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Summary

A pilot-scale test of a moving-bed configuration of a UOP IONSIV® IE-911 ion-exchange column was performed over 17 days at Severn Trent Services facilities. The objectives of the test, in order of priority, were to determine if aluminosilicate precipitation caused clumping of IE-911 particles in the column, to observe the effect on aluminum-hydroxide precipitation of water added to a simulant-filled column, to evaluate the extent of particle attrition, and to measure the expansion of the mass-transfer zone under the influence of column pulsing. The IE-911 moved through the column with no apparent clumping during the test, although analytical results indicate that little if any aluminosilicate precipitated onto the particles. A precipitate of aluminum hydroxide was not produced when water was added to the simulant-filled column, indicating that this upset scenario is probably of little concern. Particle-size distributions remained relatively constant with time and position in the column, indicating that particle attrition was not significant. The expansion of the mass-transfer zone could not be accurately measured because of the slow loading kinetics of the IE-911 and the short duration of the test; however, the information obtained indicates that back-mixing of sorbent is not extensive.

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Introduction

Replacement technologies for the in-tank precipitation process at the Savannah River Site (SRS) were investigated during the year 2000 and the beginning of 2001 under the direction of the Tanks Focus Area of the U.S. Department of Energy (DOE), Environmental Management Division (EM-50) (Dimenna et al. 2000; Harmon et al. 2000). The three candidate technologies were non-elutable crystalline silicotitanate (CST) ion exchange, small-tank tetraphenylborate precipitation, and caustic-side solvent extraction. Issues with the CST process included leaching of niobium (Nb) and silicon (Si) from the engineered form of CST (Krumhansl et al. 2000; Taylor and Mattus 1999, 2001; Wilmarth et al. 2000), UOP IONSIV® IE-911 (IE-911), and precipitation of aluminosilicates from the SRS simulants onto the IE-911 particles (Su et al. 2001). An investigation of the chemical behavior of leached Nb showed that precipitates of a hexameric Nb species could form and lead to plugging of the ion-exchange columns (Krumhansl et al. 2001). Initially it was thought that leaching of Si could lead to precipitation of aluminosilicates because of the large amount of aluminum (Al) in the simulant. However, studies showed that leached Si contributed little to aluminosilicate precipitation (Su et al. 2001). The simulants themselves were shown to be unstable with respect to aluminosilicate precipitation. Similar behavior of the actual wastes could lead to restricted liquid flow or, in the extreme instance, plugging of the column(s). In addition, inadvertent admission of solutions other than clarified feed to the column could potentially cause the precipitation of aluminum hydroxide, which could form a gelatinous layer and prevent liquid flow through the column.

Calculations of the heat-transfer characteristics of the baseline IE-911 ion-exchange columns clearly demonstrated that the columns could be sufficiently cooled only by liquid flow of feed or decontaminated salt solution (Lee 2001). External cooling would not be sufficient to maintain a normal processing temperature, i.e., <50°C, in the columns. Therefore, scenarios in which flow through the IE-911 ion-exchange columns was restricted or the column(s) became plugged were considered to represent a serious deficiency for this technology.

In response to the identification of this deficiency, alternative column designs that had the potential to avoid column plugging were evaluated (Yen et al. 2001). Two of the most promising designs are based on a moving bed of ion exchanger. One of these, the counter-current ion-exchange (CCIX®) column, consists of a modified Higgins loop where fresh ion exchanger is pulsed into the column from the end that feed exits the column whereas spent ion exchanger is removed from the column at the end that fresh feed enters the column. Thus, the feed and ion exchanger are moving in opposite directions through the column. It was thought that this arrangement would be beneficial for application of IE-911 because the ion exchanger would be pulsed about once per day, breaking up any clumps that might have formed. Also, stepwise movement of ion exchanger through the column could sweep with it any plug of aluminum hydroxide that might form. Therefore, a pilot-scale test of the CCIX® column was performed under conditions mimicking the flow of clarified feed and IE-911.

Severn Trent Services (STS, Tampa, Florida) has extensive experience in CCIX® technology and agreed to perform a pilot-scale study on their equipment. Personnel from STS, UOP, Westinghouse Savannah River Company, Savannah River Technology Center (SRTC), Oak Ridge National Laboratory

(ORNL), and Pacific Northwest National Laboratory (PNNL) collaborated and performed tests to evaluate the formation of aluminosilicates and aluminum hydroxide and the behavior of the mass-transfer zone (MTZ). The results of these tests are included in this report.

Test Objectives

The objectives for the STS alternative column configuration demonstration were as follows, in order of priority.

Objective 1: Demonstrate that aluminosilicate precipitation does not interfere with sorbent movement through the CCIX® column.

Several tests with IE-911 indicated that precipitation of aluminosilicate onto the IE-911 particles could interfere with unloading IE-911 from columns. The IE-911 particles became lightly bound together and did not move freely when sluicing was attempted. As pointed out by review committees, this could become a serious issue with the ion-exchange approach to cesium removal if the baseline design of three 16×5 ft columns in series were used where the lead column is loaded with ~5 MCi of Cs-137.

Thus, the moving-bed approach using the CCIX® system had to be tested under conditions at which aluminosilicate was expected to precipitate onto the IE-911 particles. The ability to move IE-911 through the column as aluminosilicate is precipitating would demonstrate that pulsing the column is sufficient to avoid the formation of clumps, to eliminate them after they have formed, or to move them through the column as an intact unit.

Previous experiments showed that aluminosilicate precipitates rather quickly from SRS-average simulant in the presence of IE-911 (Taylor and Mattus 2001). Therefore, a three-week test was planned to pass SRS-average simulant through a pilot-scale CCIX® column packed with IE-911. Samples of the IE-911 would be taken from the end of the column where simulant enters and at points along the column. These samples would be analyzed for precipitated aluminosilicate to assess the amount of aluminosilicate that precipitated. As the IE-911 was removed from the end of the column where simulant entered, it would be replaced by fresh IE-911 at the end of the column from which simulant was removed.

Objective 2: Demonstrate that aluminum-hydroxide formation does not produce plugging from which it is impossible to recover.

At least one incident of column plugging after a column test of IE-911 was reportedly caused by inadvertent admission of water to the column with subsequent formation of aluminum hydroxide and the inability to sluice IE-911 from the column (Walker 2000). However, subsequent attempts to reproduce the conditions that led to this plugging have been unsuccessful (Stallings et al. 2001). As pointed out by the Technical Advisory Team to DOE EM-50, such plugging could produce a situation where heat transfer is hindered and a fully loaded 16×5 ft column could overheat with catastrophic consequences.

