. Upon repeating the K_d measurements in the McCabe simulant, the Batch 38B K_d (1370 mL/g) again proved significantly smaller than the Batch 2 K_d (1900 mL/g). We conclude a significant difference exists in cesium absorption properties between the two batches. The difference may result from differences in the amount or type of binder used to form the engineered particles.

TABLE II. Water Content in IONSIV® IE-911 Samples.

Batch*	Water Content (wt %)	Laboratory
38B	6.9	SRTC
2	13.0	SRTC
4	3.0	SRTC
4	4.79	ORNL
1	5.46	ORNL

---

*The complete UOP lot number for the preproduction batch (Batch 38B) is IE-910 07398-38B. The complete lot number for production batches begins with "99909681000" and ends with 1, 2, 3, or 4. For simplicity, the listing delete the leading portions of the lot numbers.

TABLE III. Comparison of As-Received Particles

Batch	K_d* (mL/g)	Source**
1	2250	ORNL
2	1940, 2240	SRTC
4	1970	SRTC
4	2150	ORNL
38B	1530	SRTC

---

*Each entry represents the average of two measurements. Multiple entries for Batch 2 indicate duplicate measurements made at different times. Measurements used SRS average waste solution (Table I).

**Laboratory where measurements occurred.

Effects of Particle Fines

As-received IONSIV® IE-911 nominally contain particles of 30 to 60 mesh (600 to 200 micron), but washing the particles produces a cloudy solution presumably due to <200 micron fines. Column operations remove fines from ion exchange material to avoid problems such as plugging the screens holding the packing in the column or contamination of the effluent stream from fines passing through the screen. Testing examined whether fines present in as-received particles used in K_d tests biased the K_d measurements relative to washed particles used in column tests. Table IV lists results of tests comparing as-received particles to particles after removal of fines. The ORNL researchers removed fines by sieving the larger particles while SRTC personnel removed fines by flushing upflow with water. In both laboratories, the particles with fines removed yielded smaller K_ds, although the differences (-3.6 and -13.9%) may fall within the reproducibility of the measurements. However, the consistently lower value for particles with fines removed suggests a small effect.

TABLE IV. Effect of Fines on K_ds*

Batch	Condition	K_d (mL/g)	Source
2	as-received	1940	SRTC
	fines-removed**	1870	SRTC
		(Change: -3.6%)	
4	as-received	2149	ORNL
	fines-removed**	1850	ORNL
		(Change: -13.9%)	

---

*Measured in SRS average waste simulant at room temperature.

**The SRTC tests removed fines by flushing with water; ORNL tests removed fines by sieving.

Accuracy of the Texas A&M K_d model for SRS waste compositions.

Researchers tested the accuracy of the Texas A&M K_d model by investigating the effects of feed type (i.e., ratio of hydroxide to nitrate), total salt concentration, potassium concentration, temperature, and cesium concentration (particularly at very low cesium concentrations). Table IV lists the test matrix as well as the measured and predicted K_ds.

Predictions and measurements agreed within ±15% in all but two tests. Excluding the two outliers, the difference averages -2%. Including the outliers yields an average bias of -8%. Thus, agreement between predictions and measurements, on the average, appears quite good and reflects the experimental uncertainty. In most cases, the Texas A&M model accurately predicts cesium adsorption behavior. The results of this investigation do not support a "dilution" factor between the model (based on powdered form of CST) and the engineered particles. The dilution factor derived from the work of McCabe apparently reflects the poorer performance of the developmental sample rather than a generic dilution by the inert binder.

Two measurements differed significantly from predictions: one with a solution of low salt concentration (i.e., K_d -23% relative to predicted) and one with average solution at the high temperature(i.e., K_d -45% relative to predicted). Although Anthony stated the model loses accuracy at sodium concentrations below 1 molar, one expects a much smaller error than observed at 4 M. Review of the experiment did not reveal anything unusual. Resolution of this discrepancy requires further inquiry. The test at 45 °C exceeds the temperature limit of the Texas A&M K_d model capability (i.e., the computer model will not accept temperatures above 44 °C). The model needs additional experimental data at higher temperature to improve on these predictions.

TABLE V. Measured and Predicted Cesium K_d's*

Variable	Test	Waste type	$[Na^+]$ (M)	$[K^+]$ (mM)	$[Cs^+]$ (mM)	Temp. (°C)	K_d (mL/g) Measured	K_d (mL/g) Predicted
Waste type	1	Average	5.6	15	0.14	22	1940	2230
	2	High OH^-	5.6	30	0.37	22	2240	2460
	3	High NO_3^-	5.6	4.1	0.14	22	1997	1920
$[Na^+]$	4	Average	4.0	15	0.14	22	2840	3690
	5	Average	7.0	15	0.14	22	1560	1540
$[K^+]$	6	Average	5.6	1.5	0.14	22	2260	2440
	7	Average	5.6	150	0.14	22	1350	1370
[Cs+]	8	Average	5.6	15	0.001	22	2530	2360
	9	Average	5.6	15	0.01	22	2540	2350
	10	Average	5.6	15	1.0	22	1700	1490
Temperature	11	Average	5.6	15	0.14	45	800	1450

---

*Measured at SRTC on IONSIV® IE-911,Batch 2. Predictions based on Texas A&M K_d model (Ref. 4). Concentrations listed are at start of test.

Column Tests

Column tests at different feed rates measured kinetic aspects of cesium absorption on engineered CST particles. Table VI lists the details of the three column runs. Figure 1 shows the cesium breakthrough curves for the three experiments. The figure includes predictions of column performance provided by Professor R. G. Anthony of Texas A&M (Appendix B) prior to the tests. The following summarize differences between the predicted and observed cesium breakthrough curves.

