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**INTERIM REPORT ON A MULTI-DAY
TEST OF THE CAUSTIC-SIDE
SOLVENT EXTRACTION FLOWSHEET
FOR CESIUM REMOVAL FROM A
SIMULATED SRS TANK WASTE**

by R. A. Leonard, S. B. Aase, H. A. Arafat,
D. B. Chamberlain, C. J. Conner,
M. C. Regalbuto, and G. F. Vandegriff



Argonne National Laboratory, Argonne, Illinois 60439
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April 2001

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ABSTRACT

A caustic-side solvent extraction (CSSX) process to remove cesium from Savannah River Site (SRS) high-level waste was tested for 71 hours in a 33-stage minicontactor (2-cm centrifugal contactor). This multi-day demonstration used an average SRS simulant for the waste feed. The two key process goals were achieved: (1) the cesium was removed from the waste with decontamination factors greater than 40,000, and (2) the recovered cesium was concentrated by a factor of 15 in dilute nitric acid. These goals were maintained for 71 h as 1.4 L of solvent was recycled 42 times at 14 mL/min while processing 180 L of SRS simulant at 43 mL/min. The average decontamination factor was 159,000 for cesium and the average concentration factor was 14.9. The process had to be shut down twice for minor problems, which were fixed and testing resumed. The results confirmed that the CSSX process could be used to help decontaminate the millions of gallons of SRS waste now stored in underground tanks.

I. INTRODUCTION

Savannah River Site (SRS) has 34 million gallons of high-level waste in 48 tanks that need to be decontaminated [LEVENSON-2000]. As a part of this process, the cesium will be removed from waste containing both supernatant liquid and dissolved salt cake, then vitrified for disposal. After the cesium is removed, the decontaminated solution will be immobilized in low-level grout.

Work performed at Argonne National Laboratory (ANL) in FY1998 showed that cesium can be extracted from caustic aqueous solutions representative of the high-level waste at SRS using solvent extraction carried out in centrifugal contactors [LEONARD-1999, -2001]. After additional work at Oak Ridge National Laboratory (ORNL) in FY1999 [BONNESEN-2000] to improve the solvent and at ANL in FY2000 [LEONARD-2000] to improve stage efficiency in

the 2-cm centrifugal contactor, the improved caustic-side solvent extraction (CSSX) flowsheet required for removing cesium from high-level waste at SRS was demonstrated with a waste simulant. Three proof-of-concept tests were performed [LEONARD-2000]. In these tests, the CSSX process was demonstrated both for once-through solvent operation and for solvent recycling up to four times in three hours. The improved CSSX process achieved both key process goals: (1) the cesium was removed from the waste with decontamination factors greater than 40,000, and (2) the recovered cesium was concentrated by a factor of 15 in dilute nitric acid.

The test reported here extends the above-mentioned work by carrying out a multi-day test of the CSSX flowsheet. In this test, 1.4 L of solvent was used to process 180 L of SRS simulant, so that the solvent was recycled a total of 42 times. The initial test plan was to operate at the nominal maximum throughput of the 2-cm contactor, which is 40 mL/min (both phases) and occurs in the extraction section, so that the SRS simulant feed rate would have been 28.7 mL/min and the aqueous strip feed rate would have been 1.90 mL/min. At these rates, it would take 105 h (4.4 d) to process the 180 L of simulant. This initial plan gave rise to the name “5-day test.” In subsequent hydraulic tests of the 2-cm contactor, we successfully used a maximum throughput of 60 mL/min (both phases) for the CSSX solvent and flowsheet conditions in the extraction section, allowing an SRS simulant feed rate of 43.0 mL/min and an aqueous strip feed rate of 2.85 mL/min. The higher feed rate for the aqueous strip feed is used, since experience indicates that higher flow rates improve stage efficiency [LEONARD-1999, -2000]. At these flow rates, it takes only 71 h (2.94 d) to process the 180 L of simulant while keeping the total number of solvent cycles, the desired experimental parameter, unchanged at 42.

