

Operations Review of the Savannah River Site Integrated Salt Disposition Process - 11327

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ABSTRACT

The Savannah River Site (SRS) is removing liquid radioactive waste from its Tank Farm. To treat waste streams that are low in Cs-137, Sr-90, and actinides, SRS developed the Actinide Removal Process and implemented the Modular Caustic Side Solvent Extraction (CSSX) Unit (MCU). The Actinide Removal Process contacts salt solution with monosodium titanate to sorb strontium and select actinides. After monosodium titanate contact, the resulting slurry is filtered to remove the monosodium titanate (and sorbed strontium and actinides) and entrained sludge. The filtrate is transferred to the MCU for further treatment to remove cesium. The solid particulates removed by the filter are concentrated to ~ 5 wt %, washed to reduce the sodium concentration, and transferred to the Defense Waste Processing Facility for vitrification. The CSSX process extracts the cesium from the radioactive waste using a customized solvent to produce a Decontaminated Salt Solution (DSS), and strips and concentrates the cesium from the solvent with dilute nitric acid. The DSS is incorporated in grout while the strip acid solution is transferred to the Defense Waste Processing Facility for vitrification.

The facilities began radiological processing in April 2008 and started processing of the third campaign ("MarcoBatch 3") of waste in June 2010. Campaigns to date have processed ~1.2 million gallons of dissolved saltcake. Savannah River National Laboratory (SRNL) personnel performed tests using actual radioactive samples for each waste batch prior to processing. Testing included monosodium titanate sorption of strontium and actinides followed by CSSX batch contact tests to verify expected cesium mass transfer. This paper describes the tests conducted and compares results from facility operations. The results include strontium, plutonium, and cesium removal, cesium concentration, and organic entrainment and recovery data. Additionally, the poster describes lessons learned during operation of the facility.

INTRODUCTION

In 2001, the Department of Energy (DOE) identified the CSSX process as the preferred processing option for removing cesium from SRS wastes. The first step of the process, housed within the Actinide Removal Process, involves sorption of radioactive strontium and alpha-emitting actinides onto monosodium titanate [1]. These solids, as well as entrained metal hydroxides (also known as "sludge") are removed from the liquid using ultrafiltration in a conventional cross-flow filter. [2] The treated liquid is then processed to remove cesium. The CSSX process for removal of cesium from alkaline solutions uses a novel solvent invented at the Oak Ridge National Laboratory. [3, 4] This solvent consists of a calix[4]arene-crown-6 extractant (BOBCalixC6[®]) dissolved in an inert hydrocarbon matrix (Isopar[®] L). An alkylphenoxy alcohol modifier (1-(2,2,3,3-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol, also known as Cs-7SB) added to the solvent enhances the extraction power of the calixarene and prevents the formation of a third phase. An additional additive, trioctylamine (TOA), improves stripping performance and mitigates the effects of any surfactants present in the feed stream. The solvent extraction process was successfully demonstrated with actual SRS high level waste supernate during testing performed at SRNL by 2002. [5, 6] In February 2004, DOE commissioned construction of a smaller version (i.e., 11.4 – 34.1 L/min (3-9 gpm) processing rate for salt solution) of the final process for interim treatment of waste until construction of the full-scale Salt Waste Demonstration Facility completes. This smaller facility is known as the Modular Caustic Side Solvent Extraction (CSSX) Unit, or MCU. Figure 1 shows a schematic of the combined operations, known as the Integrated Salt Disposition Project.