Thus, the CCIX® approach had to be tested to determine if a "worst-case" plug of aluminum hydroxide caused by an inadvertent admission of water could be removed from the column by pulsing. The ability to remove such a plug combined with an engineered exclusion of water from the system would provide high confidence in process recovery from all credible plugging scenarios.

Following the lead of the incident when a plug of aluminum hydroxide was formed, water instead of simulant would be fed into the CCIX® column after all other tests were completed. It was hoped that this would form a plug of aluminum hydroxide at some point in the column. If such a plug were formed, the column would be pulsed to determine if the bed could be moved under such conditions.

Objective 3: Determine the rate of attrition of IE-911 in the CCIX® column.

One issue with the moving-bed approach to ion-exchange removal of Cs-137 is the ability of IE-911 to remain intact as it is pulsed through the 22-ft column. Movement of IE-911 as it is loaded and pulsed through CCIX® could cause attrition. In addition, studies of IE-911 have demonstrated that the zirconium hydroxide binder is affected by the highly caustic simulant so that embrittlement is possible (Nyman et al. 2001). As a result, the physical behavior of IE-911 as it is pulsed through the CCIX® column had to be studied. The particle-size distribution of the IE-911 would be examined to provide data on the integrity (attrition) of the particles as a function of the number of pulses.

As mentioned above, samples of the IE-911 would be removed from the CCIX® column and from several sampling points. Those portions of IE-911 located nearest to the pulse vessel at the beginning of the test or added in the first few days would be of particular interest because they would experience the maximum number of pulses, ~21. Those portions of IE-911 that were farthest from the pulse vessel at the beginning of the test or were added near the end of the test experienced the fewest number of pulses and would be of minor interest as far as particle-size distribution was concerned.

Objective 4: Characterize the MTZ.

Models of the MTZ are well behaved for the fixed-bed columns of the baseline design. However, movement (pulsing) of the bed in the CCIX® option was expected to impact the wave front (and MTZ) due to back mixing. The magnitude of this effect must be determined to estimate the column length needed to contain the entire MTZ and to provide a certain length of guard column.

Thus, samples of simulant and IE-911 would be collected at given intervals and at given times along the CCIX® column. The samples would be analyzed for cesium content to determine how the MTZ established itself in the initial three weeks of operation. It should be noted that model calculations indicated that the CCIX® column would not approach equilibrium operating conditions due to the limited duration of this test. In addition, the movement of IE-911 through the column would be expected to complicate interpretation of the results. The key objective therefore would be to clarify somewhat the pattern of movement of IE-911 particles through the CCIX® column. This would be qualitatively accomplished by using a bolus (~450 mL) of IE-911 "labeled" with Fe to give it a brownish color. This bolus would be placed into the column near the end with the pulse vessel. The extent to which this bolus dispersed as it traveled through the column would be monitored by visual observation.

Experimental

UOP produced and delivered IE-911 (~80 lb). The IE-911 was prepared according to the procedures that were used to prepare the IE-911 caustic-washed pre-production (Pre-Prd-CW, UOP #MH9098-9) batch. This procedure is called the revised manufacturing process and removes >90% of the leachable Nb and >50% of the leachable Si from the material (Wilmarth et al. 2001) in order to reduce the likelihood of plugging due to precipitation of leached Nb and aluminosilicates formed with leached Si. Most (~78 lb) of the IE-911 was shipped to STS. Small samples (~1 lb each) were sent to SRTC (as a reference material) and ORNL (as a reference material and for Fe labeling).

The demonstration was performed at the STS facilities in Tampa, Florida. STS set up the column and performed tests designed to meet the objectives of the demonstration. These tests were designed to determine the risk of aluminosilicate plugging, IE-911 particle attrition, and aluminum-hydroxide plugging. In addition, STS purchased chemicals, pretreated IE-911, formulated and dispositioned simulant, operated the equipment, shipped samples, and wrote a report on the demonstration.

Column Set-Up

A CCIX® pilot-scale column was set up according to the diagram in Figure 1.

The column was ~22 ft long and ~2 in. in diameter. Sampling points in the column were separated by at most 48 in. The sampling points provided the capability to withdraw both liquid and solid samples. In addition, there were sampling points for the simulant feed, "spent" sorbent, sorbent wash zone, and effluent simulant.

The column was packed with pretreated IE-911. UOP supplied the pretreatment procedure to STS. ORNL supplied a bolus of Fe-labeled IE-911 that was pretreated and placed in the column near the end with the pulse vessel. STS set up two 2-in.-Ø columns for backwashing fines out of the IE-911 prior to it being charged into the CCIX® column. The initial charge of sorbent was pretreated with NaOH (2 N) in the column. Sorbent added to the column after start-up was pretreated with NaOH in the smaller columns.

The $NaNO_3$/NaOH simulant was prepared in 200-gal batches using the recipe in Table 1. The simulant was filtered (~0.5-μm polypropylene) when transferred to a 50-gal feed tank and before it was pumped to the column.

