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Full-Scale Testing of a Caustic Side Solvent Extraction System to Remove Cesium from Savannah River Site Radioactive Waste

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Abstract: Savannah River Site (SRS) personnel have completed construction and assembly of the Modular Caustic Side Solvent Extraction Unit (MCU) facility. Following assembly, they conducted testing to evaluate the ability of the process to remove non-radioactive cesium and to separate the aqueous and organic phases. They conducted tests at salt solution flow rates of 3.5, 6.0, and 8.5 gpm.

During testing, the MCU Facility collected samples and submitted them to Savannah River National Laboratory (SRNL) personnel for analysis of cesium, Isopar® L, and modifier [1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol]. SRNL personnel analyzed the aqueous samples for cesium by Inductively-Coupled Plasma Mass Spectroscopy (ICP-MS) and the solvent samples for cesium using a Parr Bomb digestion followed by ICP-MS. They analyzed aqueous samples for Isopar® L and modifier by gas chromatography (GC).

The conclusions from the cesium analyses follow.

- The cesium in the feed samples measured 15.8 mg/L, in agreement with expectations.
- The decontamination factor measured 181–1580 at a salt solution flow rate of 3.5 gpm, 211–252 at a salt solution flow rate of 6.0 gpm, and 275–878 at a salt solution flow rate of 8.5 gpm.
- The concentration factor measured 11.0–11.1 at 3.5 gpm salt solution flow rate, 12.8–13.2 at 6.0 gpm salt solution flow rate, and 12.0–13.2 at 8.5 gpm salt solution flow rate.

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- The organic carryover from the final extraction contactor (#7) varied between 22 and 710 mg/L Isopar® L. The organic carryover was less at the lowest flow rate.
- The organic carryover from the final strip contactor (#7) varied between 80 and 180 mg/L Isopar® L.
- The organic carryover in the Decontaminated Salt Solution Hold Tank (DSSHT) and the Strip Effluent Hold Tank (SEHT) was less than 10 mg/L Isopar® L, indicating good recovery of the solvent by the coalescers and decanters.

Keywords:

INTRODUCTION

The Department of Energy identified the Caustic Side Solvent Extraction (CSSX) process as the preferred technology for removing cesium from radioactive waste solutions at the Savannah River Site (SRS) (1,2). As a result, the Washington Savannah River Company (WSRC) designed and built the modular caustic side solvent extraction unit (MCU) in the SRS Tank Farm to process liquid waste for an interim period until the Salt Waste Processing Facility (SWPF) begins operations.

In the CSSX process (see Fig. 1), solvent contacts SRS liquid salt waste in centrifugal contactors. During contact, cesium transfers from the aqueous phase (i.e., salt solution) to the solvent, and the aqueous and organic phases are separated. The solvent is stripped of cesium by dilute nitric acid (0.001 M) in subsequent contactors. Following separation of the strip solution from the solvent, the strip effluent is transported to the Defense Waste Processing Facility. The decontaminated aqueous salt solution is transferred via a piping system for ultimate disposal through the Saltstone Production Facility. The stripped solvent is washed with 0.01 M NaOH to prepare it for reuse in the process. The decontaminated salt solution (DSS) and strip effluent (SE) solution will contain droplets of solvent that have not been separated in the centrifugal contactors. Previous testing has shown that the concentration of organic solvent in the decontaminated salt solution and strip effluent exiting the contactors can be as much as 2000 mg/L. Because of the cost of the solvent and the adverse impacts on downstream facilities, SRS has installed coalescers and decanters downstream of the contactors to recover this solvent from these streams.

The solvent for this process contains four components. The extractant is a calixarene-crown ether, calix[4]arene-*bis*(tert-octylbenzo-crown-6), called BOBCalixC6. The solvent contains a modifier, which is an alkyl

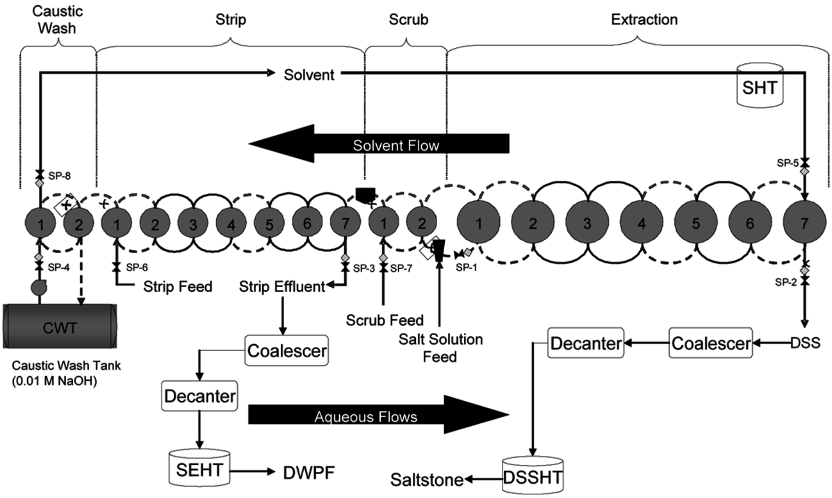

Figure 1. Solvent extraction contactor layout.