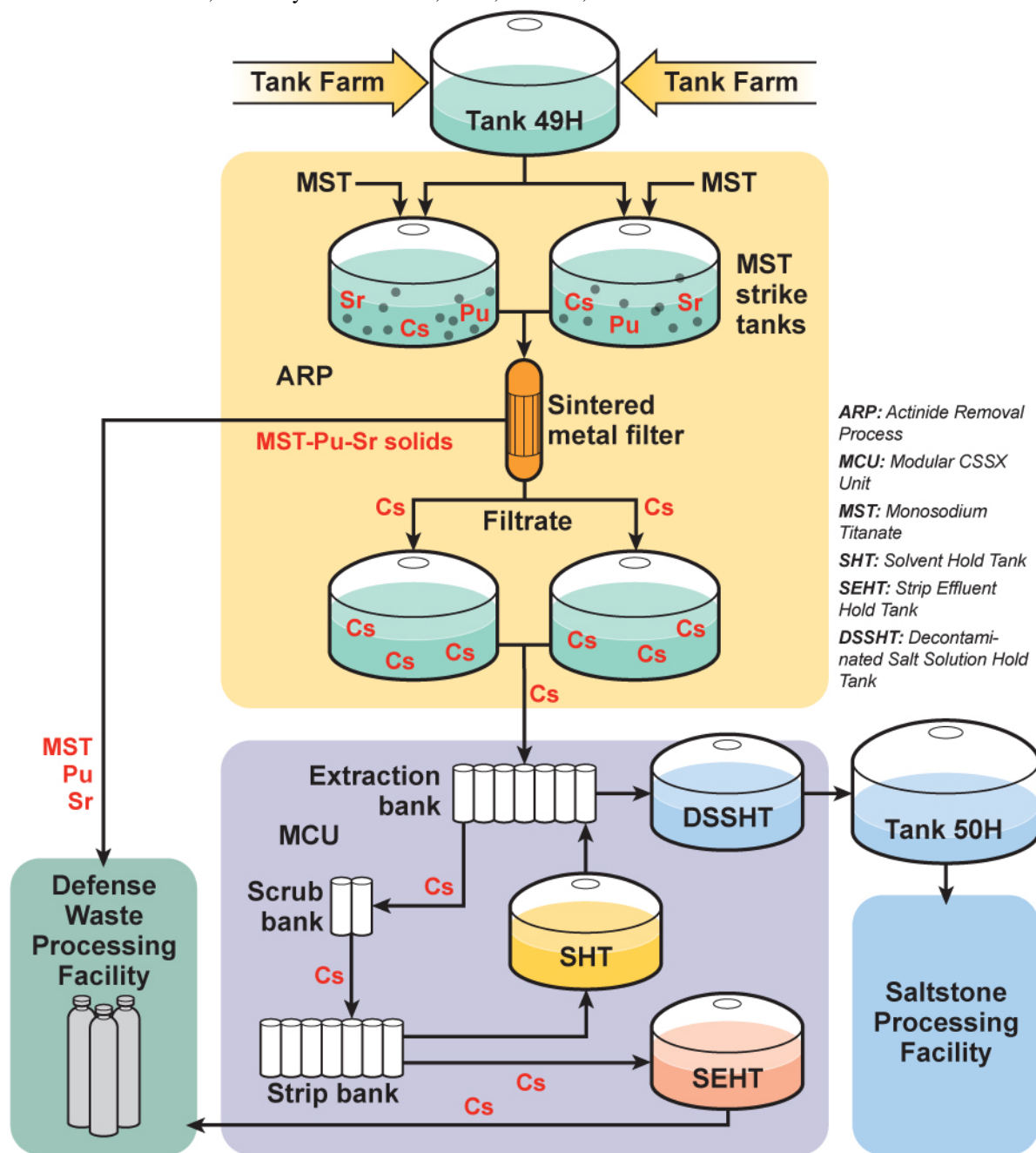


Fig. 1. Schematic of Integrated Salt Disposition Processing Operations Within Overall Waste Treatment System.

By June 2007, the integrated operations began using simulated salt solutions procured from vendors. By December of 2007, the first waste for processing was transferred and blended into Tank 49H. During December and into early January 2008, Operations personnel collected samples of waste from Tank 49H and transported them to SRNL for chemical analyses and process demonstrations to qualify the material for processing. After testing at SRNL, the first waste blend (“Macrobatches 1”) started in April 2008, and ended in September 2008 after processing ~140,000 gallons. A second Macrobatches of waste was tested at SRNL and processed in the plant from February 2009 to July 2010, processing ~730,000 gallons. Macrobatches 3 was tested at SRNL this summer and the plant started processing this material in June 2010, with an estimated processing goal of 1.1 million gallons processed. As of the end of December 2010, ~350,000 gallons have been process in Macrobatches 3. Samples of Macrobatches 4 are currently being tested at SRNL for future operations.

LABORATORY TESTING

Upon receipt of samples at SRNL, personnel combined and pulled aliquots of the various tank samples for an extensive suite of chemical and radionuclide analyses. Table I shows the measured data for key constituents for the composite of samples for Macrobatches 1-4. Values for all constituents represent the measurement on the as-received, combined sample.

Table I. Concentrations of Key Constituents in First Waste Feed Samples.

Analyte	Macrobatch 1	Macrobatch 2	Macrobatch 3	Macrobatch 4 a
Density (g/mL)	1.25	1.27	1.27	1.28
Na (M)	4.94	5.55	6.83	6.13
K (M)	0.00610	0.00621	0.0123	0.0110
Free Hydroxide (M)	0.613	2.14	2.68	2.05
Nitrate (M)	2.79	2.13	2.56	2.42
Cs-137 (Bq/L)	2.40E+09	1.98E+09	2.42E+09	1.76E+09
Pu-238 (Bq/L)	1.55E+06	9.81E+05	4.22E+05	NM
Sr-90 (Bq/L)	1.45E+07	1.08E+07	5.22E+06	6.96E+06

NM = not yet measured

a These values are from preliminary samples; confirmatory measurements are underway

The as-received waste had very few solids. The design assumption for the Actinide Removal Process is a concentration of ~600 mg/L of entrained solids; the measured value is only ~2% of the design assumption. The 600 mg/L design assumption equates to nearly equal amounts as the monosodium titanate added for each portion of waste treated. The facility treats multiple portions (or batches) of waste until reaching a calculated inventory of ~5 wt % solids in the monosodium titanate strike tank. At that time, the solids are washed to reduce the sodium concentration in the liquid and the accumulated solids transferred to the Defense Waste Processing Facility for vitrification. Since the waste contains very little sludge, the facility will process nearly twice the nominal amount of waste prior to requiring a transfer of the solids. A full description of the laboratory testing and chemical analysis of the waste are available in separate reports. [7,8,9,10,11,12]

Personnel added water and monosodium titanate (at 0.4 g of monosodium titanate solids per L of waste composite) to portions of the combined salt solution in proportions emulating facility operating conditions. After the addition of monosodium titanate, the researchers filtered samples of the solution at various time intervals (i.e., 6, 12 and 24 hours after monosodium titanate addition) and analyzed for plutonium (Pu) and strontium (Sr) concentrations in the liquid. A control experiment was also performed. After 24 hours, plutonium and strontium decontamination factors (DF- ratio of starting feed concentration versus concentration in the filtered liquid) were measured.