- Column performance generally matched predictions in the intermediate and fast column runs.
  - Columns performance prior to 50% breakthrough was slightly better than predicted.
  - In the fast column run, performance after 50 % breakthrough was slightly poorer than predicted.
- Cesium removal greatly exceeded predictions during the early phase of the slow column run.
  - C/C_0 of 0.0025 % was not reached until 300 column volumes, compared to the prediction of 103 column volumes.

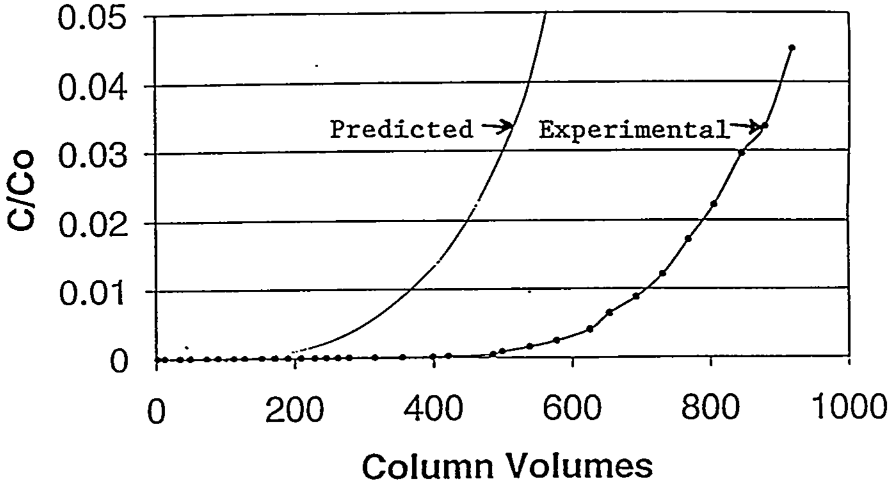
The scatter in the data shown in Figure 1 comes largely from analytical variability. The column data in the intermediate and fast column runs came from inductively coupled plasma mass spectroscopy (ICP-MS). The typical reproducibility of the data is approximately ±10% with sample dilution variances during preparation dominating the error. Future work should consider improvements in the dilution method or use of radioactive tracers to provide better resolution as C/Co approaches 1.

TABLE VI. Details of Ion Exchange Column Runs

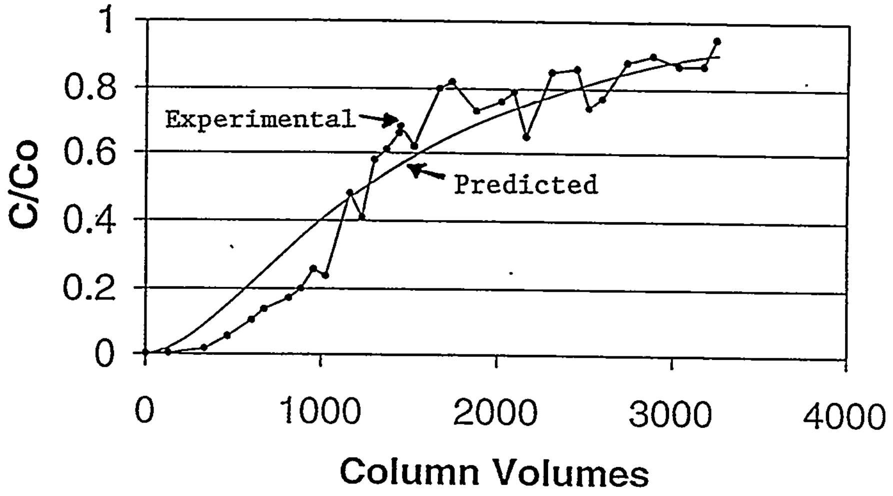
	Slow	Intermediate	Fast
Column size			
Diameter (cm)	1.50	1.50	1.43
Length (cm)	10.0	10.0	11.0
Volume (cm^3)	17.7	17.7	17.7
Flowrate			
Range (mL/min)	0.43-0.49	1.63-1.82	6.42-6.61
Average (mL/min)	0.47	1.73	6.53
(CV/h)	1.59	5.88	22.1
Superficial velocity (cm/min)	0.27	0.98	4.1
IONSIV® IE-911 batch	2	2	4

FIGURE 1. Cesium Breakthrough During Column Runs

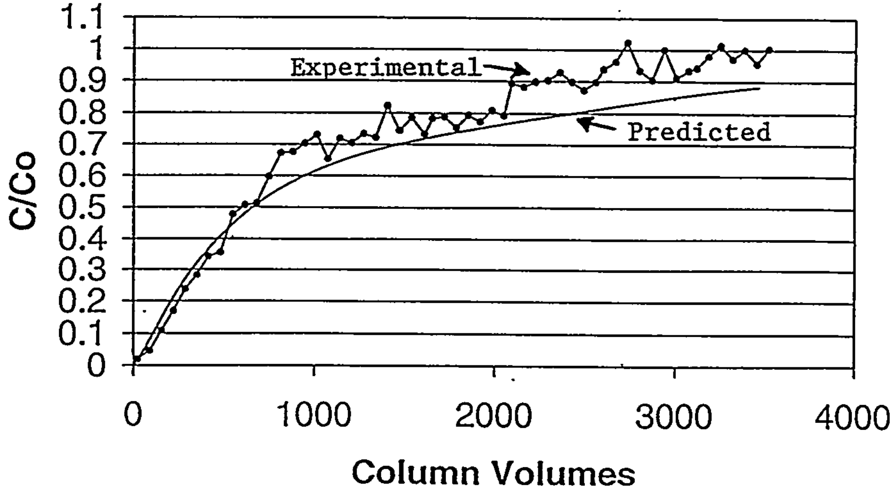
Slow Column
(1.59 CV/h)



Intermediate Rate
(5.8 CV/h)



Fast Rate
(22.1 CV/h)



ACKNLWLEDGMENTS

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APPENDIX A
Oak Ridge National Laboratory K_d Measurements

BATCH TESTS WITH IONSIV® IE-911 AND A SIMULANT OF THE SAVANNAH RIVER SITE "AVERAGE" SUPERNATANT: DISTRIBUTION COEFFICEINTS VS TIME

R. D. Hunt, K. K. Anderson, J. L. Collins, and D. D. Lee

1. INTRODUCTION

The Department of Energy (DOE) is required by law to treat and safely dispose of the radioactive wastes from its nuclear weapon production activities. The primary radionuclide in the DOE liquid wastes, supernatants, is ^{137}Cs. At the Savannah River Site (SRS), the In Tank Precipitation (ITP) process was selected as the baseline technology to remove ^{137}Cs from the supernatants, which are stored in underground storage tanks. In the ITP process, tetraphenylborate reacts with the water-soluble cesium to form a precipitant. The treated supernatant would then be immobilized in grout or saltstone and stored in vaults at the SRS. However, problems were encountered during the full-scale ITP processing.