This task is part of the integrated scope of work supporting the SRS High Level Waste Salt Processing Project (SPP). The work was performed in collaboration with Savannah River Technology Center (SRTC) and ORNL. Personnel at SRTC are performing tests with real waste, including batch solvent extraction and solvent irradiation. They are also carrying out a flowsheet test with real waste in a 2-cm centrifugal contactor. Personnel at ORNL are responsible for solvent development and commercialization. They are also evaluating the effect of heat and irradiation on the solvent so that solvent cleanup methods can be developed. Finally, ORNL personnel are measuring stage efficiency in a larger (5-cm) contactor to demonstrate that efficiency increases as the contactor size increases.

In the overall SPP work, the CSSX process is being compared with two alternative processes that also remove cesium from tank waste. These processes are (1) the tetraphenylborate (TPB) precipitation process, where TPB is used to precipitate out the cesium in small tanks, and (2) the crystalline silicotitanate (CST) ion exchange process, where CST is used

to remove the cesium in packed beds [LEVENSON-2000]. Before the end of FY2001, one of these three processes will be chosen as the basis for the cesium removal component of a plant designed to treat all the SRS tank waste.

II. EQUIPMENT AND MATERIALS

This solvent extraction was performed in a 33-stage 2-cm annular centrifugal contactor located in an ANL glovebox. The contactors, which are manufactured in banks of four stages, were built at ANL [ANL-1994]. The 33 contactor stages were modified to improve stage efficiency to $88 \pm 4\%$ as discussed elsewhere [LEONARD-2000]. In addition, a chiller bar was attached to the extraction stages to keep the temperature between 20 and 32°C. This was done because the distribution ratio for cesium is very sensitive to temperature, increasing with decreasing temperature. Thus, if the extraction section gets too hot, it will no longer be able to maintain the desired decontamination factor. The chiller bar was cooled by water recirculated from a cooling bath located outside the glovebox. A chiller bath temperature of 7.5°C was used to maintain an extraction section temperature of $26 \pm 2^\circ\text{C}$. The block temperatures for the strip section were $35 \pm 3^\circ\text{C}$. Since higher temperatures improve stripping, no chiller bar was needed.

Highly alkaline simulant for tank supernate waste was prepared using a method supplied by SRS personnel [PETERSON-2000] and is designated “average SRS simulant” or “SRS simulant”. The simulant was spiked with 0.5 mCi/L of Cs-137. Its composition is given in Table 1. The scrub feed was 0.05 M HNO₃, the strip feed was 0.001 M HNO₃, and the solvent wash feed was 0.01 M NaOH. All four aqueous feeds were prepared at ANL. The solvent, which was prepared at ORNL and shipped to ANL, had four components: (1) an extractant, a calixarene crown, calix[4]arene-bis(*tert*-octylbenzo-crown-6) designated BOBCalixC6, (2) a modifier, an alkyl aryl polyether, 1-(2,2,3,3,-tetrafluoropropoxy)-3-(4-*sec*-butylphenoxy)-2-propanol, also called Cs-7SB, (3) a suppressant, an alkyl amine, trioctylamine (TOA), which suppresses impurity effects to ensure that the Cs can be stripped from the solvent, and (4) a diluent, a mixture of branched hydrocarbons, Isopar[®]L. The solvent composition is 0.01 M BOBCalixC6, 0.50 M Cs-7SB, and 0.001 M TOA in Isopar[®]L and is designated the “CSSX solvent.”

Table 1. Composition of Average SRS Simulant

Component	Conc., mol/L	Component	Conc., mg/L
Na ⁺	5.6	Chromium	75
K ⁺	0.015	Ruthenium	0.82
Cs ⁺	0.00014 ^a	Palladium	0.41
OH ⁻	2.06	Rhodium	0.21
NO ₃ ⁻	2.03	Iron	1.44
NO ₂ ⁻	0.50	Zinc	8
AlO ₂ ⁻	0.28	Tin	2.4
CO ₃ ²⁻	0.15	Mercury	0.05
SO ₄ ²⁻	0.14	Lead	2.1
Cl ⁻	0.024	Silver	0.01
F ⁻	0.028	Tri-n-butyl phosphate (TBP)	0.5
PO ₄ ³⁻	0.007	Di-n-butyl phosphate (DBP)	25
C ₂ O ₄ ²⁻	0.008	Mono-n-butyl phosphate (MBP)	25
SiO ₃ ²⁻	0.03	n-Butanol	2
MoO ₄ ²⁻	0.000078	Formate	1500
NH ₃	0.001	Tri-methylamine	10
Copper	1.44 ^b		

^aThis is the total Cs concentration in the average SRS tank waste. It is the Cs concentration used here with the addition of Cs-137 at a tracer level of 0.5 mCi/L. The Cs-137 concentration in the average SRS tank waste is 22.6% of the total Cs concentration.