Table II. Laboratory Results from Monosodium Titanate Strikes on Macrobatch Samples.

Analyte	Macrobatch 1 (Bq/L)	Macrobatch 2 (Bq/L)	Macrobatch 3 (Bq/L)
Before MST Addition			
Pu-238	1.10E+06	9.10E+05	4.00E+05
Sr-90	8.99E+06	7.07E+06	3.65E+06
24 hours after MST Addition			
Pu-238	5.18E+04	1.61E+05	4.29E+04
Sr-90	1.52E+05	9.99E+04	5.18E+04
DF Values			
Pu-238	21.1	5.64	9.31
Sr-90	59.3	70.7	70.4

Plutonium DF varied from 5.64 to 21.1, and the strontium DF varied from 59.3 to 70.7. The Pu and Sr DF values agree reasonably well with predictions, 11 for Pu and 63 for Sr, using a previously published model and the

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measured concentrations of the feed. [13] The variances in the DF values are likely from differences bulk chemical composition, in the experimental procedures and in the analyte starting values.

For each macrobatch, personnel used the filtered liquid from the monosodium titanate test to examine the cesium mass transfer behavior with the solvent. They performed sequential batch contacts for the extraction, (two) acid scrubs, and (three) acid strip stages of the process. Table III contains the measured cesium distribution values and the acceptable ranges assumed for the design of the facility.

Table III. Cesium Distribution Values Measured in Sequential Batch Contacts for Extraction, Acid Scrub, and Acid Strip Stages of Process.

Stage	Extraction	Scrub #1	Scrub #2	Strip #1	Strip #2	Strip #3
Acceptable Range	>8	>0.6, <2	>0.6, <2	<0.2	<0.16	<0.16
Macrobatches 1	9.07	1.60	1.29	0.070	0.046	0.042
Macrobatches 2	14.64	1.51	2.13	0.74	0.09	0.031
Macrobatches 3	7.44	1.80	2.58	0.12	0.037	0.036

While most of the values are within acceptable ranges, we note that in several cases, Scrub#2 or Strip#1 is out of range. This is most likely due to pH swings caused by entrainment of variable amounts of salt solution into the scrubbing stage and has not been a matter of concern at MCU.

The low extraction value in Macrobatches 3 is a matter of concern. A repeat of the extraction part of the ESS test provided even poorer values. This is the first case in any Macrobatches or simulant test where the extraction value had dropped below acceptable limits. We theorized that poor mixing was the reason for the poor extraction value, and performed a set of three extraction tests (using the same solvent and Tank solution) designed to prove or disprove this theory. In one test, we provided no mixing at all. In two identical tests, we doubled the mixing time from 1 minute to 2 minutes and increased the vigor of mixing. Table IV lists the results from those tests.

Table IV. Cesium Distribution Values for New Mixing Tests

Test	Extraction Result
Original	7.44
Repeated	3.82
New – No Mixing	3.37
New – High Mixing	16.85
New – High Mixing	15.89

The results of these new mixing tests clearly show the importance of adequate mixing. The original Macrobatches 3 extraction test was poorly mixed, and the new mixing program provides extraction results that are some of the best we have seen in any ESS test, and ones that are close to the model calculated estimate of ~16.5. From this discovery, we have changed our procedures to ensure adequate mixing.

SUMMARY OF COMBINED ACTINIDE REMOVAL PROCESS /MCU FACILITY PERFORMANCE

Processing of the first actual tank waste began in May 2008. Since that time, the facility has completed two processing campaigns and started a third (Table V). The fourth campaign is already in preparation and qualification.

Table V. Macrobatches Processing Dates

Macrobatches	Time Frame	Material Processed (gal)
1	May – September 2008	140,000
2	February 2009 – June 2010	730,000
3	July 2010 – May 2011 (est)	~350,000 (as of 12/1/2010)
4	June 2011 (est)	To Be Determined

Even though each batch of material is thoroughly analyzed and tested before being used in the facilities, the output of the Actinide Removal Process /MCU must be monitored for quality control. This ensures that we are not transferring too much Cs-137, Pu-238 and Sr-90 to Saltstone (grouted low-level waste). Furthermore, routine testing of the strip effluent (SE – cesium loaded strip acid) for Isopar[®] L ensures that the quantity of combustible material sent to the Defense Waste Processing Facility is limited.

During Actinide Removal Process /MCU operations, semi-regular samples of the strip effluent and decontaminated salt solution (DSS – cesium depleted caustic salt solution) were taken and analyzed. Typically, SRNL was tasked with analyzing for Pu-238, Sr-90, Cs-137 and non-radioactive elements (Na, Al, etc), depending on the need.

Pu-238 Performance:

Plutonium removal is a function of the monosodium titanate performance at Actinide Removal Process and is not affected by the solvent extraction system at MCU. Figure 2 shows the Pu-238 in the DSS samples over the life of Macrobatch 1 processing, while Figure 3 shows the same for Macrobatch 2. There is not enough data yet available from Macrobatch 3 processing to present in this document. The SE sample data is not presented due to the low levels of plutonium present in the strip effluent. While Pu-239/240 is also present, the low activity of this isotope is harder to track than Pu-238. We have also plotted the range of blank results as these samples are prepared in a contaminated background.