These difficulties with the ITP process have led to the evaluation of alternative technologies and/or concepts to the currently configured ITP process. The High-Level Waste (HLW) Salt Disposition Team at the SRS is currently performing this assessment. After an initial screening of all potential alternatives, the Salt Disposition Team selected four primary options to evaluate further before the final down selection. Crystalline silicotitanate (CST), an inorganic ion exchanger, was chosen as one of the four alternatives. Since nearly all of the CST tests have been performed on supernatants from Hanford and Oak Ridge, the Salt Disposition Team has requested personnel at the SRS and Oak Ridge National Laboratory (ORNL) to determine the performance of the engineered form of CST, IONSIV® IE-911,with actual and simulated SRS supernatants. It should be noted the UOP, the manufacturer of IONSIV® IE-911, has made the engineered form of CST in a minimum of four different batches, and some variability between the batches can be expected.

In batch tests at the SRS, IONSIV® IE-911 (batch 2) was used to remove ^{137}Cs from actual SRS supernatant from Tank 22H (McCabe and Oji, 1998). These SRS batch tests demonstratated that IONSIV® IE-911 (batch 2) is very effective for cesium removal with a distribution coefficient (Kd) of 14,850 mL/g. These SRS results also indicate that a CST column could process 14,290 column volumes of supernatant before exhaustion. Other SRS personnel are performing batch and column tests on IONSIV® IE-911 (batch 4) with a simulant of the "average" SRS supernatant. ORNL staff members also used the simulant of the "average" SRS supernatants in the batch tests with IONSIV® IE-911 from batches 1, 3, and 4.

Several of the ORNL batch tests required modifications to the CST or to the supernatant simulant. In some of the tests, the CST was used "as received". In other cases, the "treated" CST was used after the IONSIV® IE-911 was treated in NaOH and washed with H_2O. In an effort to determine the effects of fines, a portion of the CST was sieved, and CST particles between 300 and 600 microns were used in a few of the tests. Finally, 2000 mg/L of dibutylphosphate (DBP) was added to a portion of the supernatant simulant to determine the effect of DBP on the performance of CST. The Kds of IONSIV® IE-911 at 24, 72, and 144 h were measured under the following conditions:

1) Batches 1 and 4 - "as received"
2) Batches 1, 3, and 4 - "treated"
3) Batch 4 - "as received" and "treated" - particle size of 300 to 600 microns
4) Batch 4 - "treated" - dibutylphosphate in the supernatant.

This letter report presents the ORNL results from the batch tests with the CST and the simulated SRS supernatant.

2. SAMPLES

2.1 IONSIV® IE-911

CST is a new class of inorganic ion exchangers invented and developed through a collaborative effort between Sandia National Laboratories (SNL) and Texas A&M University. The composition of CST is proprietary, and no information on the specifics of CST synthesis or composition is known. CST is produced as a very fine powder that could be used in a batch process, but it is not suitable for use in an ion-exchange column. An engineered form, IONSIV® IE-911, was developed by UOP, Des Plaines, Illinois, under a Cooperative Research and Development Agreement with SNL. During the Cesium Removal Demonstration at ORNL, columns of IONSIV® IE-911 successfully removed Cs from 25,000 gal of tank supernatant (Walker et al., 1998).

2.2 SIMULANT OF THE "AVERAGE" SRS SUPERNATANT

The recipe for the simulant of the "average" SRS supernatant, which is shown in Attachment 1, was provided for SRS personnel. The SRS simulant was made in two batches, which were 0.5 and 1 L. After the simulant solutions were prepared as described in Attachment 1, they were spiked with ^{137}Cs so the Cs concentration in the solutions could be monitored with gamma counter. Even though the solutions were thoroughly mixed with magnetic stir bars, a small amount of fine solids could be observed in the solutions. After samples were mixed for 5 days, they were passed through a 0.45 μm high capacity in-line filter (Gelman Sciences) and combined. The density of the composite supernatant was 1.254 g/mL, which is very comparable to the density of the solutions before filtration. A portion of the composite supernatant was used to prepare the sample with 2000 mg/L of dibutylphosphate (DBP); 0.4058 g of DBP (Eastman Chemicals) was added to 200 mL of the composite simulant. Even though the supernatant with DBP was thoroughly mixed, an organic layer appeared on top of the solution.

3. EXPERIMENTAL PROCEDURES

3.1 Water Content

The water contents in the "treated" CST (batches 1, 3, and 4) and "as received" CST (batches 1 and 4) were determined before the batch tests were initiated. Approximately 0.5 g of IONSIV® IE-911 was placed into a previously weighed aluminum foil boat. After the boat and the CST were weighed with a Mettler AE200 balance, they were heated at 105°C until the dried CST reached a constant weight. Before the dried CST was weighed, the hot boat was placed in a desiccator until the CST had cooled to ambient temperature.