aryl polyether, to keep the extractant dissolved in the solvent and increase its ability to extract cesium in the extraction section. The modifier is 1-(2,2,3,3,-tetrafluoropropoxy)-3-(4-sec-butylphenoxy)-2-propanol, and is called Cs-7SB. The solvent contains a suppressant, trioctyl amine, which suppresses the effects of anionic organic impurities and improves the back-extraction of cesium from the solvent in the stripping section. The diluent is Isopar® L, a mixture of branched hydrocarbons (3–6).

The centrifugal contactors used in the MCU were manufactured by Costner Industries Nevada Corporation (CINC) and installed on a contactor assembly. This contactor assembly is installed in a concrete "contactor enclosure". The process uses two sizes of contactors, a V-05 and a V-10. The V-05 is 5 inches in diameter, while the V-10 is 10 inches in diameter.

The extraction step of the CSSX process in the MCU takes place in a bank of seven V-10 contactors. The aqueous salt solution is fed to the contactor assembly from the Salt Solution Feed Tank (SSFT) or Decontaminated Salt Solution Hold Tank (DSSHT). Feed from the DSSHT is used for start-up and shut-down of the cycle, while feed from the SSFT is "new" feed. The feed solution enters the contactor assembly and is fed to the extraction aqueous feed heat exchanger. This heat exchanger is supplied with cooling water to bring the feed solution to 23 +/− 3°C. The feed solution is combined with scrub solution from scrub contactor #2 and potential solvent from the strip effluent decanter. The aqueous feed gravity flows into extraction contactor #1. The solvent feed to the extraction contactors comes from the Solvent Hold Tank (SHT). The solvent enters the contactor assembly and passes through the extraction solvent heat exchanger to cool the solvent to 23 +/− 3°C, and then is fed to extraction contactor #7. The aqueous and solvent streams flow through the extraction contactor bank countercurrent to one another, with the decontaminated salt solution exiting the bank at extraction contactor #7 and the solvent exiting at extraction contactor #1. The decontaminated salt solution flows from extraction contactor #7 to the DSS Coalescer which increases the droplet size of solvent carried over in the aqueous stream facilitating its removal in the DSS Decanter (DSSD). In the DSSD, the solvent is separated from the aqueous phase, with the solvent being returned to the Solvent Hold Tank, while the decontaminated salt solution is transferred to the Decontaminated Salt Solution Hold Tank. The V-10 contactors in the extraction bank are surrounded by water jackets, and these jackets are supplied with water to maintain the process solution temperature within 23 +/− 3°C over the entire extraction bank.

The solvent exiting the extraction contactor bank gravity flows into scrub contactor #2. The two contactors in the scrub bank are CINC V-05 contactors. The aqueous scrub solution is fed to the scrub bank from the Scrub Feed Tank. When the scrub solution enters the contactor assembly, it directly feeds scrub contactor #1 and when it exits scrub contactor #2, it is combined with the salt solution feed and fed to the extraction contactors.

The solvent exits scrub contactor #1 and gravity flows to the strip solvent heater, which raises the solvent temperature to 33 +/− 3°C, and then flows to strip contactor #7 in the strip bank. The strip bank contains seven V-05 contactors. Strip solution is fed to the strip bank from the Strip Feed Tank. It enters the strip aqueous heater where the temperature is controlled to 33 +/− 3°C and is then fed to strip contactor #1. The seven contactors in the strip bank are surrounded by water jackets, and these jackets are supplied with water to maintain the process solution temperature within 33 +/− 3°C over the entire extraction bank. The strip effluent solution flows from strip contactor #7 to the Strip Effluent Coalescer which serves to increase the droplet size of solvent carried over in the aqueous stream facilitating its removal in the Strip Effluent Decanter. In the decanter, the solvent is separated from the aqueous stream, with the solvent being returned to the extraction bank, while the strip effluent solution is transferred to the Strip Effluent Hold Tank. The solvent solution exits strip contactor #1 and gravity flows to wash contactor #2.

In the wash contactors, dilute sodium hydroxide (0.01 M) is fed to wash contactor #1 from the Caustic Wash Tank (CWT) and is returned to the Caustic Wash Tank from wash contactor #2. The solvent exits the wash bank at wash contactor #1 and is returned to the Solvent Hold Tank.

SRS personnel have completed construction and assembly of the MCU facility. Following assembly, they conducted testing to evaluate the ability of the process to remove non-radioactive cesium and to separate the aqueous and organic phases.