Table VI lists the starting Pu-238 activity, the average final Pu-238 activity for the DSS samples, and the average decontamination factor (DF) from these values (starting ÷ final). This is a somewhat insensitive data set as we use the entire set of data knowing that early samples may not be at steady state. There is a large variation in the plutonium data – this is due to the fact that the monosodium titanate filter cake that accumulates in the strike tanks is irregularly transferred to DWPF. The longer the interval before the transfer, the more monosodium titanate is present, which results in a greater plutonium and strontium removal. Similarly, the batch contact time before transfer of the solution to MCU has a large variability due to facility outages and other factors.

Table VI. Pu-238 Removal from Tank Waste for Macrobatch 1 and Macrobatch 2

Macrobatch	Starting Pu-238 (Bq/L)	Average Final Pu-238 (Bq/L)	Average DF
1	1.55E+06	2.38(±2.59)E+03	650
2	9.17E+05	2.00(±1.56)E+04	45.9

For this main reason, the large apparent differences in the DF between Macrobatch 1 and 2 are misleading. A DF of 45.9 indicates a 97.9% removal of plutonium, while a DF of 650 indicates a 99.8% removal, which amounts to very small differences from a mass removal standpoint. The important point to consider is that in the facility, the plutonium removal is superior to what we measure in the laboratory.

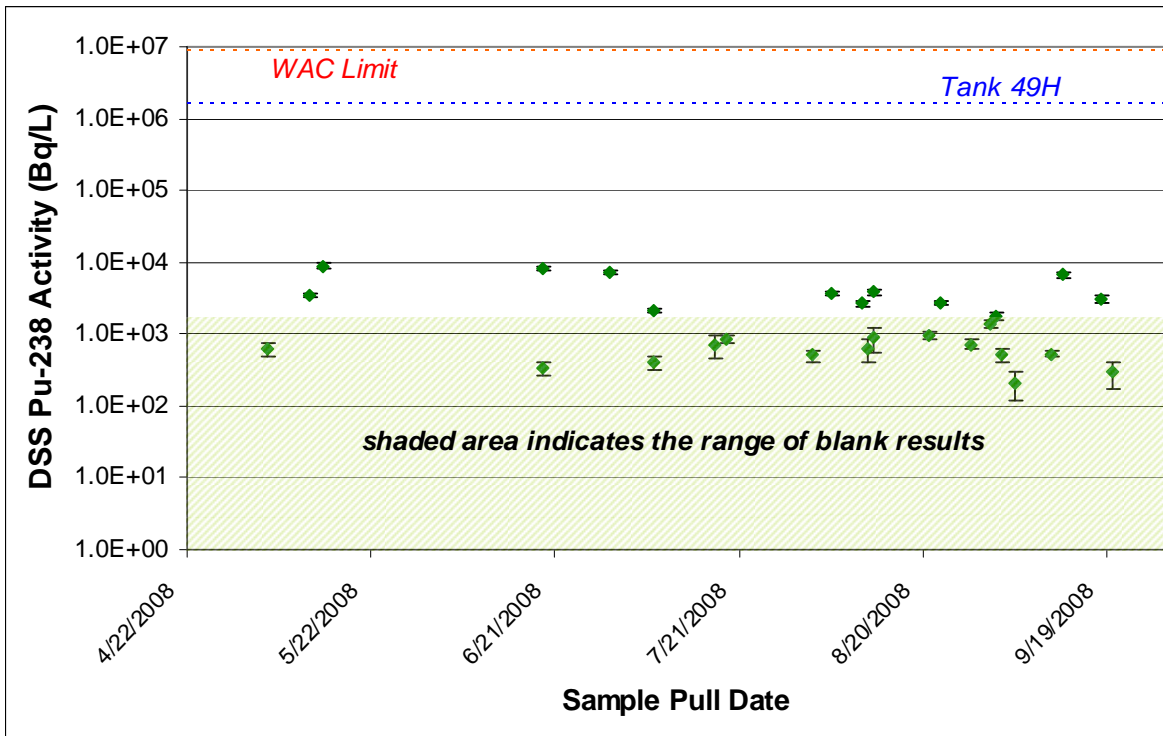


Fig.2. Plutonium-238 Concentrations in Macrobatch 1 Samples from Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank.