3.2 Kd Measurements

The Kd measurements were made using batch equilibration tests and techniques, which were previously used with IONSIV® IE-911 (Davidson et al. 1998). Duplicate samples of CST and simulant were used for each of the test conditions. Approximately 0.21 g (batches 1 and 4) or 0.25 g (batch 3) of IONSIV® IE-911 was placed into a previously weighed 50–mL Oak Ridge centrifuge tube with a sealing cap. The goal of this step was to place 0.200 g of CST on a dry weight basis into each tube. After the CST and tube were weighed, approximately 40 mL of the supernatant simulant was added to the centrifuge tube, which was weighed after the transfer. In the initial test plan, 50 mL of the simulant was to be added to each tube. However, the capacity of a 50–mL Oak Ridge centrifuge tube is slightly more than 40 mL. The volume of simulant was calculated from the specific gravity of the supernatant. The IONSIV® IE-911 and the simulated supernatant were mixed with a Labquake™ shaker. The action of the mixer is a back-and-forth rocking motion that was set to rock from -45° to +45° from the horizontal plane at ~20 cycles per minute. At the end of 24, 72, and 168 h, the tubes were reweighed to check for leakage. Then, the tubes were then centrifuged for 20 min at 2800 rpm with an International Equipment Company Centra 7 tabletop centrifuge. A small aliquot (3 mL) was removed from each sample with a glass pipet and placed into 15-mL tube. The small aliquots were then filtered through 0.22-μm nylon filters to remove any of the remaining fine particulates. The filtering system consist of a 20-mL glass syringe, a luer-lock filter, and a short piece of tygon tubing. Samples (0.5-1.0 mL) of the filtered supernatants were pipetted into preweighed counting tubes for radiochemical analysis. The remaining liquid in the 15-mL tube was set aside for subsequent tests as necessary. At the completion of all of the transfers, the Oak Ridge centrifuge tube and the counting tubes were weighed. The volumes of the filtered supernatants in the 15-mL and counting tubes were kept as small as possible to minimize changes to the liquid (mL)/solid (mg) ratio during each test. The ^{137}Cs activity was determined using an LKB Wallac 1282 Compugamma Universal Gamma Counter. Samples were counted for 40 min, and an empty tube was counted so the background radiation could be determined and deducted from subsequent measurements. Finally, a control test was also run. 40 mL of supernatant was added to a 50-mL polypropylene centrifuge tubes and mixed for 24, 72, and 168 h. No detectable change in the ^{137}Cs activity of the simulant was noted.

4. CALCULATIONS

The results of batch tests are reported as distribution coefficient (Kd) and percent removal (% R). These values are calculated in the following manner:

$$\text{Kd} = [(C_i - C_f)/C_f][V/m] \text{ (units are mL/g)}$$
$$\% \text{ R} = 100[(C_i - C_f)/C_i].$$

C_i and C_f are the initial and final counts of ^{137}Cs per gram of supernatant, respectively. V is the volume of simulant, and m is the dry mass of the IONSIV® IE-911.

5. RESULTS

5.1 Water Content

The results from the water content experiments on IONSIV® IE-911 are presented in Table 5.1. With batch 1 and batch 4, the water contents were very comparable, and water content was not significantly affected by the treatment with NaOH and H_2O. However, the water content in the "treated" batch 3 of IONSIV® IE-911 was significantly higher than the other batches. This observation was confirmed with a second, larger test with IONSIV® IE-911 (batch 3). It should be noted that the dried IONSIV® IE-911 quickly absorbs moisture even at ambient temperature.

Table 5.1. Water Content in Different Batches of IONSIV® IE-911

Batch number	Condition	Initial weight (g)	Dry Weight (g)	Water Content
1	Treated	0.5037	0.4794	4.82%
1	As received	0.5036	0.4761	5.46%
3	Treated	0.5041	0.4038	19.90%
3	Treated	0.9997	0.7967	20.31%
4	Treated	0.5025	0.4760	5.27%
4	As received	0.5031	0.4790	4.79%

APPENDIX A (continued)

5.2. Kd Measurements

The Kd and % R results from the batch equilibration tests are provided in Table 5.2. The Kd results were corrected for changes in the liquid (mL)/solids (g) ratio during the tests. In addition, the Kd vaules are based on the "dry" weight of the CST.

Table 5.2 Batch Adsorption Data for the Removal of ^{137}Cs from the Simulant of the "Average" SRS Supernatant with IONSIV® IE-911

		Mixing Time								
Batch	Condition	24 h Liq (mL)/ Solid (mg)	24 h Kd (mL/mg)	24 h % R	72 h Liq (mL)/ Solid (mg)	72 h Kd (mL/mg)	72 h % R	168 h Liq (mL)/ Solid (mg)	168 h Kd (mL/mg)	168 h % R
1	Treated	202	434	68.3	182	1377	88.3	175	2034	92.1
1	Treated	202	365	64.4	188	1712	90.1	182	2029	91.8
	(Average)		400	66.4		1545	89.2		2032	92.0
1	As Received	196	675	77.5	177	2073	92.1	171	2238	92.9
1	As Received	206	596	74.3	179	2041	91.9	172	2267	93.0
	(Average)		636	75.9		2051	92.0		2253	93.0
3	Treated	204	387	65.5	188	1153	86.0	181	1508	89.3
3	Treated	203	327	61.6	182	1162	86.5	175	1399	88.9
	(Average)		357	63.6		1158	86.3		1454	89.1
4	Treated	199	451	69.4	181	1234	87.2	175	1480	89.4
4	Treated	202	363	64.3	180	1237	87.3	173	1607	90.3
	(Average)		407	66.9		1236	87.3		1544	89.9
4	As Received	198	703	78.0	184	1530	89.2	177	2123	92.3
4	As Received	206	592	74.2	187	841	81.8	182	2175	92.3
	(Average)		648	76.1		1186	85.5		2149	92.3
4	Treated/Sieved	201	308	61.9	186	1065	85.1	177	1667	90.4
4	Treated/Sieved	201	337	60.5	185	1892	91.1	177	1578	89.9
	(Average)		323	61.2		1479	88.1		1623	90.2
4	As Received/ Sieved	205	337	62.2	178	1262	87.6	169	1818	91.5
4	As Received/ Sieved	205	546	72.7	188	1709	90.1	185	1881	91.1
	(Average)		442	67.5		1486	88.9		1850	91.3
4	Treated/ Dibutylphosphate	194	525	73.0	175	1017	85.3	168	1567	90.3
4	Treated/ Dibutylphosphate	196	393	66.7	173	1498	89.7	166	1490	90.0
	(Average)		459	69.9		1258	87.5		1529	90.2