^bCopper is measured in mg/L.

III. FLOWSHEET TESTS

Using a 33-stage 2-cm centrifugal contactor with a stage efficiency of $88 \pm 4\%$, the CSSX process was tested using the flowsheet shown in Fig. 1. This flowsheet is different from that used earlier [LEONARD-2000] in that a solvent wash stage has been added: stage 33. The solvent wash stage is located next to the extraction section so that the organic strip effluent (EP) can be sampled directly to determine the effectiveness of the strip section in removing cesium from the solvent. In this test, designated "CS27", 1.4 L of CSSX solvent was used to process 180 L of SRS simulant with full solvent recycle for a total of 42 times. The results are compared to the key process goals: (1) a cesium decontamination factor greater than or equal to 40,000, and (2) a cesium concentration factor of 15.

The organic solvent was designed so that it will extract cesium from the aqueous SRS simulant as the two phases flow countercurrent to each other in the extraction section: as the solvent flows from stage 1 to 15 (see Fig. 1), its Cs concentration increases; as the SRS simulant flows in the opposite direction, from stage 15 to 1, its Cs concentration decreases. The effectiveness of the extraction section is defined by the decontamination factor, which is the concentration of cesium in the aqueous SRS simulant (DF) entering stage 15 divided by that in the aqueous raffinate (DW) exiting stage 1. Figure 2 shows that the decontamination factor in test CS27 met the goal throughout the test. The average decontamination factor for the test, $159,000 \pm 45,000$, exceeded the process goal by a factor of 4.

The solvent leaving the extraction section at stage 15 is loaded with essentially all the Cs that entered with the SRS simulant waste feed (DF). The solvent enters the scrub section, stages 16 and 17 in Fig. 1, where weakly extracted impurities are removed along with any entrained aqueous phase. In addition, the entrained alkaline aqueous phase is neutralized and made slightly acidic. The scrubbed solvent then enters the strip section at stage 18 where all of the Cs is stripped from the solvent into the aqueous phase and comes out into the aqueous strip effluent (EW). Since the flow of the aqueous strip feed (EF) is 1/15 of the aqueous waste feed (DF) flow and since essentially all of the Cs entering in the DF feed goes out in the aqueous strip effluent (EW), the Cs is concentrated by a factor of 15 in the EW effluent. This $[EW]/[DF]$ ratio, called the concentration factor, is plotted in Fig. 3 as a function of time for test CS27. The average value of the concentration factor is 14.9 ± 1.1 , which meets the second process goal. At 1043 min into test CS27, the waste (DF) feed rate was determined to be low and was increased by 10%. The average concentration factor was 13.8 ± 0.4 before this change, and 15.7 ± 0.7 afterwards. These results show clearly how the concentration factor depends on the waste (DF) and strip (EF) feed rates and is controlled by them.