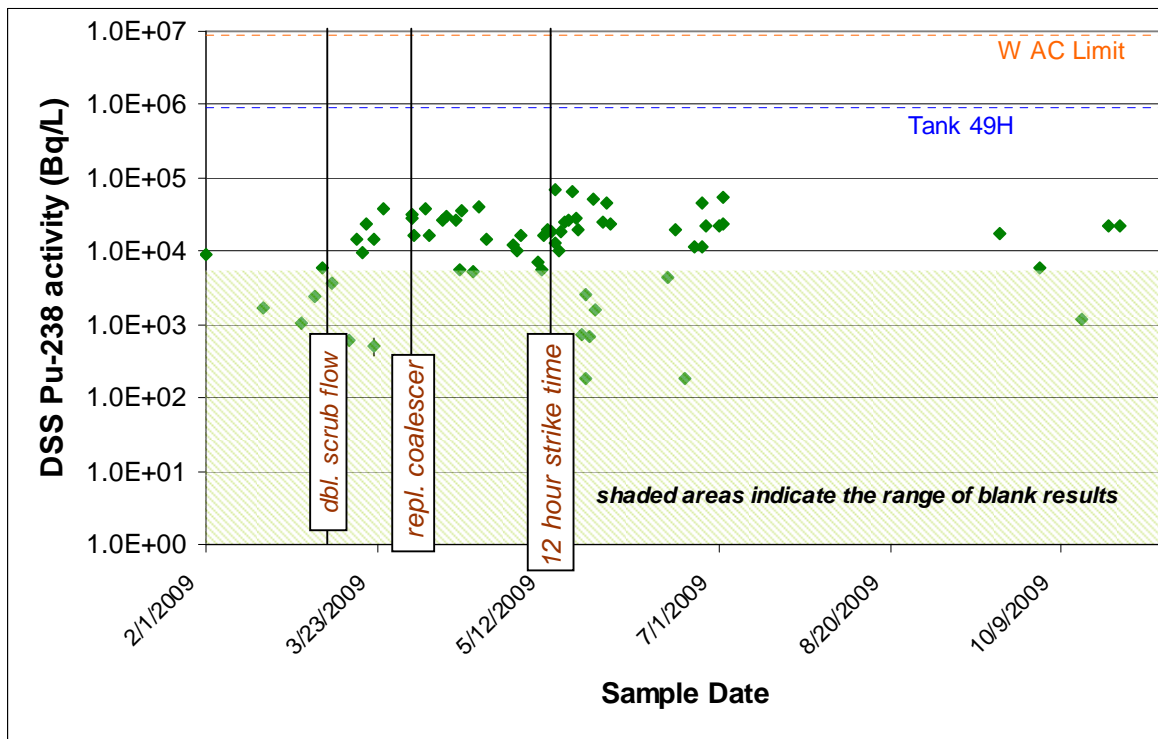


Fig.3. Plutonium-238 Concentrations in Macrobatch 2 Samples from Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank.

During Macrobatch 3, SRS varied several operating parameters; we doubled the scrub acid flow, changed out the strip effluent coalescer, and reduced the monosodium titanate strike time from 24 to 12 hours. None of these changes had an apparent effect on the plutonium removal. They are shown in Figure 3 for completeness.

Sr-90 Performance:

As with plutonium, the strontium removal is a function of the monosodium titanate performance at the Actinide Removal Process and is not affected by the solvent extraction system at MCU. Figure 4 shows the Sr-90 in the DSS samples over the life of Macrobatch 1 processing, while Figure 5 shows the same for Macrobatch 2. There is not enough data yet available from Macrobatch 3 processing to present in this document. The SE sample data is not presented due to the low levels of strontium present in the strip effluent.

Table VII lists the starting Sr-90 activity, the average final Sr-90 activity for the DSS samples, and the average decontamination factor (DF) from these values (starting ÷ final). This is a somewhat insensitive data set as we use the entire set of data knowing that early samples may not be at steady state. There is a large variation in the strontium data (although to a lesser extent than the plutonium data) – this is due to the same factors as described for the plutonium data.

Table VII. Sr-90 removal from tank waste for Macrobatch 1 and Macrobatch 2

Macrobatch	Starting Sr-90 (Bq/L)	Average Final Sr-90 (Bq/L)	Average DF
1	1.45E+07	3.52(±2.23)E+04	411
2	1.13E+06	6.07(±2.12)E+04	180

For this main reason, the large apparent differences in the DF between Macrobatch 1 and 2 are misleading. A DF of 180 indicates a 99.4% removal of plutonium, while a DF of 411 indicates a 99.8% removal, both of which represent very small differences from a mass removal standpoint. The important point to consider is that in the field, the plutonium removal is superior to what we measure in the laboratory.

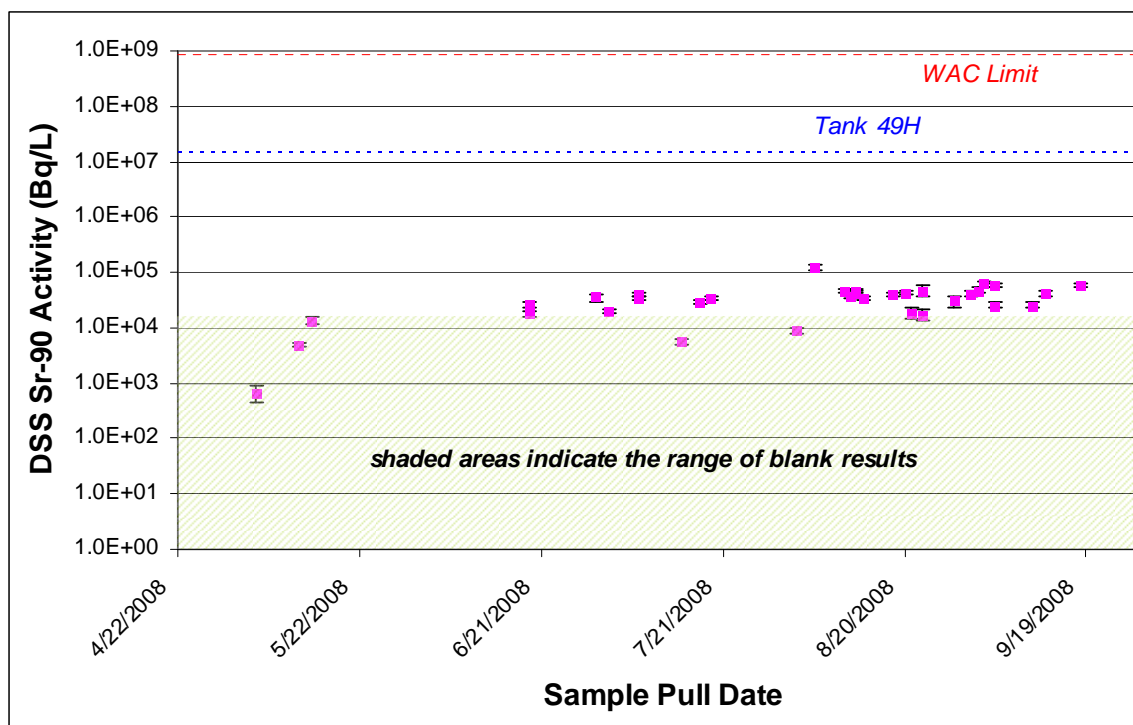


Fig.4. Strontium-90 Concentrations in Macrobatch 1 Samples from Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank.