It is important to note that three 24-h samples, which were kept in the 15-mL tubes for several days, were filtered and counted after the results from the 24-h and 72-h tests were obtained. This delay in filtration led to a very significant improvement in the Kd results for the 24-h solutions. For the "treated" batch 3, the "treated/dibutylphosphate" batch 4, and the "as received/sieved" batch 4 samples, the Kds improved from 327 to 1727 (delayed filtration), from 393 to 1928, and from 337 to 2325, respectively. The Kd values for the 24-h samples with the delay in filtration were substantially higher than the Kd values in the 168-h tests with the same samples. This dramatic improvement in Kd values is most likely due to very fine CST particles. These very fine CST particles were probably produced by particle collisions during the rocking of the samples. These particles apparently remained in solution after centrifugation at 2800 rpm. Higher speed centrifugation may be needed to remove these particles after mixing. Futher tests to evaluate this effect are underway.

6. ACKNOWLEDGMENTS

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APPENDIX B
Texas A&M Predictions of Column Performance

Simulation of Column Test for SRS Waste Simulant

By

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1. Introduction

This report details the methods and results for the simulations of three proposed SRS column experiments. The experimental column was 1.5 cm in diameter and 10 cm in length and packed with the CST UOP IONSIV™ IE-911. The operating conditions were provided in handouts from the recent meeting at Savannah River. The waste simulant is the "average" SRS waste. Three flow rates were simulated 0.25 cm/min, 1.0 cm/min, and 4.0 cm/min.

An ion exchange column model, which uses a two-phase homogeneous particle balance, linear isotherm, and accounts for dispersion, was used to predict breakthrough curves for the given flow rates. The equilibrium for the system was estimated using the equilibrium model developed by Z. Zheng (1996). Diffusion coefficient was estimated from batch experiments conducted on simplified waste solutions. Axial dispersion and film mass transfer coefficients were estimated from correlations.

2. Equilibrium

The equilibrium partition coefficient or K_d was estimated from a previously developed equilibrium model (Zheng, 1996). The waste composition was provided in a handout and was labeled the "average" simulant.

The model predicted K_d was 1570 ml/g. Savannah River data, comparing K_d experimental to K_d model for the SRS simulant, indicated that no correction was needed for the model predicted equilibrium value.

APPENDI X B (continued)

3. Diffusion Coefficient

Diffusion coefficients for standard simulant were used as estimates for the actual diffusion coefficients in the SRS waste. From batch kinetic experiments, a diffusion coefficient of 2.7×10^{-11} m^2/s was estimated for IE-911-0001 granules in standard simulant. This value was also used to model column experiments in standard simulant and provided good fits of the column data. Thus, a value of 2.7×10^{-11} m^2/s was used to model the IE-911-0001 behavior in the SRS solution.

4. Modeling Methods and Results

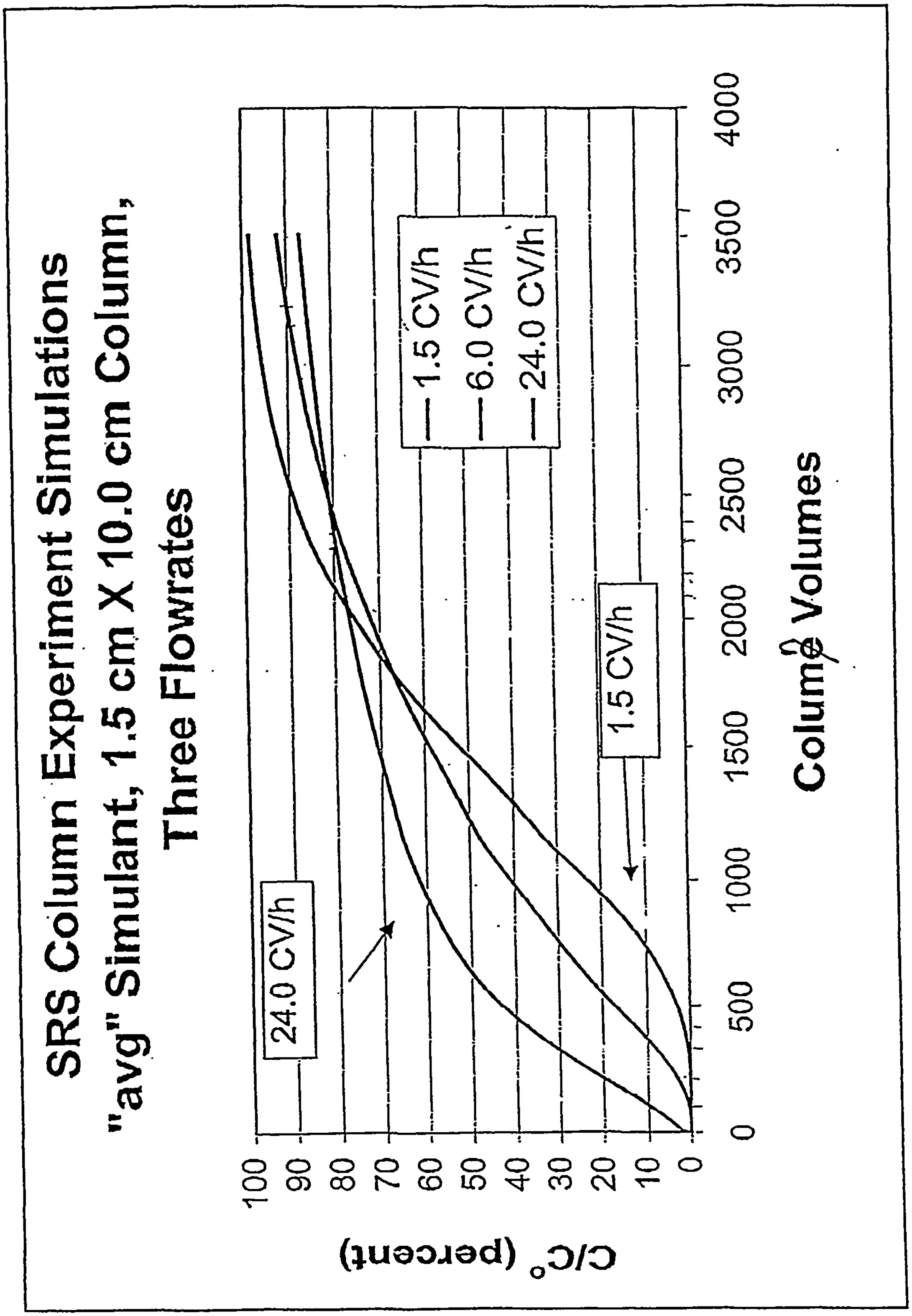
A two-phase homogeneous column model was used to generate breakthrough curves for various operating conditions for the SRS waste solution. The equilibrium K_d and diffusion coefficient were estimated as described above. The column size was given as 1.5 cm diameter and 10 cm length. The velocities simulated were 0.25 cm/min (1.5 CV/h), 1.0 cm/min (6.0 CV/h), and 4.0 cm/min (24 CV/h).