TESTING

The simulant testing was conducted at salt solution flow rates of 3.5 gpm, 6.0 gpm, and 8.5 gpm, and associated solvent flow rates of 1.17 gpm, 2.0 gpm, and 2.83 gpm, respectively. The strip, scrub, and wash flow rates were equal and set to 0.23 gpm, 0.4 gpm, and 0.57 gpm, respectively. The salt solution was spiked with non-radioactive cesium, equivalent to the maximum expected feed concentration of 1.1 Ci/gal ^{137}Cs. A DF of 12 is needed to meet the decontaminated salt solution effluent criteria of <0.1 Ci/gal ^{137}Cs.

The sample times specified in these tests are based on the elapsed time to turnover the solvent hold tank four times. The CSSX process will have reached equilibrium prior to four turnovers.

Personnel conducted the tests as follows. A vendor (Blue Line Chemical) prepared simulated SRS salt solution (see Table 1). MCU personnel added nonradioactive cesium to the salt solution to achieve a cesium concentration of 14.9 mg/L (equivalent to 1.1 Ci/gal ^{137}Cs). They

Table 1. SRS simulated salt solution composition

Species	Concentration (molar)
KNO_3	0.015
NaOH	2.07
$NaNO_3$	2.02
$NaNO_2$	0.50
$NaAlO_2$	0.28
Na_2CO_3	0.15
Na_2SO_4	0.14
NaCl	0.024
NaF	0.028
Na_2HPO_4	0.007
$Na_2C_2O_4$	0.008
Na_2SiO_3	0.030
Na_2MoO_4	0.00007

mixed the salt solution with agitators and by recirculating it between the salt solution receipt tank (SSRT) and salt solution feed tank (SSFT) for 10 hours. Following the 10 hour recirculation, they collected samples from the SSRT and SSFT, and analyzed them for cesium.

Once they determined that the cesium concentration was at the desired level, they adjusted the salt solution feed rate to 3.5 gpm. They adjusted the speed of the extraction contactors to 1200 rpm, and the scrub, strip, and wash contactors to 1800 rpm. They set the caustic wash flow rate to 0.23 gpm, the strip flow rate to 0.23 gpm, the scrub flow rate to 0.23 gpm, and the solvent flow rate to 1.17 gpm. They started the test and collected samples at 0, 342, 458, and 684 minutes.

At the completion of the test described above, they increased the salt solution flow rate to 6.0 gpm. They increased the extraction contactor speed to 1800 rpm, and the scrub, strip, and wash contactor speed to 2100 rpm. They increased the caustic wash, strip, and scrub flow rates to 0.40 gpm. They increased the solvent flow rate to 2.0 gpm. They collected samples at 0, 200, 268, and 400 minutes.

At the completion of the test described above, they increased the salt solution flow rate to 8.5 gpm. They set the extraction contactor speed to 1800 rpm, and the scrub, strip, and wash contactor speed to 2100 rpm. They increased the caustic wash, strip, and scrub flow rates to 0.57 gpm. They increased the solvent flow rate to 2.83 gpm. They collected samples at 0, 142, 190, and 283 minutes.

They collected samples from the inlet and outlet of selected contactors to measure cesium removal from the salt solution, cesium transfer from the solvent to the strip acid, and organic solvent carryover into the decontaminated salt solution and strip acid.

They collected samples from the Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank to measure the effectiveness of the coalescers in recovering the solvent from the aqueous streams. The Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank samples for Isopar® L obtained during the test were placed in a container with ice to prevent Isopar® L evaporation prior to analysis.

Following those tests, they performed a solvent cleanup test in which they recycled decontaminated salt solution through the contactors to remove cesium from the solvent. For this test, they adjusted the salt solution flow rate to 6.0 gpm, the extraction contactor speed to 1800 rpm, the scrub, strip, and wash contactor speed to 2100 rpm, the caustic wash, scrub, and strip flow rates to 0.4 gpm, and the solvent flow rate to 2.0 gpm. They fed the contactors from the Decontaminated Salt Solution Hold Tank rather than the salt solution feed tank. They collected samples after 50, 100, 200, 300, and 400 minutes to measure the cesium concentration in the salt solution and solvent hold tank.

Following that test, they stopped and restarted the MCU process to determine its ability to rapidly reestablish process efficiency after shutdown and restart. They collected samples after 30 minutes to measure cesium in the decontaminated salt solution, strip effluent, and Solvent Hold Tank. The operating parameters for this test were the same as in the solvent cleanup test.

ANALYSES

The authors performed the ^{133}Cs analysis by ICP-MS. The aqueous samples (decontaminated salt solution and strip effluent) were submitted directly to the ICP-MS. The solvent samples were digested using a Parr Bomb digestion prior to analysis by ICP-MS. They analyzed salt and strip samples for Isopar® L and modifier by gas chromatography with flame ionization detection (GC-FID) and gas chromatography-mass spectrometry (GC-MS).