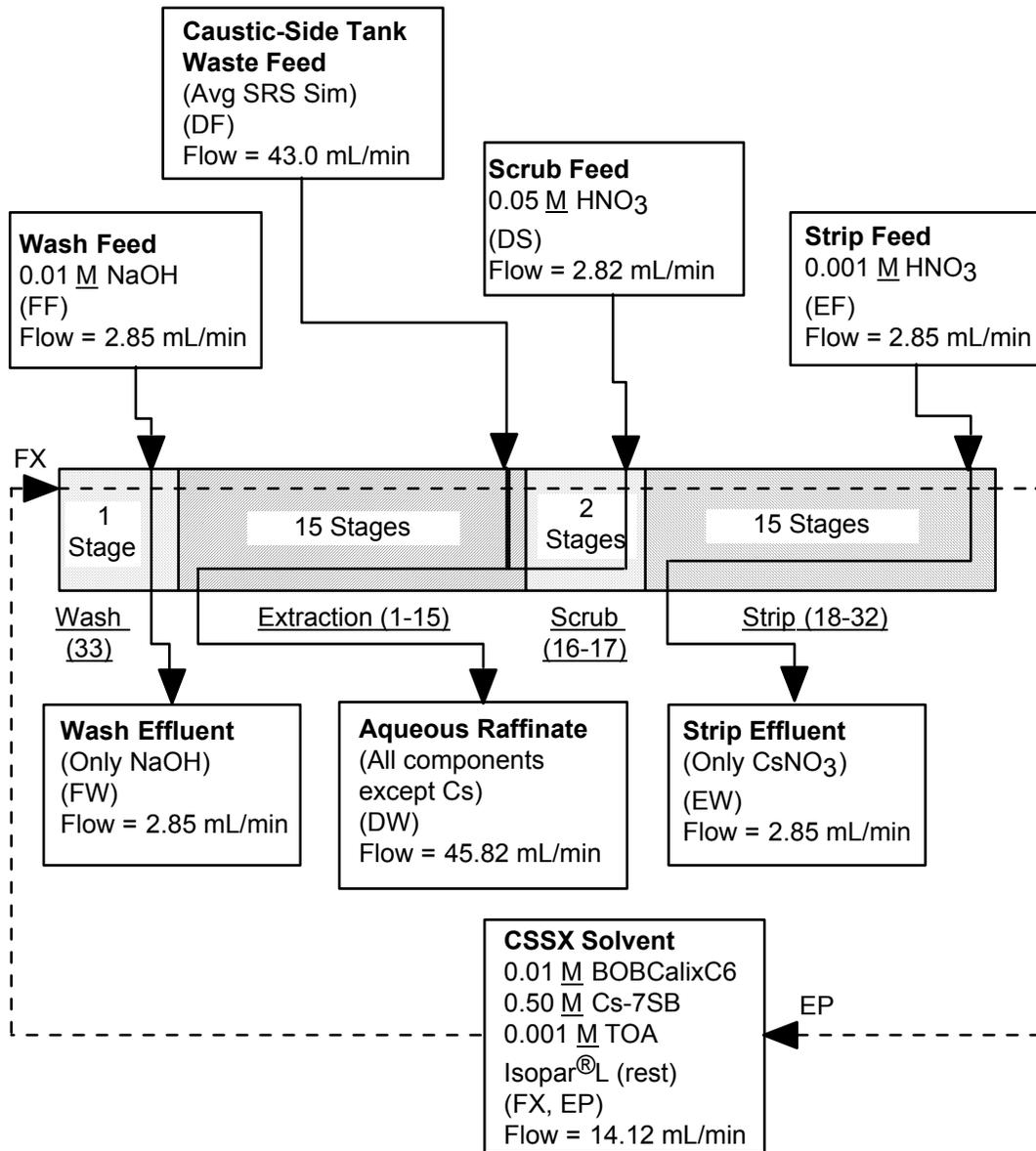


Fig. 1. CSSX Flowsheet for Solvent Recycle Test (CS27). Nominal flow rates are shown.