The attached figure shows the breakthrough curves for the three simulations under the given conditions. Table 1 lists the column volumes at which DF= 40,000 (or the outlet concentration is 0.0025%) for each simulation.

Table 1. Column volumes at DF = 40,000 for three column simulations for SRS waste

Velocity (cm/min)	CVs at DF = 40,000
0.25 (1.5 CV/h)	103
1.0 (6.0 CV/h)	6
4.0 (24 CV/h)	0.2

APPENDIX B (continued)



APPENDIX C
Experimental

Preparation of Salt Solutions

Researchers prepared simulated SRS waste solutions with the compositions shown in Table I using reagent grade chemicals. Tests used carrier free tracer Cs-137, >99% radionuclidic purity, obtained from E. I. du Pont de Nemours & Co., NEN Research Products, Boston, MA. Table V lists additional salt composition variations used in the K_d tests. For those solutions in which the total sodium ion concentration varied, the ratio of the anions remained fixed at the values listed in Table I. Initial cesium and potassium concentrations verification used atomic absorption spectroscopy. Personnel measured the densities of the salt solutions by weighing filled, 100-mL or larger volumetric flasks.

Crystalline Silicotitanate

These experiments used crystalline silicotitanate in the engineered form, IONSIV® IE-911, manufactured by UOP LLC., Molecular Sieves Division, Des Plaines, IL. Lot #99909681002 was used in the slow and medium speed column runs and in the majority of the K_d tests. Lot #99909681004 was used in the fast column run and in a few K_d tests. The particle size range for both lots was 30-60 mesh. The water content of the as-received particles was determined by drying 0.2 gram samples to constant weight at 105 °C. The packed density of the particles was measured by filling a 25 mL graduated cylinder with as-received particles or with particles that had equilibrated with water in the laboratory air for two days. Table C-1 lists the weight loss on drying and packed density for each lot.

K_d Tests

Researchers used 0.1 g CST in 25.0 mL of salt solution. The slurry was agitated on a shaker for 72 hours at room temperature (22±2 °C) unless otherwise specified. Following agitation, the slurry was filtered using cellulose nitrate disposable filters (0.2 micron nominal pore diameter). The filtrate was analyzed by gamma spectroscopy to determine the final cesium concentration.

TABLE C-1. Properties of CST Particles

Lot#	99909681002	99909681004
Weight loss on drying (wt %)	13.0	2.7
Packed density (g/mL)		
As received	0.96	1.01
After air equilibration	--	1.08

Column Tests

Table VI summarizes the ion exchange column dimensions and test conditions. Figure 1 shows the breakthrough curves (C/C_0) for the three column runs. Tables C-2, C -3, and C-4 list the data. Prior to use in column tests, the CST was pretreated in three stages. First, the particles were equilibrated with water vapor in the laboratory air for two days. Next, the particles were wetted with water, placed in a column, and flushed upflow to remove fines (2-3 L of water per 20 mL CST). Finally, 2.2 M NaOH solution (1 L) was pumped in a loop through the column for 24 to 48 hours. To prepare the packed columns, the apparatus was assembled and filled with water. The slurry of flushed CST particles was added dropwise, allowing the particles to sink and disengage air bubbles. The column was settled and packed by gently tapping on the side. No change in volume of the column packing was detected during column runs. The columns were fed from 2 to 12 liter reservoirs. The spent feed solution was collected, analyzed for potassium and cesium, replenished with cesium, and reused. Flowrates were determined each time the feed solution was replaced.

Analytical Methods

Analytical Development Section of SRTC performed the following analyses.

Potassium ion concentrations on aqueous samples were measured by flame atomic absorption using a Varian SpectrAA-400 spectrometer. Each sample was diluted in a suppressant solution of cesium. The method is described in Manual L16.1, Procedure #ADS-1549.

Cold cesium concentrations were measured by Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) using a VG Elemental PQ2 instrument. Samples were diluted 1000X and acidified to a pH < 2 using 0.6 wt % nitric acid. The method is described in Manual L16.1, Procedure #ADS-1553.

Tracer Cs-137 was measured by gamma spectroscopy counting methods using sample volumes ranging up to 10 mL and counting periods of up to 60 minutes. A high-purity germanium gamma spectroscopy system operated through a Canberra Genie PC software interface was used for the analysis. The method is described in Manual L16.1, Procedure #ADS-2420.