Testing

Two testing phases were performed. During the initial ~18 days (during the first 2 days the column was not pulsed), average simulant was processed through the CCIX® column. The column was operated according to procedures defined by STS. The column was pulsed approximately once per day so that the sorbent bed moved ~12 in. during the plugging test as described below in Sorbent Pulsing. This portion of the test was used to quantify the aluminosilicate solids formation.
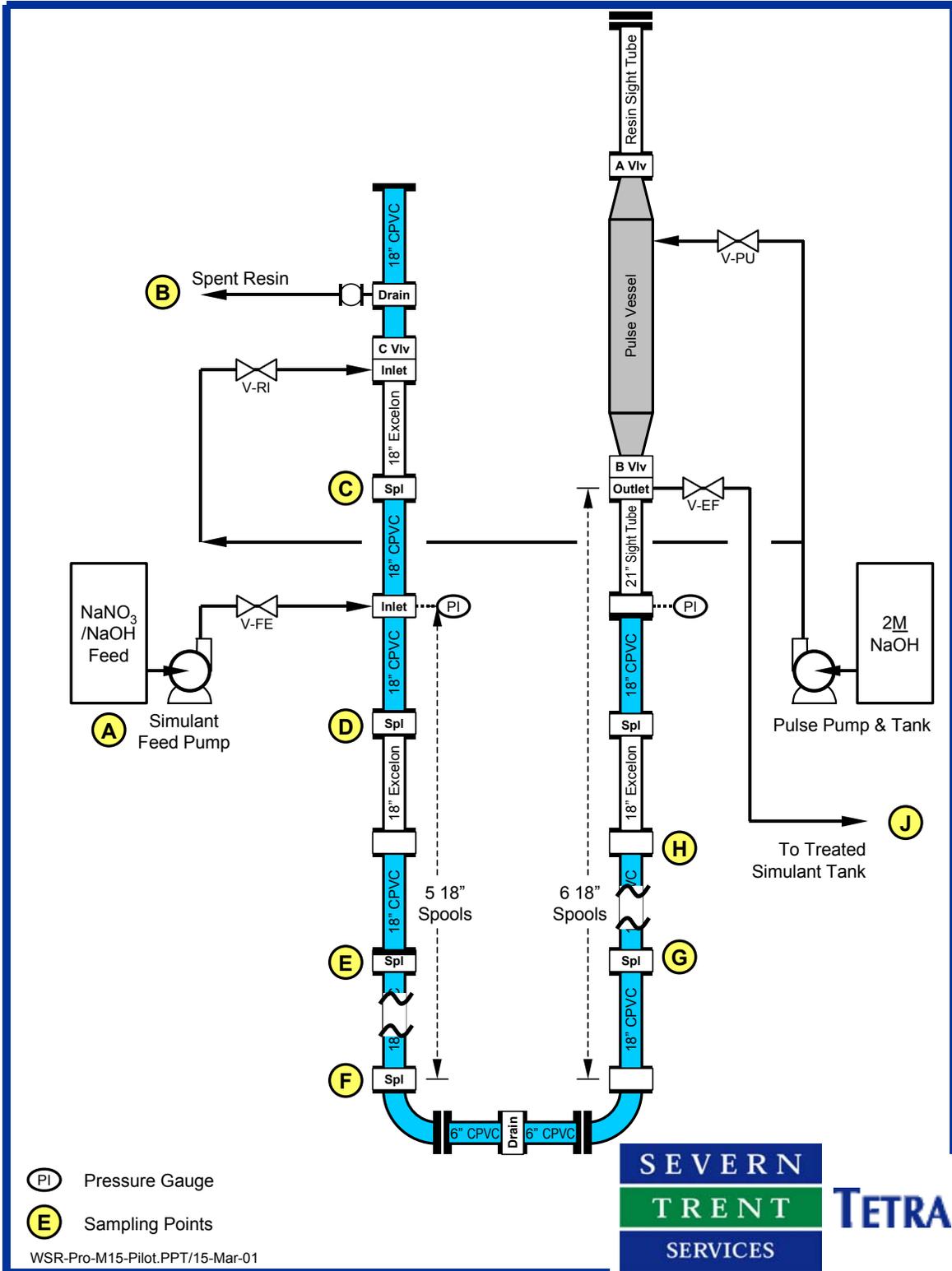
Figure 1. CCIX™ Continuous I-IX System Pilot Unit Configuration

Table 1. Formulation for SRS Average Simulant

Step No.	Chemical/Action	Formula	250-Gal Amt.	170-Gal Amt.	lbs
1	Deionized (DI) water	H_2O	600 L	408 L	
2	Agitate the liquid				
3	Caustic soda	NaOH	119.23 kg	81.08 kg	178.8
4	Aluminum nitrate hydrate	$Al(NO_3)_3 \cdot 9H_2O$ (add slowly)	110.03 kg	74.82 kg	165.0
5	Mix for 1 h				
6	Sodium carbonate	Na_2CO_3	16.05 kg	10.91 kg	24.1
7	Sodium sulfate	Na_2SO_4	20.16 kg	13.71 kg	30.2
8	Trisodium phosphate	$Na_3PO_4 \cdot 12H_2O$	3.48 kg	2.37 kg	5.2
9	Sodium oxalate	$Na_2C_2O_4$	0.51 kg	0.35 kg	0.8
10	Sodium silicate	$Na_2SiO_3 \cdot 9H_2O$	1.07 kg	0.73 kg	1.6
11	Sodium molybdate	$Na_2MoO_4 \cdot 2H_2O$	0.54 kg	0.37 kg	0.8
12	Sodium chloride	NaCl	1.36 kg	0.92 kg	2.0
13	Sodium fluoride	NaF	1.27 kg	0.86 kg	1.9
14	Potassium nitrate	KNO_3	1.43 kg	0.97 kg	2.1
15	Cesium chloride	CsCl	25.80 g	17.54 g	17.5 g
16	Sodium nitrate	$NaNO_3$	96.10 kg.	65.35 kg	144.1
17	Sodium nitrite	$NaNO_2$	33.95 kg	23.09 kg	50.9
18	Fill with DI water to make 946 L (250 Gal)		946 L	643 L	
19	Mix solution for 1 h				

The second phase of the test involved short-duration operation using water as the feed to evaluate aluminum-hydroxide precipitation. This part of the test evaluated the capability of the CCIX® system to continue operation and to deinventory the IE-911 as needed to maintain or reestablish simulant flow. This involved repeated pulsing during which liquid flow and pressure were monitored.

Sampling

To identify trends associated with sampling, it was important that the timing from day to day be consistent. This included the pulsing process since the effect of this disruption needed to be better understood. All samples were numbered so that the times at which they were collected could be related to the time at which the pulses occurred. To aid in the understanding of sample schedule, amounts, and disposition for analysis, a Sample Plan was developed as a spreadsheet (Table 2). Phase 1 samples are defined and were taken according to the schedule described below.

Table 2. STS/DOE CCIX® Test Sampling Plan

Phase 1 (Aluminosilicate precipitation test)									
Sampling Schedule for Days 1,[(a)] 2, 3, 14, 15, 16									
	Time, h	Absorption Zone Sampling Points					Spent IE-911	Simulant Feed	Simulant Effluent	Wash Zone
Sample ID[(b)]		D	**E**	**F**	**G**	**H**	**B**	**A**	**J**	**C**
Daily, one taken after pulse	6	S,S/L	S,S/L	S,S/L	S,S/L	S,S/L			L	
	12	S,S/L	S,S/L	S,S/L	S,S/L	S,S/L			L	
	18	S,S/L	S,S/L	S,S/L	S,S/L	S,S/L			L	
	24	S,S/L	S,S/L	S,S/L	S,S/L	S,S/L	S	L	L	L
Sampling Schedule for Days 4-13										
		Absorption Zone Sampling Points					Spent IE-911	Simulant Feed	Simulant Effluent	Wash Zone
Sample ID[(b)]		D	**E**	**F**	**G**	**H**	**B**	**A**	**J**	**C**
Daily after pulse		S,S/L	S,S/L	S,S/L	S,S/L	S,S/L	S	L	L	L
Phase 2 (Aluminum-hydroxide precipitation test)										
Sample Schedule for Day 17										
		Absorption Zone Sampling Points					Spent IE-911	Simulant feed	Simulant effluent	Wash zone
Sample ID[(b)]		D	**E**	**F**	**G**	**H**	**B**	**A**	**J**	**C**
12-hour		S,S/L	S,S/L	S,S/L	S,S/L	S,S/L	S	L	L	L
(a) Day 1 is considered that day on which pulsing began, which was actually the third day after simulant was introduced to the column. (b) Sample types: Solid (S, 11 mL of solids with liquid), solid and liquid (S/L, 11 mL of solid with 25 mL of liquid above solid), and liquid (L, 25 mL).										