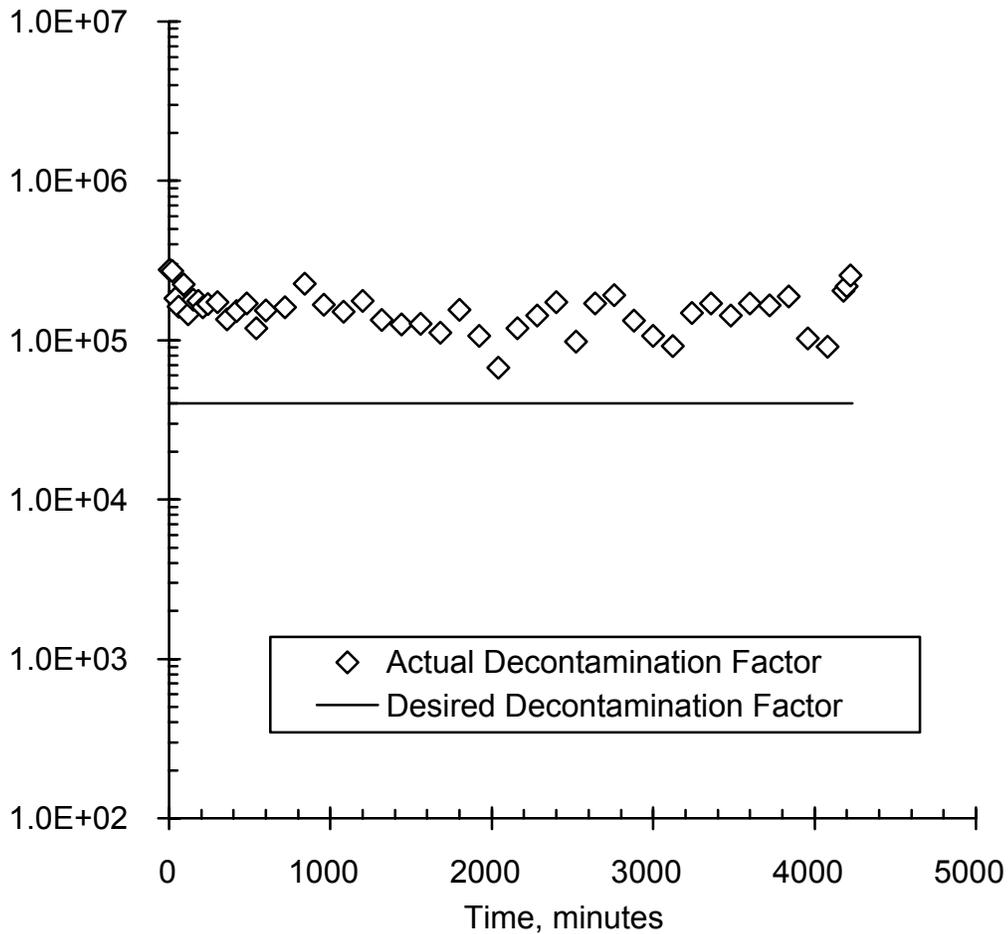


Fig. 2. Changes in the Decontamination Factor vs Time for Test CS27

As the organic solvent flows through the strip section, it moves countercurrent to the aqueous strip solution of 0.001 M HNO₃. This solution was chosen because it effectively strips the cesium from the solvent: as the solvent flows from stage 18 to 32 in Fig. 1, its Cs concentration decreases, and as the aqueous strip solution flows in the opposite direction, from stage 32 to 18, its Cs concentration increases. The effectiveness of the strip section is measured by the stripping factor, which is the Cs concentration in the aqueous SRS simulant (DF) entering stage 15 divided by that in the organic effluent (EP) exiting stage 32. Figure 4 shows that the stripping factor in test CS27, which has the same process goal as the decontamination factor so that the solvent can be recycled, met this goal throughout the test. The average stripping factor for the test, $137,000 \pm 46,000$, exceeded the process goal by a factor of 3.2.