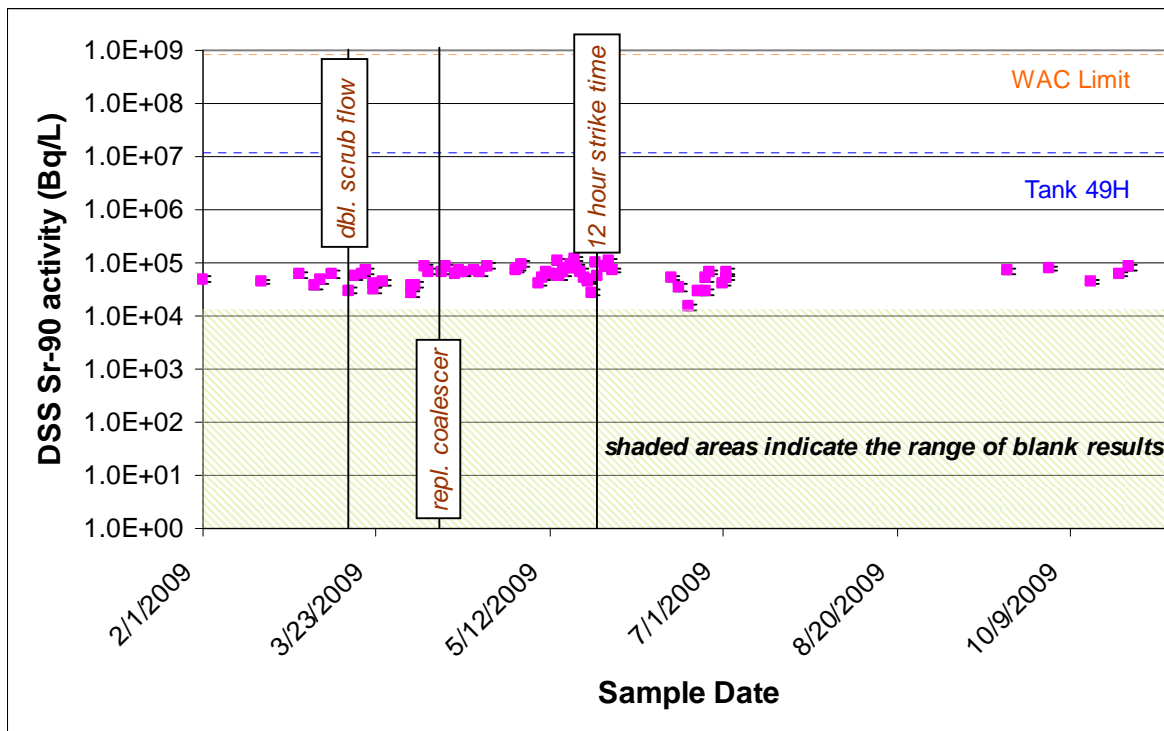


Fig.5. Strontium-90 Concentrations in Macrobatch 2 Samples from Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank.

During Macrobatch 3, SRS varied several operating parameters; we doubled the scrub acid flow, changed out the strip effluent coalescer, and reduced the monosodium titanate strike time from 24 to 12 hours. None of these changes had an apparent effect on the strontium removal. They are shown in Figure 5 for completeness.

Cs-137 Performance:

Cesium removal is a solely a function of the solvent extraction performance at MCU and is not affected by the monosodium titanate strikes at the Actinide Removal Process. Figure 6 shows the Cs-137 in the SE and DSS samples over the life of Macrobatch 1 processing, while Figure 7 shows the same for Macrobatch 2. There is not enough data yet available from Macrobatch 3 processing to present in this document.

Table VIII lists the starting Cs-137 activity, the average final Cs-137 activity for the SE and DSS samples, and the average decontamination factor (DF) from these values (starting ÷ final). This is a somewhat insensitive data set as we use the entire set of data knowing that early samples may not be at steady state.

Table VIII. Cs-137 Removal from Tank Waste for Macrobatch 1 and Macrobatch 2

Macrobatch	Starting Cs-137 (Bq/L)	Average Final SE Cs-137 (Bq/L)	Average Final DSS Cs-137 (Bq/L)	Average DF
1	2.40E+09	2.47(±1.23)E+10	3.57(±4.19)E+07	67.3
2	1.98E+09	1.37(±0.779)E+10	1.29(±0.882)E+07	153

The variations in the data set as well as the DF are due to several factors; the amount of starting cesium, the amount of potassium (interferes with cesium extraction), contact time, the presence of cold start-up heels, and solvent density. Therefore, the differences in the DF values between Macrobatch 1 and 2 should not be viewed as an indication of serious process changes.