Simulant Feed Samples. One sample "A" of a minimum of 25 mL was taken each day and when a new feed batch was put on line. Feed samples were stored in 125 mL bottles and were pulled from the feed tank, not the mix tank.

Absorbent MTZ (Column) Samples. Four sets of samples were scheduled to be taken from each of the five sample points (D, E, F, G, and H) every day for the first three and last three days of the Phase 1 part of the test. One set of samples (5 points) was taken after the daily pulsing event at 1900 hours. All four sets of samples were spaced at 6-h intervals—0100, 0700, 1300, and 1900 hours. For days 4 through 17, one sample would be taken from each of the five sample points once per day. However, approximately four days after the Phase 1 test began, sample port G no longer yielded samples and was taken out of service.

Samples from each location (D, E, F, G, and H) were taken as follows. Solid and liquid were flushed from the sample port (25 mL) into a 125-mL bottle labeled "Slurry" with the appropriate letter. The bottle was rinsed with the flushed solid and liquid, which were discarded. Then, solids with liquid (11 mL) were collected in the rinsed bottle.

Samples of each point (D, E, F, G, and H) with solids and liquid (12 mL) and a minimum of 25 mL of liquid above the solids were collected in centrifuge tubes rinsed with 1 M NaOH. Both levels were marked on the tubes. The samples were processed as described below in the "Centrifuge Method."

Spent Absorbent Samples. One sample of spent sorbent "B" with a minimum of 11 mL of solid with liquid was collected for each pulse event at 1900 hours. During the aluminosilicate plugging test, this amounted to one sample per day.

Simulant Effluent Samples. For the first three and last three days, four samples of simulant effluent "J" with a minimum of 25 mL per day were taken at the same time as the absorbent MTZ (column) samples. For days 4 through 17, one sample with a minimum of 25 mL was taken at the same time as the absorbent MTZ (column) samples.

Wash Zone Sample. One sample of liquid "C" with a minimum of 25 mL was taken each day after the pulse.

Sample Preparation

Centrifuge Method. Samples of IE-911 (~12 mL) and simulant (~25 mL) were collected in a plastic centrifuge tube from a sample point. A set of filled centrifuge tubes was weighed by taring a 125-mL beaker on a top-loading balance and setting a tube and lid on the scale to ensure that the system was balanced. Extra liquid was pulled from the tube with a plastic sampler so that its weight matched that of the tube opposite it in the centrifuge (±0.2 g). The set of tubes was centrifuged at 2600 rpm for 4 min, after which the supernates were clear. The supernates were withdrawn and saved as the "Centrate" samples in 125-mL bottles. The contents of the tubes were rinsed with 1 M NaOH (15 to 20 mL), weighing each one as noted above to ensure that the system was balanced. The tubes were agitated to mix the solids and liquids. The tubes were centrifuged until the supernates were clear. The supernates were withdrawn and discarded. Deionized water (15 to 20 mL) was added to the tubes, weighing each one to ensure that the system was balanced. The tubes were agitated to mix the solids and liquids. The tubes were centrifuged until the supernates were clear. The supernates were withdrawn and discarded. The water rinse was repeated. The supernates were withdrawn and discarded. Using a funnel, spatula, and deionized water, the solid (IE-911) was separated into one sample bottle labeled "ORNL" (~1 to 2 gm) and one sample bottle for SRTC (the remainder of the IE-911). Each sample was placed in a 30-mL bottle. The centrifuge tubes were rinsed of solids with deionized water and 1 M NaOH.

Shipping

All samples were shipped from STS to SRTC and ORNL via express mail. STS enlisted the services of a person qualified to ship Dangerous Goods so that all applicable regulations were met. All sample bottles were sealed with Teflon® tape.

Analyses

SRTC analyzed liquid samples for Cs content using inductively coupled plasma mass spectrometry (ICP-MS) and Al, Si, etc., content using inductively coupled plasma atomic emission spectroscopy (ICP-AES) and the solids for particle-size distribution. ORNL analyzed solid samples for Al, Si, etc., content (destructive dissolution followed by ICP-AES). All samples were received at SRTC and ORNL and archived for possible subsequent analysis.

Simulant feed samples (liquid) and absorbent MTZ samples (liquid) from days 7, 10, 13, 16, and 17 and absorbent MTZ samples (solid) from days 1, 4, 7, 14, and 17 were analyzed for Cs, Al, Si, etc., content. Spent absorbent samples and absorbent MTZ samples (solid) from days 1, 4, 7, 10, 13, 16, and 17 were analyzed for particle-size distribution. In addition, liquid samples from day 1 were analyzed for particle-size distribution.

Simulant effluent samples were collected, inspected for solids, and archived for possible future analysis of Cs and Al, Si, etc., content. Wash zone samples were separated into liquid and solid phases and archived for possible subsequent analysis.

The ICP analyses at ORNL were performed using a model 61E Trace ICP from Thermo Jarrell Ash, following standard U.S. Environmental Protection Agency method SW846-6010B. Standard reference samples from the National Institute of Standards and Technology were analyzed along with the test samples to verify the accuracy of the results. The IE-911 samples were digested using nitric and hydrofluoric acids in a microwave oven to solubilize the materials; then boric acid was added to complex the fluoride ion prior to analysis.

Sorbent Pulsing

Every 24 h, the sorbent bed was advanced, or pulsed, 12 in. through the CCIX® column. Pretreated sorbent (~550 mL) was added to the pulse vessel prior to this event. The simulant feed flow was stopped. Loop valves B and C were opened. The sorbent in the pulse vessel was moved hydraulically into the adsorption vessel using 2 N NaOH as the conveying media. Sorbent displaced from the vessel above C valve was collected in a bucket.

Aluminum-Hydroxide Formation

The aluminum-hydroxide-formation test was run by feeding water instead of simulant to the column and observing the pressure. The water contained a dye (blue food coloring) that was intended to make it easily distinguishable from simulant that was already in the column. Two scenarios were considered.