The ICP-MS used for the analyses is a Thermo-Elemental Plasma Quad II. This instrument provides multi-element analyses of aqueous solutions, and the analytical results can be expressed as either elemental or isotopic concentrations. The instrument aerosolizes the sample and transports the aerosol to the argon plasma. In the high temperature plasma (~10,000°K) metallic species are ionized. The ions generated by the plasma enter the mass spectrometer through a sampling cone set near the end of the plasma. The ions are separated by a quadrapole mass filter and focused on a detector. The detector provides either an ion count or an analog signal. The signal from the detector is amplified, measured, and stored in a multi-channel analyzer, and these measurements are used to calibrate the instrument and determine the concentrations of the elements of concern.

The authors performed the solvent sample digestions as follows. Approximately 0.1–0.2 g of the well-mixed sample was transferred to the Teflon™ cup of a Parr Bomb dissolution container. A 3 mL aliquot of high-purity concentrated nitric acid was added and the dissolution container sealed. Typically eight containers were heated simultaneously in an oven pre-heated to 175°C. Heating was continued for at least three hours after the oven temperature re-equilibrated to 175°C. After cooling to room temperature, the containers were opened and the nitric acid solutions were diluted to 10 mL with de-ionized water. No immiscible organic fraction or solution cloudiness was evident after this treatment, indicating that the oxidation of the organic fraction in the samples was complete.

Personnel performed the GC-FID and GC-MS analyses as follows. They weighed the sample bottle. They either added hexane to the sample bottle (1/4 of sample volume) or transferred the sample to a larger bottle and rinsed the sample bottle with the hexane. They recorded the weight of the bottle, sample, and hexane. They removed the top layer of liquid and placed it in a vial with a Teflon^{TM} cap. They recorded the empty bottle weight. They dried the hexane with sodium sulfate, collected aliquots, and analyzed them.

GC-MS analysis or GC-FID analysis was employed to identify organic compounds in the samples. Analytical separations were carried out on a Hewlett Packard 6890 gas chromatograph, equipped with a 30 m DB-XLB column, with 0.18 mm diameter and 0.20 μ film thickness for GC-MS. The GC-FID uses a 30 m DB-5ms column, with 0.2 mm diameter and 0.33 μ film thickness. Quantification was performed using a Hewlett Packard 5973 mass selective detector. The mass spectrometer tuning was confirmed within 24 hours prior to each measurement using perfluorotributylamine.

RESULTS

Cesium Removal

Table 2 shows the analysis of the feed solution along with the control submitted. The feed cesium concentration measured 15.8 mg/L in both samples versus a target of 14.9 mg/L. The 15 mg/L control sample measured 14.6 mg/L (3% difference), well within the standard analytical uncertainty of ±10%. The analytical uncertainty on all measured values is ±10%, unless otherwise stated. The basis for the analytical uncertainty is a reflection of the analytical precision and the experimental error from sample preparation and handling.

Table 3 shows the cesium concentration in the samples from the test conducted with a salt solution flow rate of 3.5 gpm. The Decontamination Factor (DF) varied between 181 and 1580, with an average value

Table 2. Feed solution cesium concentration

Sample	Cesium (mg/L)
MCU-CS-I-SSRT	15.8
MCU-CS-I-SSFT	15.8
15 mg/L Control	14.6

Table 3. Cesium concentration (mg/L) during test with 3.5 gpm salt solution flow rate

Sample ID	Time (min)	Feed	DSS	SE	Solvent	DF	CF
MCU-CS-I-SSRT		15.8					
MCU-CS-I-SSFT		15.8					
MCU-CS-A-EC-A-O-1	342		<0.010				
MCU-CS-A-EC-A-O-2	342		0.017				
MCU-CS-A-EC-A-O-3	458		0.069				
MCU-CS-A-EC-A-O-5	684		0.087				
MCU-CS-A-EC-A-O-6	684		0.044				
MCU-CS-A-SC-A-O-1	342			174			
MCU-CS-A-SC-A-O-3	458			176			
MCU-CS-A-SC-A-O-5	684			174			
MCU-CS-A-EC-O-I	0				1.062		
MCU-CS-A-EC-O-I-1	342				0.209		
MCU-CS-A-EC-O-I-3	458				0.849		
MCU-CS-A-EC-O-I-5	684				0.400		
MCU-CS-A-EC-O-I-6	684				0.588		
Minimum			<0.010	174		181	11.0
Maximum			0.087	176		1580	11.1
Average			0.045	175		348	11.1
Standard deviation			0.033	1.1			

DSS: decontaminated salt solution.

SE: strip effluent.

DF: decontamination factor (DF $= Cs_{feed}/Cs_{DSS}$).