TABLE C-2 Data from Slow Column Run

Date	Time	Elapsed	Total	Column	Rate	Cs-137	K(AA)	C/Co (Cs)		Cs-137	Cs	K
		Time (hr)	Volume (L)	Volumes	(mL/h)	d/m/ml	mg/L	(%)		d/m/mL	(mg/L)	(mg/L)
1-Sep	9:24	0	0	0	28.4				New feed	3.51E+05	17.9	534
	11:02	1.6	0.045	2.5		5.4	45.4	0.0015				
	16:45	7.4	0.21	11.9		3.8	537	0.0011				
2-Sep	6:45	21.4	0.608	34.3		0.7	532	0.0002				
	16:46	30.4	0.863	48.8		<3.5	508	<.001				
3-Sep	6:47	45.4	1.289	72.8		<3.5	533	<.001				
	16:54	55.5	1.576	89.1		12	522	0.0034				
4-Sep	6:46	69.4	1.971	111.4	28.4	<2.6	654	<.001				
	16:45	79.4	2.256	127.5	28.5	<2.0	603	<.001				
5-Sep	7:52	94.5	2.686	151.8		<1.9	589	<.001				
	19:29	106.2	3.02	170.6	28.5	<2.4	592	<.001				
6-Sep	7:34	118.2	3.355	189.5	27.9	<2.9	578	<.001				
	19:38	130.2	3.69	208.5		<2.6	589	<.001				
7-Sep	8:18	142.9	4.044	228.5	27.9	1.3	581	0.0004				
	19:55	154.5	4.341	245.3	25.6	2.3	567	0.0007	New feed	3.24E+05	19.9	485
8-Sep	7:46	166.4	4.646	262.5	26.2	<3.2	696	<.001			20.1	595
	16:46	175.4	4.933	278.7		<7		<.002				
9-Sep	7:46	191.4	5.593	316	29.1	5.1	614	0.0016				
10-Sep	7:30	214.1	6.291	355.4		9.4	609	0.0029				
11-Sep	9:20	240	7.051	398.4	29.4	36.4	662	0.011				
12-Sep	7:55	263.5	7.447	420.7		70	592	0.022				
13-Sep	7:30	287.1	8.599	485.8		148	595	0.046				
14-Sep	7:20	311	8.831	498.9	29.2	277	581	0.085				
15-Sep	7:30	335.1	9.534	538.6		529		0.163				
16-Sep	7:32	359.2	10.219	577.3	29.1	829		0.256	New feed	3.68E+05	17.7	
17-Sep	13:35	389.2	11.066	625.2	28.2	1498		0.416				
18-Sep	7:22	407	11.567	653.5		2308		0.641				
19-Sep	7:50	431.5	12.257	692.4	27.9	3149		0.875				
20-Sep	8:03	455.7	12.933	730.7		4393		1.220				
21-Sep	7:20	479	13.59	767.8	27.91	6198		1.722				
22-Sep	7:15	502.9	14.257	805.5	27.9	7998		2.222				
23-Sep	8:50	528.5	14.971	845.8	27.9	10646		2.957				
24-Sep	6:41	550.4	15.561	879.2	26.96	12118		3.366				
25-Sep	8:07	575.8	16.246	917.9	26.96	16039		4.455				

TABLE C-3. Data from Intermediate Column Run

Date	Time	Elapsed Time (h)	Total Volume (L)	Column Volumes	Rate (mL/m)	Cs (mg/L)	C/Co)		Cs (mg/L)	K (mg/L)
27-Aug	10:37	0	0.000	0.0		0.03	0.002	Initial feed	19.4, 18.81	540, 559
	22:32	11.9	1.164	65.8						
28-Aug	10:27	23.8	2.328	131.5	1.63	0.06	0.003			
	22:22	35.7	4.741	267.9						
29-Aug	10:17	47.7	5.958	336.6	1.69	0.29	0.015			
	22:12	59.6	7.129	402.8						
30-Aug	10:07	71.5	8.300	468.9	1.64	0.99	0.052			
	22:02	83.4	9.513	537.5						
31-Aug	9:57	95.3	10.727	606.1	1.7	1.92	0.101			
	22:07	107.5	11.979	676.8		2.6	0.135	New feed	24.4, 17.01	511
1-Sep	10:07	119.5	13.210	746.3	1.71					
	22:07	131.5	14.441	815.9		3.5	0.17			
2-Sep	10:07	143.5	15.673	885.5	1.71	4.1	0.20			
	22:07	155.5	16.904	955.0		5.3	0.26			
3-Sep	10:07	167.5	18.135	1024.6	1.71	5.0	0.24			
	22:07	179.5	19.366	1094.1						
4-Sep	10:07	191.5	20.597	1163.7	1.71	10.0	0.48			
	22:07	203.5	21.836	1233.7		8.5	0.41	New feed	16.9, 15.9, 15.6	579
5-Sep	10:07	215.5	23.074	1303.6	1.72	9.3	0.58			
	22:07	227.5	24.305	1373.2		9.7	0.61			
6-Sep	10:07	239.5	25.537	1442.7	1.71	10.5	0.66			
	22:07	251.5	25.660	1449.7		10.9	0.68			
7-Sep	10:07	263.5	25.784	1456.7	0.172					
	22:07	275.5	27.023	1526.7		10.0	0.62			
8-Sep	10:07	287.5	28.261	1596.7	1.72					
	22:07	299.5	29.507	1667.0		12.8	0.80			
9-Sep	10:07	311.5	30.752	1737.4	1.73	13.1	0.82			
	22:07	323.5	32.005	1808.2				New feed	19.6, 19.5	522
10-Sep	10:07	335.5	33.258	1879.0	1.74	14.3	0.73			
	22:07	347.5	34.511	1949.8						
11-Sep	10:07	359.5	35.763	2020.5	1.74	14.9	0.76			
	22:07	371.5	37.009	2090.9		15.5	0.79	New feed	19.5	
12-Sep	10:07	383.5	38.255	2161.3	1.73	12.7	0.65			
	22:07	395.5	39.522	2232.9						
13-Sep	10:07	407.5	40.789	2304.5	1.76	16.6	0.85			
	22:07	419.5	42.049	2375.7						
14-Sep	10:07	431.5	43.309	2446.8	1.75	16.8	0.86			
	22:07	443.5	44.598	2519.7		14.4	0.74			
15-Sep	10:07	455.5	45.887	2592.5	1.79	15.0	0.77			
	22:07	467.5	47.161	2664.5				New feed	17.1	
16-Sep	10:07	479.5	48.435	2736.5	1.77	15.0	0.88			
	22:07	491.5	49.731	2809.7						
17-Sep	10:07	503.5	51.027	2882.9	1.8	15.4	0.90			
	22:07	515.5	52.338	2956.9						
18-Sep	10:07	527.5	53.648	3031.0	1.82	14.9	0.87			
		539.5	54.908	3102.2						
19 Set	10:07	551.5	56.168	3173.3	1.75	14.8	0.87			
	22:07	563.5	57.428	3244.5		16.2	0.95			