In scenario 1, the column pressure would remain <60 psig. The value of 60 psig was chosen because that is the pressure at which the pump would be stopped. In this instance, samples of the slurry and liquid would be taken from sampling points H, F, E, and D when the interface passed the sight tube just below sampling point H. After collection, samples would be washed with water only in preparation for shipment. Water would be pumped into the column until the water/simulant interface reached the sight tube above sampling point H. At this time flow through the column would be stopped for 4 h. The interface would be observed during this time. The flow would be started again.

The pressure could also remain normal (22 to 25 psig). A sample would be taken from sampling point H and washed with water. Water would be pumped through the column and out through the effluent line to flush it completely. The column would then be pulsed twice.

In addition, the pressure could increase or already be high (but <60 psig). The pressure would be allowed to stabilize. Flow would be stopped. The column would be pulsed to move the plug past the U-bend at the bottom of the column using spent, water-washed IE-911 collected from the previous week. Samples would be taken from the spool piece that contains the plug and from the spent sorbent. The pulse system inventory would be replaced with water that would be used for the pulses.

In scenario 2, the pressure rises above 60 psig or flow stops. If the pressure rises above 60 psig, the pump would be stopped. The pump would also be stopped if flow through the column stopped. The column would be pulsed using 2 M NaOH and recycled IE-911 until the plug was visible in the sight tube near port D or C. The sight tube that contained the plug would be removed. A sample of the plug would be obtained for analysis.

Waste Disposal

STS collected all liquid and solid waste in appropriate containers. The services of a qualified waste-disposal company were enlisted so that the waste disposal was performed according to regulations. STS supplied PNNL with documentation of the waste disposal.

Results

Aluminosilicate Test

The IE-911 showed no indication of clumping during the course of the test when it was pulsed through the CCIX® column. At the start of the test, the column was loaded with IE-911, charged with simulant without Cs or oxalate, and left to stand for two days because of a shortage of materials to formulate enough simulant to start pulsing immediately. It was considered that the presence of aluminum and silicon in the simulant would be sufficient to initiate the aluminosilicate precipitation process. Samples were numbered starting from the day that regular pulsing was started, i.e., about two days after the column was charged with simulant.

The analysis of IE-911 particles taken at various sampling points along the CCIX® column was intended to demonstrate the presence of precipitated aluminosilicate on the IE-911 particles. Table 3 gives the results of those analyses for Al and Si. Complete results for analyses of all elements can be found in Appendix A. It can be seen that the highest amount of aluminum and silicon on the particles occurred on the first day after the column was started. This result is not consistent with the kinetics of aluminosilicate precipitation from SRS simulants that have been observed in the past, according to which aluminosilicates are first observed after about one month. It probably indicates that aluminosilicate had precipitated in the simulant after it was formulated and before it was pumped through the column. As the simulant was pumped through the column, the aluminosilicate was entrained with the IE-911 particles and incorporated in the sample that was analyzed. It would have to be assumed that the aluminosilicate was then washed out of the column during the next few days. If this did indeed occur, then the fact that the IE-911 could be moved through the column even with entrained solids indicates that the moving-bed technology may be advantageous for processing actual wastes.

Table 3. Analysis of IE-911 Particles for Precipitated Al and Si

Days of Testing	Al and Si in Solid IE-911 (µg/g) from Sampling Points (Figure 1)							
	D		E		F		H	
	Al	Si	Al	Si	Al	Si	Al	Si
1	1400	146955	1563	167181	1147	129851	1388	92794
4	818	77908	806	79276	955	78215	1016	78980
7	768	78376	904	76148	878	77980	634	70582
14	1138	79140	1141	76156	1073	76944	1164	78814
17	988	80271	1050	80473	1088	80215	907	82579

The ratios of Al and Si to Ti in the sample give another indication of the amount of aluminosilicate in the sample. This occurs because the amount of Ti is determined by the amount of CST present in the particles whereas the amounts of Al and Si are affected by the presence of precipitated aluminosilicates. Table 4 shows the results for the Al/Ti and Si/Ti ratios calculated from the analytical data. It can be seen

that the ratios are highest for the 1-day sample. This again suggests that the initial sample contained a precipitate of aluminosilicate although the precipitation kinetics would suggest that this precipitate formed in the simulant and did not precipitate out on the IE-911 particles. Also, the Si/Ti ratios except for the 1-day samples have values of 0.50 ± 0.03. This is close to the ratio that is expected from the empirical formula for CST and was found in previous analyses of IE-911 (Nyman et al. 2001). This suggests that aluminosilicate precipitation onto the IE-911 particles was not significant during the course of the test.

Table 4. Al/Ti and Si/Ti Ratios for IE-911 Samples

| | Al/Ti and Si/Ti Ratios in Samples from Sampling Points (Figure 1) | | | | | | | |
| | D | | E | | F | | H | |
Days of Testing	Al/Ti	Si/Ti	Al/Ti	Si/Ti	Al/Ti	Si/Ti	Al/Ti	Si/Ti
1	0.0089	0.94	0.0099	1.06	0.0072	0.82	0.0090	0.60
4	0.0052	0.50	0.0051	0.50	0.0061	0.50	0.0064	0.50
7	0.0048	0.49	0.0059	0.50	0.0055	0.49	0.0042	0.47
14	0.0072	0.50	0.0073	0.49	0.0070	0.50	0.0074	0.50
17	0.0064	0.52	0.0068	0.52	0.0071	0.52	0.0058	0.53

Aluminum-Hydroxide Precipitation Test

Only qualitative results were obtained from the test of aluminum-hydroxide precipitation. After completion of the aluminosilicate precipitation test, simulant was replaced by water as the feed. The water was colored with blue food coloring so that it could be distinguished from the simulant that was present in the column. On the downward-flowing side of the column, the water replaced simulant quite distinctly as judged from samples collected from sampling point D. However, the replacement was not as distinct on the upward-flowing side, probably due to more efficient mixing of the comparatively dense (1.25 g/mL) simulant with the lighter water (1.0 g/mL). Thus, flow was stopped when it was certain that water had reached the sight glass closest to the pulse vessel as judged from the coloration and specific-gravity measurements of samples withdrawn from sampling point H. The pressure in the column and the liquid flow rate remained constant during the entire process. An aluminum-hydroxide plug did not form after the column was left under static conditions for several hours, as judged by the lack of pressure build-up when the flow was again started. Sorbent was successfully pulsed twice after the test was completed. However, a third attempt to pulse sorbent through the column was unsuccessful due to equipment failure. It was later demonstrated that the equipment failure was in no way connected with plugging or other properties of the sorbent because the column could be pulsed normally five days after this occurrence.