CF: concentration factor (DF $= Cs_{strip}/Cs_{feed}$).

of 348. This DF exceeds the target of 12, and shows effective cesium removal from the simulated waste stream. The Concentration Factor (CF) varied between 11.0 and 11.1. The concentration factor is slightly below the target of 12. The cesium concentration in the solvent entering the extraction contactors was less than 1.1 mg/L, confirming effective cesium removal from the solvent during the stripping process.

Table 4 shows the cesium concentration in the samples from the test conducted with a 6.0 gpm salt solution flow rate. The DF varied between 211 and 252, with an average value of 227. This DF exceeds the target of 12, and shows effective cesium removal from the simulated waste stream. The CF varied between 12.8 and 13.2, which meets the target. The cesium concentration in the solvent entering the extraction contactors was less than 1 mg/L, confirming effective cesium removal from the solvent during stripping, again.

Table 4. Cesium concentration (mg/L) during test with 6.0 gpm salt solution flow rate

Sample ID	Time (min)	Feed	DSS	SE	Solvent	DF	CF
MCU-CS-I-SSRT		15.8					
MCU-CS-I-SSFT		15.8					
MCU-CS-B-EC-A-O-1	200		0.075				
MCU-CS-B-EC-A-O-3	268		0.071				
MCU-CS-B-EC-A-O-5	400		0.063				
MCU-CS-B-SC-A-O-1	200			204			
MCU-CS-B-SC-A-O-3	268			202			
MCU-CS-B-SC-A-O-5	400			209			
MCU-CS-B-SC-A-O-6	400			207			
MCU-CS-B-EC-O-I	0				0.749		
MCU-CS-B-EC-O-I-1	200				0.227		
MCU-CS-B-EC-O-I-3	268				0.214		
MCU-CS-B-EC-O-I-5	400				0.191		
Minimum			0.063	202		211	12.8
Maximum			0.075	209		252	13.2
Average			0.070	205.5		227	13.0
Standard deviation			0.006	3.1			

DSS: decontaminated salt solution.

SE: strip effluent.

DF: decontamination factor (DF $= Cs_{feed}/Cs_{DSS}$).

CF: concentration factor (DF $= Cs_{strip}/Cs_{feed}$).

Table 5 shows the cesium concentration in the samples from the test conducted with a 8.5 gpm salt solution flow rate. The DF varied between 275 and 878, with an average value of 470. This DF exceeds the target of 12, and shows effective cesium removal from the simulated waste stream. The CF varied between 12.0 and 13.2, which meets the target. The cesium concentration in the solvent entering the extraction contactors was less than 1 mg/L, confirming effective cesium removal from the solvent by stripping, again.

The data in Tables 3–5 show no correlation between the cesium decontamination factor (DF) and the salt solution flow rate over the conditions tested. They also show no correlation between concentration factor (CF) and the salt solution flow rate over the conditions tested.

Tables 6 and 7 show the cesium concentration in the samples collected during the Solvent Cleanup Test. Table 6 shows the cesium in the decontaminated salt solution samples. The cesium concentration decreased with time during this test, and all samples contained less than 0.2 mg/L cesium.

Table 5. Cesium concentration (mg/L) during test with 8.5 gpm salt solution flow rate

Sample ID	Time (min)	Feed	DSS	SE	Solvent	DF	CF
MCU-CS-I-SSRT		15.8					
MCU-CS-I-SSFT		15.8					
MCU-CS-C-EC-A-O-1	142		0.057				
MCU-CS-C-EC-A-O-3	190		0.032				
MCU-CS-C-EC-A-O-5	283		0.028				
MCU-CS-C-EC-A-O-6	283		0.018				
MCU-CS-C-SC-A-O-1	142			208			
MCU-CS-C-SC-A-O-3	190			190			
MCU-CS-C-SC-A-O-5	283			199			
MCU-CS-C-EC-O-I	0				0.29		
MCU-CS-C-EC-O-I-1	142				0.14		
MCU-CS-C-EC-O-I-3	190				0.46		
MCU-CS-C-EC-O-I-5	283				0.077		
MCU-CS-C-EC-O-I-6	283				0.099		
Minimum			0.018	190		275	12.0
Maximum			0.057	208		878	13.2
Average			0.034	199		470	12.6
Standard deviation			0.017	9			

DSS: decontaminated salt solution.
SE: strip effluent.
DF: decontamination factor (DF $= Cs_{feed}/Cs_{DSS}$).
CF: concentration factor (DF $= Cs_{strip}/Cs_{feed}$).