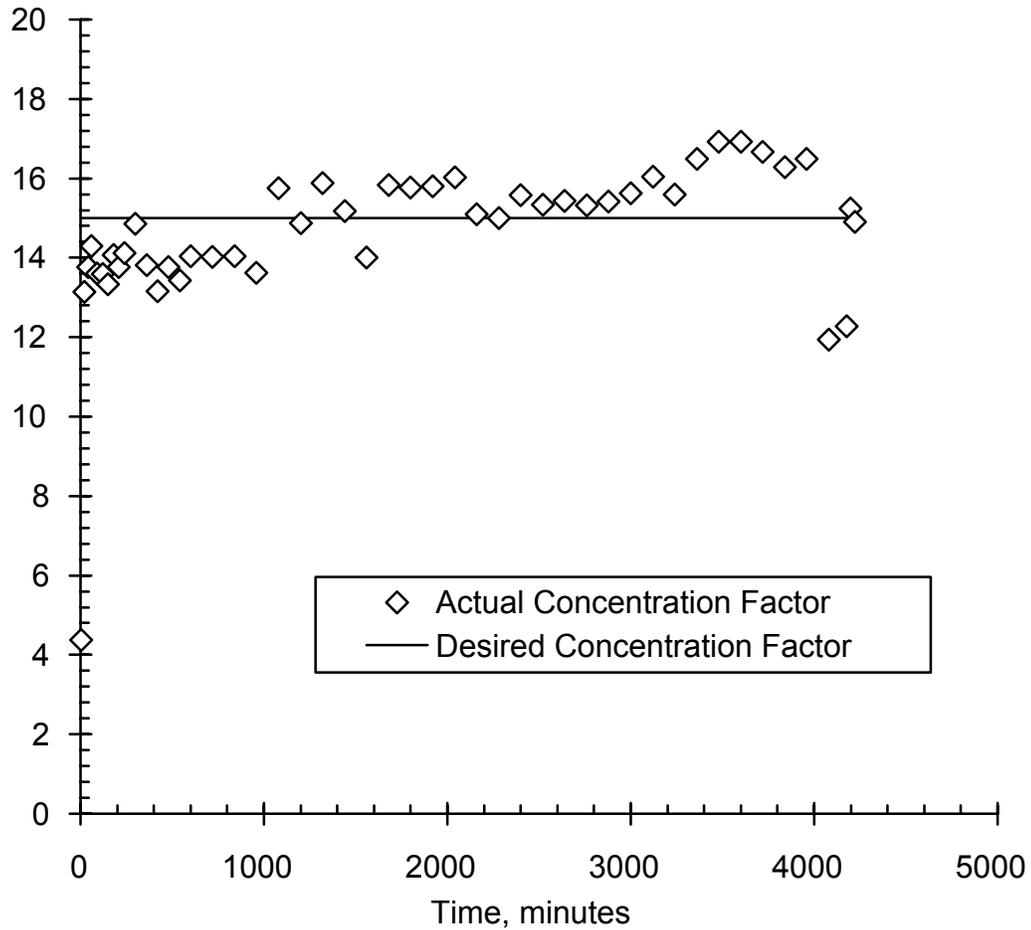


Fig. 3. Changes in the Concentration Factor vs Time for Test CS27

At 956 min into the test, the test was stopped for about one hour to replace the pump that transferred the DF feed from the DF feed drum outside the glovebox into the DF feed tank inside the glovebox. Then, a new feed line was installed from the pump into the DF feed drum, and the test was restarted. As Figs. 2–4 show, this shutdown and restart had no effect on the key process parameters.