Attrition Test

Results from the attrition test are based on the measured particle-size distributions of various samples. Table 5 lists selected manifolds of mean particle diameters and the percentages of particles in those manifolds for samples analyzed using a Microtrac SRA150 instrument. Additional data related to the

particle-size distributions can be found in Appendix B. The results show that the distributions remained fairly constant during the test. The mean particle diameters for the largest percentage of particles lie in the range 380 to 450 µm, which is typical of fresh IE-911 (Odom 2000). Two anomalous results were obtained for samples 10-24-F and 13-24-D, where large percentages of the particles had mean diameters of ~2.5 and ~2.0 µm, respectively. The reasons for these results are unknown. All mean particle diameters are within one standard difference of each other, although the relative percent differences tend to be comparatively large.

Particle-size distributions of the solids suspended in liquid samples from the first day were also measured. They typically have a large fraction of the mean particle diameters in the 1 to 2-µm range although diameters in the 6 to 7-µm range are also common.

The results indicate that no particular trend in particle attrition was observed during the test. If the particles were undergoing degradation due to splitting or erosion, then the particle-size distribution of the particles near the end of the column where the simulant enters (D and E) would have a smaller mean diameters than those of freshly added particles (F and H). Such a trend was not observed. In addition, fines generated by the degradation would show up somewhere along the column. We observed no significant differences in the mean diameters of particles throughout the column. Only in some isolated instances (10-24-B, -D and –F; 13-24-D; and 17-12-D and -H) were more than 10% of the particles less than 380 µm in mean diameter.

Table 5. Particle-Size Distributions (µm) of IE-911

Samples of Day	Distribution Manifolds[a]			
	Diameter (µm)	Percentage	Diameter (µm)	Percentage
1	416-447	96-98	114-138	2-4
4	397-424	94-97	103-126	2-5
7	372-437	90-100	78-138	1-10
10	386-454	76-95	66-141[b]	3-13
13	399-419	62-94	102-129[b]	3-7
16	386-423	92-98	101-130	2-4
17	400-435	84-96	95-137	4-9
(a) Diameter lists the ranges of mean particle diameters in a given manifold for samples from points B, D, E, F, and H on the given day and the ranges of percentages of the total particles in the manifold.				
(b) Samples 10-24-F and 13-24-D had manifolds with mean diameters of ~2.5 (19%) and ~2.0 µm (33%), respectively. The reasons for these anomalies are unknown.				

MTZ Test

Results from this test were expected to be very difficult to interpret because the ion-exchange bed was pulsed once per day. Pulsing the column was expected to disrupt the equilibrium that had set up between the simulant and sorbent. An attempt was made to estimate qualitatively the extent to which the IE-911

particles were spread out during the pulsing by adding a bolus of particles "labeled" with Fe to give them a brown color. However, the initial column packing included an air bubble trapped in the IE-911 bed. When the initial pulses occurred with the bubble in place, the air bubble contracted under pressure and expanded rapidly when the pressure was released. This caused rapid partially fluidized movement in the portion of the bed that included the bolus. Since significant mixing occurred simply because of the initial packing irregularities, the bolus was not a good indicator of the extent of back mixing during pulsing. However, two important reference points were noted. First, the bolus expanded from ~7 in. to ~21 in. after 22 to 25 pulses. In our opinion, this is considered a very moderate mixing. Second, even with air bubbles in the bed, the back mixing was not significant enough to prevent the column from being used. In fact, it was noted that the air bubbles were easily eliminated from the column upon restoration of flow after pulsing and the bed became continuously packed.

Table 6 gives the results from Cs analysis of the simulant collected from sampling points D, E, F, and H. Additional data for analysis of Cs and other elements in the liquid samples can be found in Appendixes C and D. The performance of the CCIX® column for Cs removal is seen to be rather effective in the first few days. The amount of Cs in the simulant decreases sharply from the initial concentration of about 2.1×10^4 µg/L as the simulant moves through the column and passes sampling points D, E, F, and H, in that order. This is in agreement with the shape of the MTZ calculated using VERSE model that has been developed at SRTC. According to this model, the Cs concentration drops sharply enough that intermediate concentrations would not be seen unless the wave front happened fortuitously to coincide with one of the sampling points.

Unfortunately, the data from day 16 are difficult to interpret. The reason for the relatively low values of Cs at the earlier sampling points is unknown but may be connected with the position of the MTZ in the column. The position of the MTZ would depend on both the rate of absorption and rate of movement. If these factors were adjusted so that the rate of absorption was slower than the rate of movement through the column, then the MTZ could effectively be pushed out of the column so that the simulant would encounter fresh sorbent as soon as it entered the column. This would give the observed result.

Table 6. Cs Analysis from CCIX® Column

Days of Testing	Cs in Simulant (µg/L) from Sampling Points (Figure 1)			
	D	E	F	H
7	563	69.9	49.9	34.5
10	2170	2160	313	76.7
13	4700	1430	370	63.6
16	676	437	272	108

Conclusions

The results of the tests suggest that moving-bed approaches are very promising for application to treatment of wastes where precipitation or entrainment of solids is expected to interfere with the performance of fixed-bed columns. The simulant used, SRS Average, is known to be unstable with respect to aluminoprecipitate precipitation. Although the duration of the test was apparently not long enough to enable significant quantities of aluminosilicate to precipitate onto the IE-911 particles, there was evidently a measurable quantity of aluminosilicate that precipitated out from the simulant before it was placed on the column, became entrained in the sorbent, and was then washed out of the column. This is supported by the observation of a gray solid in the simulant at the very start of the test. The gray solid was analyzed by energy dispersive spectrometry and found to consist of sodium aluminosilicates. Results from a test of longer duration should be interesting because the residence time of the IE-911 in the column could be increased so that measurable amounts of aluminosilicate could precipitate onto the IE-911 particles.