Table 7 shows the cesium concentration in the Solvent Hold Tank (SHT). The cesium concentration measured less than 0.3 mg/L. The solvent cesium concentration at the conclusion of the test with 8.5 gpm salt solution flow rate measured 0.21 ± 0.16. The initial solvent hold tank sample, collected 50 minutes after the start of the solvent cleanup test,

Table 6. Cesium concentration (mg/L) in salt solution during the solvent cleanup test

Sample ID	Time (min)	Cesium (mg/L)
MCU-CS-W-EC-A-I-1	50	0.129
MCU-CS-W-EC-A-I-3	100	0.078
MCU-CS-W-EC-A-I-5	200	0.038
MCU-CS-W-EC-A-I-7	300	0.046
MCU-CS-W-EC-A-I-9	400	0.043

Table 7. Cesium concentration (mg/L) in solvent hold tank during solvent cleanup test

Sample ID	Time (min)	Cesium (mg/L)
MCU-CS-SHT-1	79	0.030
MCU-CS-SHT-3	100	0.025
MCU-CS-SHT-5	200	0.037
MCU-CS-SHT-7	300	0.051
MCU-CS-SHT-9	400	0.293

had a cesium concentration of 0.030 mg/L, showing a large fraction of the cesium had been removed from the solvent. Subsequent samples showed similar cesium concentrations. The last sample collected showed a higher cesium concentration. We are unsure of the reason for this increase. Given that the salt solution (see Table 6) did not show a similar increase in cesium, this result is likely due to analytical error.

Table 8 shows the cesium concentration in the decontaminated salt solution, strip effluent, and Solvent Hold Tank during the System Shutdown/Restart Test. In this test, the MCU system was shut down and restarted. The DF was 268, and the CF was 11.8. These results are consistent with the results from the tests described above. During those tests, the cesium in the decontaminated salt solution averaged 0.048 ± 0.026 mg/L, the cesium in the strip effluent averaged 194 ± 15 mg/L, and the cesium in the solvent averaged 0.40 ± 0.31 mg/L. The DF averaged 491 ± 461, and the CF averaged 12.2 ± 0.9.

Table 8. Cesium concentration during system shutdown test

Sample ID	Sample	Cesium (mg/L)
MCU-CS-I-SSFT	Feed	15.8
MCU-CS-D-EC-A-O-1	DSS	0.059
MCU-CS-D-SC-A-O-1	SE	187
MCU-CS-SHT-11	SHT	0.851
	DF	268
	CF	11.8

DSS: decontaminated salt solution.

SE: strip effluent.

SHT: solvent hold tank.

DF: decontamination factor (DF $= Cs_{feed}/Cs_{DSS}$).

CF: concentration factor (DF $= Cs_{strip}/Cs_{feed}$).

Table 9. Cesium concentration (mg/L) in inlet to extraction contactor #1

Sample	Salt Solution Flow Rate (gpm)	Cesium (mg/L)
MCU-CS-A-EC-A-I	3.5 gpm	16.8
MCU-CS-B-EC-A-I	6.0 gpm	16.9
MCU-CS-C-EC-A-I	8.5 gpm	18.3
MCU-CS-I-SSRT		15.8
MCU-CS-I-SSFT		15.8

Table 9 shows the cesium concentration in the inlet to Extraction Contactor #1. The concentration is slightly higher than in the Salt Solution Receipt Tank (SSRT) and the Salt Solution Feed Tank (SSFT) (15.8 mg/L). The increase in cesium concentration is due to mixing of the salt solution with scrub prior to entering Extraction Contactor #1.

Organic Carryover

Table 10 shows the measured Isopar® L concentration in aqueous samples collected from the outlet of Extraction Contactor #7. The organic carryover varied between 22 and 709 mg/L Isopar® L. The carryover measured during the test at 3.5 gpm was much less than measured during tests at higher salt solution flow rate (31 mg/L Isopar® L versus 444–524 mg/L Isopar® L). Several plausible reasons exist for the lower organic carryover at lower flow rates. A lower flow rate leads to a longer residence time in the contactors, which allows more time for phase separation. The tests at lower flow rate had a lower rotor speed. The lower rotor speed provides less energy, which would produce larger organic droplets. The larger organic droplets separate more rapidly from the aqueous phase. The lower rotor speed leads to less turbulence. This result is consistent with the results from tests conducted previously, where the organic carryover was $\sim$ 80 mg/L Isopar® L at salt solution flow rates of 3.5 gpm, and 130–100 mg/L Isopar® L at flow rates of 4.5–8.5 gpm salt solution.

Table 11 shows the measured Isopar® L concentration in aqueous samples collected from the Decontaminated Salt Solution Hold Tank. In all samples, the Isopar® L concentration is less than 10 mg/L. These results show that the coalescer and decanter effectively recovered solvent from the decontaminated salt solution.

Table 12 shows the measured Isopar® L concentration in aqueous samples collected from the outlet of Strip Contactor #7. The organic carryover varied between 80 and 182 mg/L Isopar® L. No significant difference in organic carryover was observed between the different tests.