TABLE C-4. Data from Fast Column Run

Date	Time	Elapsed	Total	Column	Rate	Cs	C/Co		Cs	
		Time (hr)	Volume (L)	Volumes	(mL/h)	(mg/L)			(mg/L)	
21-Sep	8:05	0	0	0	6.42			New feed	17.71	
	9:06	1.02	0.393	22.2		0.342	0.0189	Control	18.39	
	12:06	4.02	1.549	87.5		0.854	0.0473			
	15:06	7.02	2.704	152.8		1.974	0.1094			
	18:06	10.02	3.860	218.1		3.072	0.1702			
	21:06	13.02	5.015	283.4		4.285	0.2374			
22-Sep	0:06	16.02	6.171	348.6		5.091	0.2820			
	3:06	19.02	7.327	413.9		6.186	0.3427			
	6:06	22.02	8.482	479.2	6.42	6.412	0.3552			
	9:18	25.2	9.712	548.7	6.53	7.07	0.4777	New feed	14.8	
	12:18	28.2	10.888	615.1		7.49	0.5061	Control	16.9	
	3:18	31.2	12.063	681.5		7.59	0.5128			
	6:18	34.2	13.238	747.9		8.84	0.5973			
23-Sep	9:18	37.2	14.414	814.3		9.94	0.6716			
	0:18	40.2	15.589	880.7		10	0.6757			
	3:18	43.2	16.765	947.2		10.4	0.7027			
	6:18	46.2	17.940	1013.6	6.53	10.8	0.7297			
	9:18	49.2	19.029	1,075.1	6.56	11.4	0.6533	New feed	16.6, 18.3	
	12:18	52.2	20.210	1,141.8		13.3	0.7181	Control	17.3	
	3:18	55.2	21.391	1,208.5		12.3	0.7049			
	6:18	58.2	22.571	1,275.2		12.8	0.7335			
24-Sep	9:18	61.2	23.752	1,341.9		12.6	0.7221			
	0:18	64.2	24.933	1,408.6		15.2	0.8223			
	3:18	67.2	26.114	1,475.4		13	0.7450			
	6:18	70.2	27.295	1,542.1	6.56	13.7	0.7851			
	9:18	73.2	28.525	1611.6	6.46	14.7	0.7332	New feed	20.5, 19.6	
	12:18	75.2	29.300	1655.4		15.7	0.7830	Control	18.8	
	3:18	78.2	30.463	1721.1		15.8	0.7880			
	6:18	81.2	31.626	1786.8		15.1	0.7531			
25-Sep	9:18	84.2	32.789	1852.5		15.9	0.7930			
	0:18	87.2	33.952	1918.2		15.5	0.7731			
	3:18	90.2	35.114	1983.9		16.2	0.8080			
	6:18	93.2	36.277	2049.6	6.46	15.9	0.7930			
	9:18	96.2	37.048	2093.1	6.53	18.2	0.8922	New feed	20.3	
	12:18	99.2	38.223	2159.5		18.0	0.8824	Control	19.2	
	3:18	102.2	39.399	2225.9		18.3	0.8971			
	6:18	105.2	40.574	2292.3		18.4	0.9020			
26-Sep	9:18	108.2	41.749	2358.7		18.9	0.9265			
	0:18	111.2	42.925	2425.1		18.3	0.8971			
	3:18	114.2	44.100	2491.5		17.8	0.8725			
	6:18	117.2	45.276	2557.9	6.53	18.3	0.8971			
	9:18	120.2	46.062	2602.4	6.61	19.5	0.9375	New feed	20.8	
	12:18	123.2	47.252	2669.6		20.0	0.9615	Control	20.2	
	3:18	126.2	48.442	2736.8		21.3	1.0240			
	6:18	129.2	49.632	2804.1		19.4	0.9327			
27-Sep	9:18	132.2	50.822	2871.3		18.8	0.9038			
	0:18	135.2	52.011	2938.5		20.8	1.0000			
	3:18	138.2	53.201	3005.7		19.0	0.9135			
	6:18	141.2	54.391	3072.9	6.61	19.4	0.9327	New Feed	20.6	
	9:18	144.2	55.177	3117.4	6.61	19.4	0.9417	Control	20.5	
	12:18	147.2	56.367	3184.6		20.2	0.9806			
	3:18	150.2	57557	3251.8		20.9	1.0146			
	6:18	153.2	58.747	3319.0		20.0	0.9709			
27-Sep	9:18	156.2	59.937	3386.2		20.6	1.0000			
	0:18	159.2	61.126	3453.5		19.7	0.9563			
	3:18	162.2	62.316	3520.7	6.61	20.7	1.0049			

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