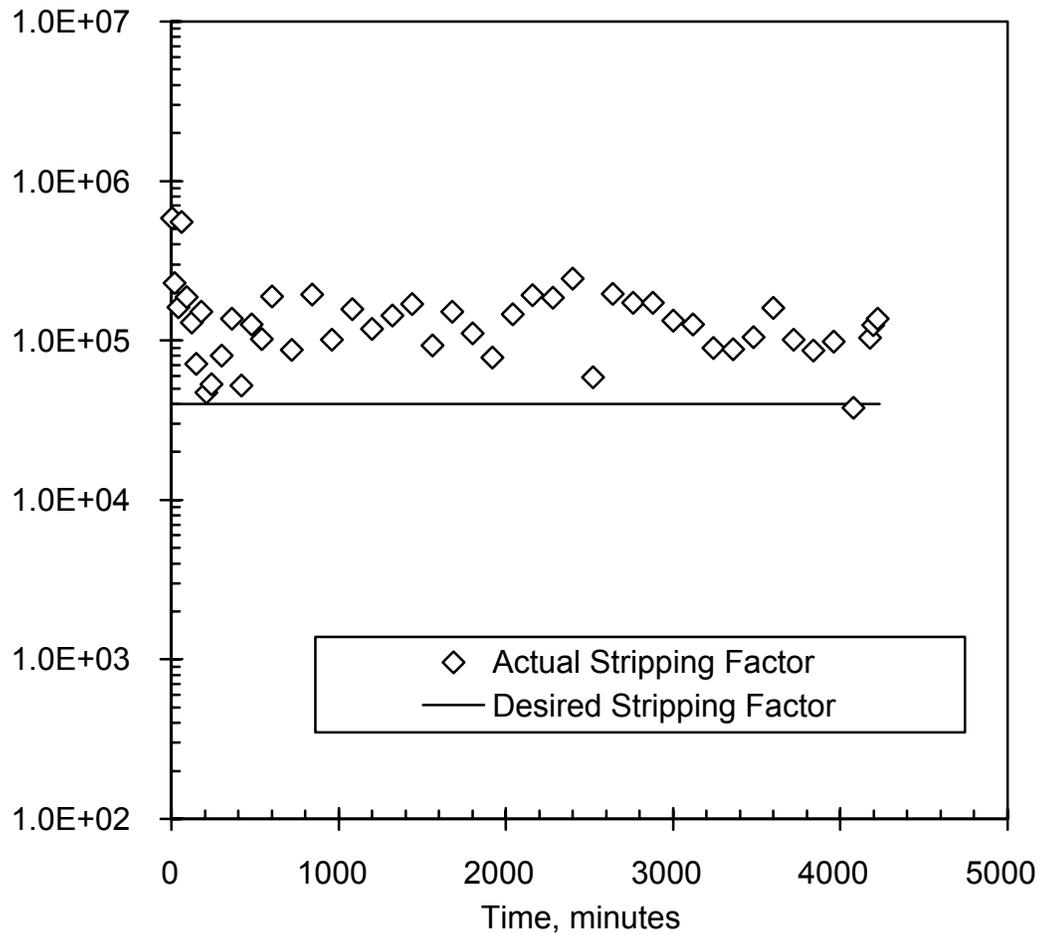


Fig. 4. Changes in the Stripping Factor vs Time for Test CS27

At 4080 min into the test, the EW effluent was found to be slightly alkaline, with a pH of 9. The Cs concentration in the solvent coming out in the EP effluent appeared unchanged. The DS feed rate was doubled to try to reduce the pH in the EW effluent, but it did not work. About one hour later, a solvent (EP) effluent sample had a Cs concentration about 1/2 that of the DF feed. At this point, 4160 min into the test, the test was stopped for a second time. Sufficient solids were found in the stage 15 rotor (where the DF feed enters the contactor extraction section) to cause some of the SRS simulant to be swept with the organic phase into the scrub section. The aqueous liquid in the first scrub stage (stage 16) had about the same yellow color as the SRS simulant in stage 15, indicating much more simulant than scrub solution in that stage. The aqueous liquid in the second scrub stage (stage 17) had about 1/2 the yellow color of the SRS simulant in stage 15. There were some solids in both scrub stages. There were more solids in extraction stages 13 and 14 than in the scrub stages, but not as much as in the SRS simulant

feed stage, stage 15. The solids were on the inside wall of the rotors, but not in the mixing zone of the contactor. An elemental analysis of the solids showed the presence of sodium, aluminum, and sulfur. X-ray diffraction (XRD) analysis indicated the solids were crystalline and contained many components. The main components identified were NaNO_3 (~80%), sodium aluminosilicate (also known as sodium montmorillonite, a clay, at ~15%), Na_2SO_4 (~5 to 10%), and $\text{Al}_2(\text{SO}_4)_3 \cdot 5\text{H}_2\text{O}$ (~5%). Other possible components were NaCl and $\text{NaAl}_3(\text{SO}_4)_2(\text{OH})_6$ (also known as natroalunite or alunite). In the sodium-containing components, K may sometimes replace the Na. Several weeks after the test, the residual liquid in the DF feed tank was filtered and these solids were also analyzed using XRD. They were found to have the same major components identified from the solids in the rotor as well as traces of two additional ones, $\text{Na}(\text{AlO}_2)_9(\text{SiO}_2)$ and $\text{NaAlO}_4\text{Al}_{12}(\text{OH})_{24}(\text{H}_2\text{O})_{12}(\text{SO}_4)_2$, both having variable H_2O of hydration. These new solids may have precipitated during the four weeks that the residual feed solution sat in the DF feed tank after the 5-day test. Thus, it appears that the solids in the contactor rotors formed before the waste (DF) feed reached the contactor.