Results from the aluminum-hydroxide test were consistent with a study of aluminum hydroxide precipitation from SRS simulant (Stallings et al. 2001). Thus, pumping water into the column filled with simulant under no conditions produced a precipitate of aluminum hydroxide. The pressure and flow rate remained at the values expected when water replaced the simulant, i.e., the pressure dropped slightly due to a reduction in viscosity. Therefore, inadvertent admission of water to an ion-exchange column filled with actual waste is not expected to represent a significant concern for column operation. Although thermodynamic and kinetic calculations of such complicated salt solutions as simulant and high-salt waste do predict the precipitation of aluminum hydroxide (gibbsite) upon dilution with water (Weber 2001), the inability to reproduce the anecdotal column plugging that was observed upon addition of water to a column filled with simulant (Walker 2000) prompted us to examine other possible causes for the rapid precipitation of aluminum hydroxide. Thus, it was observed in separate experiments that addition of an acidic solution (0.1 M) to SRS average simulant can cause the immediate precipitation of aluminum hydroxide. Equilibrium expressions for the simple aluminate—hydroxide system, i.e., $[Al(OH)_4^-]/[OH^-]$ = constant, show that dilution by water reduces the concentrations of both hydroxide and aluminum at the same rate, resulting in the inability to precipitate aluminum hydroxide, whereas dilution by acid reduces the pH faster than the aluminum concentration and rather quickly produces conditions under which aluminum hydroxide precipitates.

Particle-size distributions for samples taken along the column indicate that particle attrition was not extensive during this relatively brief test. In instances where particles smaller than 380 μm were observed, the distribution was bimodal, although the overwhelming majority of the particles were typical of starting IE-911, indicating that the IE-911 particles largely remained intact. If erosion or splitting of the particles did occur, then the degradation occurred mainly through generation of fines rather than cleavage of particles into halves, thirds, or other relatively large fragments.

Issues related to the dynamics of the MTZ within the column remain unresolved. The determination of the behavior of the MTZ was the objective of lowest priority. Therefore, only qualitative observations were made. The results for the first 13 days of column operation were rather consistent with gradual

saturation of the IE-911 with Cs. However, the result for the 16-day sample suggests that the MTZ was completely expelled from the column so that fresh sorbent was located at the end of the column where fresh simulant entered. The interplay between the rate at which the MTZ is established in the column, the rate at which IE-911 moves through the column, and the extent to which the IE-911 diffuses as it passes through the column obviously plays an important role in determining the length and position of the MTZ. A quantitative and perhaps even qualitative determination of these parameters is beyond the scope of this initial study of the CCIX® column.

In addition, the column was modeled prior to the test as a fixed column. The MTZ was examined from start-up to equilibrium. It was found that the test would run only about 25% of the time needed to reach equilibrium. The MTZ was predicted never to be greater than 8 ft in length at the end of the test period even without pulsing. It was also predicted that it would take nearly the entire test period to load the first foot of IE-911. With pulsing, the MTZ was expected to always look very short, between 3 and 8 ft in length. Since the column was pulsed 1 ft every day of the test, the IE-911 removed from the column was hardly loaded at all. This was reflected in the Cs analyses, which showed that only the first few feet at most were saturated with Cs. The fact that even the first sampling point (D) was not saturated on day 16 indicates that the leading edge of the MTZ may have been pushed out of the column. Thus, the experimental and modeling results clearly indicate that further tests of longer duration are needed to define clearly the relationship between the IE-911 loading kinetics and the pulsing rate, both of which substantially influence the MTZ.

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Appendix A

ICP-AES of Solid ID-911

Sample[b]	Concentration in Solid IE-911 Samples (mg/kg)[a]										
	Al	Ca	Cu	Fe	K	Mg	Na	Nb	Si	Ti	Zr
1-24-D	1400	3526	1614	4468	5819	1709	98259	119661	146955	156630	95614
1-24-E	1563	2976	2042	4425	5872	1657	97950	120898	167181	157884	97044
1-24-F	1147	2218	2011	4441	5697	1570	97091	120984	129851	158329	97548
1-24-H	1388	3155	1909	4873	5571	1664	98601	119149	92794	154849	95410
4-24-D	818	2818	1130	2204	5317	1622	92245	119360	77908	156776	93835
4-24-E	806	3042	1368	2794	5358	1648	91014	119872	79276	157255	95306
4-24-F	955	2008	1574	3210	5424	1531	93003	118832	78215	156010	94848
4-24-H	1016	1684	1608	3557	5515	1498	94051	119725	78980	157494	96208
7-24-D	768	1998	890	1725	5203	1546	90364	120665	78376	159050	96134
7-24-E	904	1790	713	1496	5062	1478	89027	115800	76148	152311	93011
7-24-F	878	1877	770	1675	5401	1564	93050	121725	77980	159318	97952
7-24-H	634	910	552	1283	5157	1267	88795	86709	70582	149250	92311
14-24-D	1138	2817	1268	2384	5276	1641	95324	119830	79140	157589	94640
14-24-E	1141	2117	1303	2098	5282	1523	94787	119096	76156	156608	94455
14-24-F	1073	1922	854	1660	5094	1491	91315	116858	76944	153504	93490
14-24-H	1164	1766	906	1611	5399	1466	94374	120146	78814	157679	96401
17-12-D	988	3103	405	<	5215	1556	91901	118101	80271	153495	92349
17-12-E	1050	2007	448	<	5193	1525	92109	119195	80473	154555	93643
17-12-F	1088	2495	260	<	5318	1508	92532	119078	80215	154321	94185
17-12-H	907	920	487	<	5476	1368	93653	121845	82579	156999	96124

(a) Results were below detection limits (shown in parentheses) for Ag (9), As (70), Cd (5), Cr (9), Cs (100,000), Mo (12), Ni (14), Sb (47), Se (70), Th (2300), Tl (92), U (140), V (7), and Zn (325). Results were insignificant for Ba (20.1±3.4), Be (11.8±0.5), Mn (10.4±1.7), and Sr (24.8±7.5). Boron was added as part of the digestion process so results for it are meaningless. Results for Pb were unreliable owing to a high background from Nb.

(b) See Table 2 for explanation of sample numbering.

Appendix B

Particle-Size Distribution Data for IE-911 Samples[a]