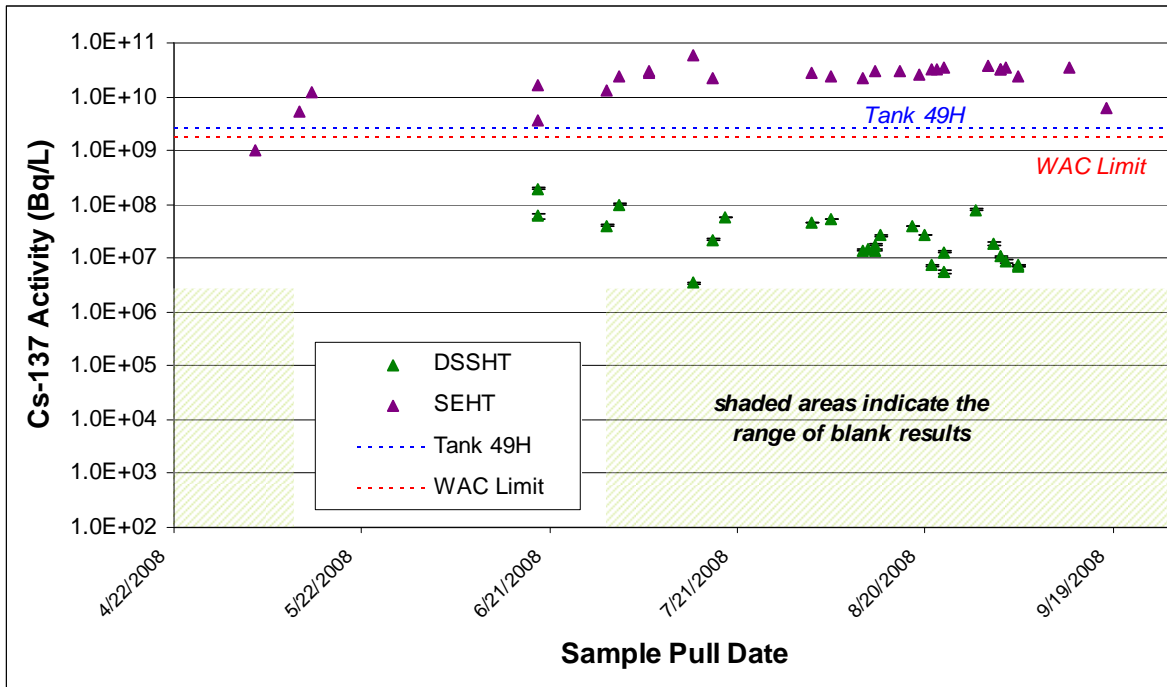


Fig.6. Cesium-137 Concentrations in Macrobatch 1 Samples from Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank.

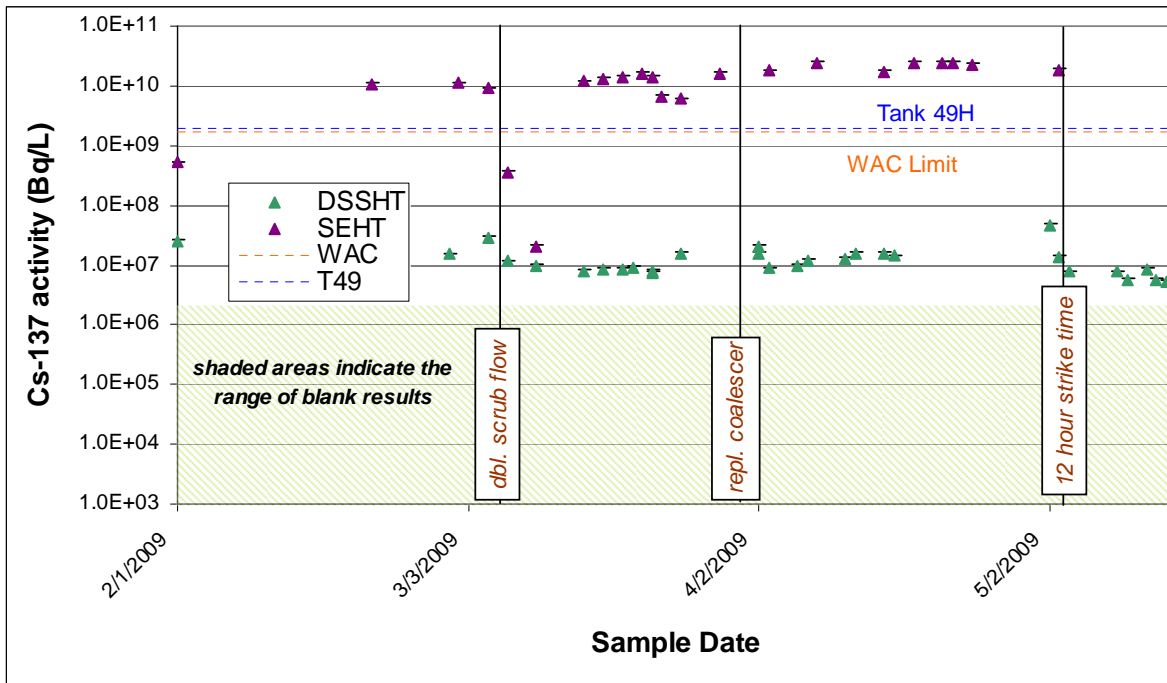


Fig.7. Cesium-137 Concentrations in Macrobatch 2 Samples from Decontaminated Salt Solution Hold Tank and Strip Effluent Hold Tank.