Table 10. Isopar® L concentration in extraction contactor #7 outlet

Sample ID	Test (gpm)	Time (min)	Isopar® L (mg/L)	Modifier (mg/L)	Isopar® L/ Modifier
MCU-ISO-A-EC-A-O-1	3.5	342	37.0	22.2	1.66
MCU-MS-1	3.5	342	33.2	14.2	2.34
MCU-ISO-A-EC-A-O-3	3.5	458	21.8	20.1	1.08
MCU-MS-3	3.5	458	33.6	19.7	1.71
MCU-ISO-A-EC-A-O-5	3.5	684	35.1	23.9	1.47
MCU-ISO-A-EC-A-O-6	3.5	684	31.7	23.6	1.34
MCU-MS-5	3.5	684	26.9	12.0	2.24
Average	3.5		31.3	19.4	1.61
Standard deviation	3.5		5.3	4.6	
MCU-ISO-B-EC-A-O-1	6.0	200	487	233	2.09
MCU-MS-7	6.0	200	501.5	154.2	3.25
MCU-ISO-B-EC-A-O-3	6.0	268	366.8	174.6	2.10
MCU-ISO-B-EC-A-O-5	6.0	400	419.6	201.7	2.08
Average	6.0		443.7	190.9	2.32
Standard deviation	6.0		62.5	34.2	
MCU-ISO-C-EC-A-O-1	8.5	142	709.1	315.0	2.25
MCU-ISO-C-EC-A-O-3	8.5	190	210.8	137.6	1.53
MCU-ISO-C-EC-A-O-5	8.5	283	651.0	286.9	2.27
Average	8.5		523.6	246.5	2.12
Standard deviation	8.5		272.4	95.4	

These results are consistent with the tests conducted previously, in which the organic carryover varied between 170 and 370 mg/L Isopar® L.

Table 11. Isopar® L concentration in DSSHT

Sample ID	Test (gpm)	Time (min)	Isopar® L (mg/L)	Modifier (mg/L)
MCU-ISO-A-DT-A-1	3.5	342	<3	<3
MCU-ISO-A-DT-A-3	3.5	458	<2	<2
MCU-ISO-A-DT-A-5	3.5	684	<2	<2
MCU-ISO-A-DT-A-6	3.5	684	<2	<2
MCU-ISO-B-DT-A-1	6.0	200	<2	<2
MCU-ISO-B-DT-A-3	6.0	268	<2	<2
MCU-ISO-B-DT-A-5	6.0	400	<3	<3
MCU-ISO-B-DT-A-6	6.0	400	<3	29.9
MCU-ISO-C-DT-A-1	8.5	142	<2	14.2
MCU-ISO-C-DT-A-3	8.5	190	4.9	7.3
MCU-ISO-C-DT-A-5	8.5	283	7.7	9.5

Table 12. Isopar® L concentration in Strip Contactor #7 Outlet

Sample ID	Test (gpm)	Time (min)	Isopar® L (mg/L)	Modifier (mg/L)	Isopar® L/ Modifier	Insoluble Modifier (mg/L)	Mod. Isopar® L/ Modifier
MCU-ISO-A-SC-A-O-1	3.5	342	80.5	66.8	1.21	43.2	1.86
MCU-ISO-A-SC-A-O-3	3.5	458	145.7	108.2	1.35	84.6	1.72
MCU-ISO-A-SC-A-O-4	3.5	458	130.4	81.0	1.61	57.4	2.27
MCU-ISO-A-SC-A-O-5	3.5	684	181.8	96.7	1.88	73.1	2.49
Average	3.5		134.6	88.2	1.53	64.6	2.07
Standard deviation	3.5		42.0	18.1			
MCU-ISO-B-SC-A-O-1	6.0	200	161.6	92.7	1.74	69.1	2.34
MCU-ISO-B-SC-A-O-3	6.0	268	158.3	89.9	1.76	66.3	2.39
MCU-ISO-B-SC-A-O-4	6.0	268	147.8	87.7	1.69	64.1	2.31
MCU-ISO-B-SC-A-O-5	6.0	400	167.8	112.0	1.50	88.4	1.90
Average	6.0		158.9	95.6	1.66	72.0	2.23
Standard deviation	6.0		8.4	11.1			
MCU-ISO-C-SC-A-O-1	8.5	142	113.9	79.7	1.43	56.1	2.03
MCU-ISO-C-SC-A-O-3	8.5	190	98.4	74.9	1.31	51.3	1.92
MCU-ISO-C-SC-A-O-5	8.5	283	99.8	73.5	1.36	49.9	2.00
Average	8.5		104.0	76.0	1.37	52.4	1.98
Standard deviation	8.5		8.6	3.3			