Sample[b]	Dia	Width	Vol%	Dia	Width	Vol%	Dia	Width	Vol%	Dia	Width	Vol%
1B	415.6	210.5	97	114.4	51.69	3						
1-24-D	436.4	223.6	97	134.7	41.59	3						
1-24-E	444.3	219.2	96	137.0	47.44	4						
1-24-F	446.7	220.5	98	135.8	42.79	2						
1-24-H	439.9	213.9	97	138.1	40.70	3						
1B[b]	6.143	7.821	42	1.493	1.180	58						
1-24-D[c]	8.275	4.194	14	4.298	1.824	16	1.403	1.063	71			
1-24-E[c]	6.834	8.369	36	1.432	1.110	64						
1-24-F[c]	23.95	11.39	3	8.458	5.137	21	4.138	1.924	16	1.471	1.159	60
1-24H[c]	6.684	7.623	34	1.424	1.099	66						
4-24-D	419.3	216.5	97	122.6	47.59	3						
4-24-E	397.2	211.2	96	102.7	29.59	2	33.52	49.79	2			
4-24-F	409.4	174.5	95	116.8	63.06	5						
4-24-H	424.3	203.0	94	126.0	70.14	5	1.682	1.035	1			
7-24-B	421.1	209.2	96	127.3	52.81	4						
7-24-D	435.9	205.2	99	137.6	37.19	1						
7-24-E	436.9	212.6	100									
7-24-F	392.0	200.4	97	99.44	35.01	3						
7-24-H	372.0	193.4	90	78.08	62.03	10						
10-24-B	386.4	181.0	87	66.02	67.21	13						
10-24-D	439.8	193.0	88	140.7	37.23	3	2.549	3.060	9			
10-24-E	420.9	207.3	95	130.3	48.52	3	2.638	2.327	2			
10-24-F	402.1	139.5	76	124.9	72.73	5	2.502	3.416	19			
10-24-H	454.1	210.5	93	138.3	52.26	5	1.990	1.268	2			
13-24-B	410.1	196.9	93	102.1	77.78	7						
13-24-D	399.4	143.4	62	119.9	61.73	3	43.10	23.37	2	1.967	2.652	33
13-24-E	416.8	206.7	94	127.0	50.24	3	2.083	1.658	3			
13-24-F	419.4	212.0	92	128.6	59.38	6	2.176	1.792	2			
13-24-H	419.4	200.3	93	125.6	61.18	6	1.993	1.109	1			
16-24-B	423.4	206.1	97	130.3	45.23	3						
16-24-D	421.3	201.2	92	121.2	61.28	4	48.58	18.56	1	1.983	1.272	3
16-24-E	386.2	207.2	97	101.1	26.16	3						
16-24-F	418.4	217.1	97	122.7	47.29	3						
16-24-H	406.9	220.6	98	103.8	24.28	2						
17-12-D	399.7	143.4	84	94.79	78.40	9	1.975	1.646	7			
17-12-E	435.1	202.2	94	136.6	62.53	4	2.434	2.138	2			
17-12-F	422.0	207.7	96	123.2	147.4	4						
17-12-H	420.6	157.8	85	117.3	101.7	7	2.068	2.485	8			

(a) Dia is the average diameter of a distribution manifold; Width is the range of the largest to smallest diameter for a given distribution manifold; Vol% is the percent of volume contained in a given distribution manifold.

(b) See Table 2 for explanation of sample numbering.

(c) Liquid samples; all other samples are slurry samples.

Appendix C

ICP-MS Analyses for Cs

Sample[a]	Cs Concentration in Original Sample (µg/L)
7A	2.15E+04
7C	518
7D	563
7E	69.9
7F	49.9
7H	34.5
7J	30.7
7Feed	2.13E+04
10A	2.07E+04
10D	2.17E+03
10E	2.16E+03
10F	313
10H	76.7
10J	38.4
13A	2.15E+04
13D	4.70E+03
13E	1.43E+03
13G	370
13H	63.6
16A	1.99E+04
16D	676
16E	437
16F	272
16H	108
16J	125
16J (NaOH)	109 (after hydroxide pulse)
17A	3.30E+03
17D	180
17E	439
17F	296
17H	92.4
17J	173
(a) See Table 2 for explanation of sample numbering.	

Appendix D

ICP-AES Analyses of Liquid Samples

Sample[a]	Concentration in Original Sample (mg/L)										
	Al	Fe	Mo	Na	P	Si	Ti	Zr	Nb	K	S
7A	7980	2.8	239	123000	563	127	<1.4	<0.5	<5	1400	5240
7C	343	4.1	1.65	43700	96.9	80.2	4.5	3.2	<5	300	23.2
7D	6140	1.0	177	94800	296	113	3.5	4.3	<5	1040	3890
7E	5720	<0.5	134	88300	229	116	2.4	4.0	<5	791	3070
7F	6880	0.7	176	102000	248	120	4.8	6.7	6.0	1020	3970
7H	7930	<0.5	242	122000	326	127	7.1	9.7	12.6	1390	5330
7J	8000	<0.5	238	124000	317	119	<1.4	7.9	12.1	1420	5240
7Feed	8300	2.0	241	122000	414	125	<1.4	<0.5	<5	1410	5510
10A	8100	1.9	229	120000	392	116	<1.4	<0.5	<5	1390	5250
10D	8000	3.6	230	118000	392	121	12.1	7.33	9.29	1390	5270
10E	8260	1.1	239	125000	403	132	30.0	16.8	24.0	1420	5470
10F	8290	0.81	240	125000	407	130	23.3	15.1	20.2	1430	5500
10H	8140	<0.5	231	124000	501	128	23.0	16.4	22.3	1440	5400
10J	8160	<0.5	237	123000	462	120	1.86	7.2	9.8	1420	5460
13A	7760	3.6	226	115000	384	110	<1.4	<0.48	<5	1310	5010
13D	7640	1.9	228	113000	365	121	25.4	14.4	18.7	1310	5050
13E	7730	1.1	228	115000	362	116	17.9	11.1	13.3	1320	5040
13F	7930	0.9	230	118000	359	123	27.1	16.9	20.3	1350	5100
13H	7770	<0.5	228	116000	354	119	19.5	14.5	19.4	1340	5040
16A	8360	2.9	241	123000	362	107	<1.4	<0.48	<5	1380	5250
16D	8090	1.5	227	119000	335	108	20.4	12.9	16.5	1360	4950
16E	7930	1.2	233	117000	349	105	7.9	7.1	7.0	1350	5080
16F	8030	1.0	227	121000	332	112	27.4	17.6	20.5	1390	5010
16H	8420	<0.5	243	123000	356	114	9.6	10.1	12.6	1430	5290
16J	8070	<0.5	226	117000	335	106	11.9	11.9	17.8	1400	4940
16J (NaOH)	4150	<0.5	119	81800	173	76	21.2	13.3	20.1	706	2590
17A	8150	2.1	229	118000	337	108	22.8	12.6	16.9	1360	5030
17D	982	<0.5	20.7	14300	23.9	11.7	7.3	3.1	<5	104	464
17E	8220	1.5	228	119000	336	105	16.6	10.9	12.4	1380	5020
17F	8150	1.0	226	118000	333	105	17.5	12.3	14.3	1370	4950
17H	8420	<0.5	236	123000	346	108	14.6	12.1	14.3	1410	5160
17J	7980	<0.5	230	117000	338	107	14.5	13.7	20.6	1340	5030
(a) See Table 2 for explanation of sample numbering.											

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