The cesium concentrations in the DSS samples decline over time, which is expected. The cesium concentration in the strip effluent acid correspondingly increases asymptotically over time also as expected. The ratio of the Cs in the SEHT to that of the feed is known as the Concentration Factor (CF). This ratio reaches a value of ~11 in Macrobatch 1 and ~12 in Macrobatch 2 compared to design expectations of 12-15.

SOLVENT QUALITY AND RECOVERY AT MCU

During operations, the Isopar[®] L evaporates from the solvent due to its volatility and the process ventilation flow. Operations personnel continuously monitor the density of the solvent to prevent the value drifting outside acceptable ranges for hydraulic operation of the centrifugal contactors. Also, solvent samples are periodically collected and transported to SRNL for analyses. During both Macrobatch 1 and 2, the facility added Isopar[®] L to replace evaporative losses. Once during each macrobatch, the facility also added whole solvent to replace slow, but unavoidable losses in the system (carryover into the aqueous streams, small leaks, etc). During Macrobatch 2, the solvent was allowed to evaporate to a degree higher than anticipated. This resulted in a stop-flow outage since the high-density solvent formed an apparent, slight emulsion with the aqueous stream. Our data indicates that solvent in the 0.89 g/mL or higher range can experience this problem. By adding Isopar[®] L back into the system and processing the solvent through the contactors, the emulsion can be cleared. To prevent further outages of this type, the facility has tightened the allowable density range for the solvent.

While the MCU facility is designed to minimize the amount of organic (mostly Isopar[®] L by volume) that leaves the system through both the SE and DSS aqueous streams, it cannot be entirely eliminated. Isopar[®] L if present in sufficient quantities can pose a flammability risk in downstream facility process vessels. The strip effluent acid and the decontaminated salt solution each pass through organic recovery equipment prior to exiting the facility. The recovery equipment consists of a coalescer flowed by a decanter tank with an included coalescer entrance stage. The equipment recovers the valuable solvent for use in the process and also minimizes transfer of Isopar[®] L to facilities receiving the aqueous effluents. The coalescer is a perforated stainless-steel cylinder (~1.5" dia.) wrapped with a continuous sheet of Ryton[®] - polyphenylene sulfide. The hydrophobic Ryton[®] surface collects small organic droplets in the aqueous streams and forces them into larger droplets, which aids in phase separation.

Samples from the SEHT and DSSHT for each batch are sent to another process support laboratory on site and analyzed to determine amount of entrained Isopar[®] L. There are tight operational limits to the concentration of Isopar[®] L allowed in the strip effluent, and the facility has had to stop or slow its operations during those times when excessive Isopar[®] L carryover is noted.

While the coalescer is reasonably efficient in removing organic from the aqueous streams, it is subject to fouling – a coalescer can be functionally similar to a filter. During Macrobatch 1 excessive pressure drops were noted in the DSS coalescer. Attempts to chemically clean the coalescer provided only limited benefit. Analysis of removed coalescer elements indicated that aluminum hydroxide or aluminosilicate solids were the culprit, which had formed from the combination of low free hydroxide, high silicon content and high aluminum content.

Transient issues were also noted in the SE coalescer. During Macrobatch 2, the experienced temporary high pressure drops. Attempts to chemically clean the SE coalescer were unsuccessful, but the problem vanished after a few weeks. Analyses of the removed coalescers are still underway.

LESSONS LEARNED

During operations, the MCU facility decontaminates the incoming waste stream to well within acceptable parameters. To date, operations have treated ~1.2 million gallons of salt solution. Physical upsets to plant operations occurred and can be attributed to two general causes. First, evaporation of the Isopar[®] L diluent slowly increases the density of the solvent causing hydraulic performance in the contactors to deteriorate. This problem has been countered by more closely monitoring the solvent density and composition, operating at lower average solvent density, and by occasional additions of Isopar[®] L to bring the solvent within specification.

Second, aluminum-containing solids that precipitate from pH swings or from a supersaturated feed foul the DSS coalescer. Several steps are in progress to minimize these upsets. The facility has added pre-filters prior to the DSS coalescer to collect the solids and increase the time between required replacements of the media. Furthermore,

personnel adjusted the composition of Macrobatches 2 and 3 by addition of sodium hydroxide solution to increase the free hydroxide concentrations to above 2 M.

The larger scale operations identified several problem areas, which we could not foresee in laboratory scale work. The scale-up lessons are valuable when doing flow sheet development work and will be utilized in the future Salt Waste Processing Facility that is currently under construction at the Savannah River Site.

CONCLUSIONS

SRS successfully treated ~1.2 million gallons of salt solution of radioactive waste through the Actinide Removal Process and the Modular Caustic Side Solvent Extraction Unit. Removal of plutonium and strontium by sorption on monosodium titanate exceeded expectations based on laboratory testing and confirmatory demonstrations. Cesium removal efficiency through the solvent extraction operations exceeded the expectations based on laboratory studies and model predictions. Using tank waste as a starting feedstock, SRS separated ~90 curies of Pu-238, ~730 curies of Sr-90, and ~147,000 curies of Cs-137 from the waste during the initial operations.

Fouling of the coalescer cartridges that remove the entrained solvent from the effluent aqueous streams is the most significant and persistent operational challenge, although design modifications to the DSS coalescer mitigated this fouling behavior. Treatment of Macrobatches 2 onward with sodium hydroxide in sufficient quantities has significantly reduced the aluminum solids formation. Studies of removed SE and DSS coalescer are still underway to proactively prevent future fouling issues not already mitigated.

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