Table 13. Isopar® L concentration in SEHT

Sample ID	Test (gpm)	Time (min)	Isopar® L (mg/L)	Modifier (mg/L)
MCU-ISO-A-ST-A-1	3.5	342	<3	22.3
MCU-ISO-A-ST-A-3	3.5	458	<3	23.6
MCU-ISO-A-ST-A-5	3.5	684	<3	26.5
MCU-ISO-B-ST-A-1	6.0	200	<3	25.2
MCU-ISO-B-ST-A-2	6.0	200	<3	21.3
MCU-ISO-B-ST-A-3	6.0	268	<2	20.1
MCU-ISO-B-ST-A-5	6.0	400	<3	24.5
MCU-ISO-C-ST-A-1	8.5	142	<3	25.9
MCU-ISO-C-ST-A-3	8.5	190	<3	17.9
MCU-ISO-C-ST-A-5	8.5	283	<3	28.8
Average			<3	23.6

Table 13 shows the measured Isopar® L concentration in aqueous samples collected from the Strip Effluent Hold Tank. In all samples, the Isopar® L concentration is less than 10 mg/L. The modifier concentration in Strip Effluent Hold Tank was 18–29 mg/L. The modifier concentration is higher than the Isopar® L concentration due to the modifier's solubility in dilute nitric acid. Given that the average modifier concentration in the Strip Effluent Hold Tank was 23.6 mg/L, we estimate the solubility of modifier in strip acid to be 23.6 mg/L. These results show that the coalescer and decanter effectively recovered solvent from the strip effluent stream.

Subtracting the soluble modifier from the measured modifier in the Strip Effluent Contactor outlet samples, we calculate an insoluble modifier concentration (see Table 12). Using the insoluble modifier concentration, we calculate a modified Isopar® L:modifier ratio, which varies between 1.72 and 2.49 with an average of 2.1. This average agrees well with the calculated Isopar® L:modifier ratio of 2.32.

CONCLUSIONS

The conclusions from the cesium analyses follow.

- The cesium in the feed samples measured 15.8 mg/L, in agreement with expectations.
- The decontamination factor measured 181–1580 at a salt solution flow rate of 3.5 gpm, 211–252 at a salt solution flow rate of 6.0 gpm, and 275–878 at a salt solution flow rate of 8.5 gpm.

- The concentration factor measured 11.0–11.1 at 3.5 gpm salt solution flow rate, 12.8–13.2 at 6.0 gpm salt solution flow rate, and 12.0–13.2 at 8.5 gpm salt solution flow rate.
- The organic carryover from the final extraction contactor (#7) varied between 22 and 710 mg/L Isopar® L. The organic carryover was less at the lowest flow rate.
- The organic carryover from the final strip contactor (#7) varied between 80 and 180 mg/L Isopar® L.
- The organic carryover in the Decontaminated Salt Solution Hold Tank and the Strip Effluent Hold Tank was less than 10 mg/L Isopar® L, indicating good recovery of the solvent by the coalescers and decanters.

REFERENCES

1. Huntoon, C.L.; Rudy, G.P. (2001) Memorandum titled Preferred Alternative for the Savannah River Salt Processing Project, June 25.
2. Dimenna, R.A.; Elder, H.H.; Fowler, J.R.; Fowler, R.C.; Gregory, M.V.; Hang, T.; Jacobs, R.A.; Paul, P.K.; Pike, J.A.; Rutland, P.L.; Smith, F.G. III, Subosits, S.G.; Taylor, G.A.; Campbell, S.G.; Washburn, F.A. (2001) Bases, Assumptions, and Results of the Flowsheet Calculations for the Decision Phase Salt Disposition Alternatives, WSRC-RP-99-00006, Rev. 3, May 24.
3. Leonard, R.A.; Aase, S.B.; Arafat, H.; Conner, A.C.; Chamberlain, D.B.; Falkenberg, J.R.; Regalbutto, M.C.; Vandegrift, G.F. (2003) Experimental Verification of Caustic-Side Solvent Extraction for Removal of Cesium from Tank Waste, Solv. Ext. Ion. Exchange, *21* (4): 505–526.
4. Wilmarth, W.R.; Mills, J.T.; Dukes, V.H.; Beasley, M.C.; Coleman, A.D.; DiPrete, C.C.; DiPrete, D.P. (2003) Caustic-Side Solvent Extraction Batch Distribution Coefficient Measurements for Savannah River Site High-Level Wastes, Sep. Sci. Tech., *38* (12–13): 2637–2645.
5. Norato, M.A.; Beasley, M.H.; Campbell, S.G.; Coleman, A.D.; Geeting, M.W.; Guthrie, J.W.; Kennel, C.W.; Pierce, R.A.; Ryberg, R.C.; Walker, D.D.; Law, J.D.; Todd, T.A. (2003) Demonstration of the Caustic-Side Solvent Extraction Process for the Removal of ^{137}Cs from Savannah River Site High Level Waste. Sep. Sci. Tech., *38* (12–13): 2647–2666.
6. Bonnesen, P.V.; Delmau, L.H.; and Moyer, B.A. (2000) A Robust Alkaline-Side CSEX Solvent Suitable for Removing Cesium from Savannah River High Level Waste, Solv. Ext. Ion Exch., *18* (6): 1079–1107.