Two steps were required to solve the problems caused by the solids. First, they were dissolved from the rotors by dipping each rotor containing solids into a beaker filled with 2 M HNO_3 . Then the Cs concentration in the strip section and that in the solvent feed tank were decreased by running the process without the DF feed for 5 hours. When the Cs concentration in the DW effluent was sufficiently low, we resumed the test. The remaining feed was then processed, with complete restoration of the decontamination factor and concentration factor. The test was over at 4233 min when all of the 180 L of SRS simulant waste had been processed.

Thus, test CS27 met both process goals, that is, a decontamination factor >40,000 and a concentration factor 15, while recycling the solvent 42 times over a test time of 71 hours. No evidence of solvent degradation was observed based on the relatively constant value for the decontamination factor and the stripping factor over the entire 71 hours of the test. A sample of the recycled solvent was sent to ORNL where a detailed chemical analysis will be done to determine whether any degradation products may have formed in the solvent over the 42 cycles. Results from test CS27 were used to guide and evaluate the test with real waste at SRTC.

IV. DISCUSSION

The data for the concentration factor can be used to examine the rate at which the process approaches steady state. The rate at which this factor approaches steady state is controlled by the rate at which the DF feed brings cesium into the contactor. The concentration factor was 4.4 at 5 min into the test and 13.1 at 25 min. At 4160 min into the test, when the contactor was

being restarted after all the Cs had been flushed out, the concentration factor reached 12.3 at 18 min into the restart. At 38 min, the concentration factor was 15.2. Thus, the CSSX process run in centrifugal contactors reaches steady state in 25 to 30 min.

When the average concentration factors were calculated, the two low data points that occurred during the two startup periods were omitted from this calculation. Also omitted was the low concentration factor at 4080 min, which was probably caused by the solids accumulation in stage 15. However, these three concentration factors are included on Fig. 3. When the average stripping factor was calculated, the low stripping factor at 4080 min was omitted for the same reason. The data points for the first hour were also omitted from the average stripping factor as they were all higher than the average. During startup, the stripping factor will be greater than the average value, so this indicates the stripping section took more than one hour to reach steady state. This is not unexpected, since we were only up to 1.4 residence times with respect to the aqueous strip flow rate. This residence time was calculated using the aqueous strip flow rate of 2.85 mL/min, the aqueous-phase volume of 8 mL/stage in the strip section, and the 15 strip stages. While these stripping factors were omitted from the average value, they are included on Fig. 4. No data points were omitted when calculating the average decontamination factor.

The solids that entered with the DF feed at stage 15 had precipitated from the SRS simulant in the 10 to 12 days between the time when the simulant was filtered and the test started. Since the DF inlet line went to the bottom of the DF feed drum, solids would have been picked up around the feed point at time zero. When the transfer pump for the DF feed drum to the glovebox failed at 956 min, a new feed pickup line was put in the drum and there would have been picked up solids at a second point. The addition of 22 L of DF feed to the 13 L of DF feed left in the DF feed drum starting at 3459 min and continuing for 2 h would have stirred up more solids and suspended them in the DF feed. Thus, there were three inadvertent transfers of solids into the contactor. After the test, solids were found in the DF feed tank in the glovebox and in the line from the DF feed drum to the DF transfer pump. The result was that enough solids eventually were fed into the contactor at stage 15 to cause the aqueous underflow in the rotor at stage 15 to become partially blocked. When this happened, some of the aqueous phase (SRS simulant) was forced over the organic weir of the rotor along with the solvent into stage 16, the first scrub stage. This highly alkaline solution in the scrub section would overwhelm the dilute nitric acid and eventually make the first strip stage, stage 18, alkaline as well. This alkaline strip stage created conditions that prevented most of the cesium from being stripped from the solvent. This allowed solvent with a high Cs concentration to move through the strip section and was the reason that we stopped to dissolve solids from stage 15 and clear cesium from the strip section.

As stated above, we were able to dissolve the solids out of the rotors, clear the excess cesium from the strip section, and resume normal process operation. This test demonstrated the importance of (1) trying to keep solids out of the contactor, (2) monitoring the pH in the scrub section or the aqueous strip (EW) effluent to detect when solids could be forcing some of the aqueous phase out of the waste (DF) feed stage along with the organic phase, and (3) having a plan for responding to solids transfer.

In the test done here, the solvent was recycled every 1.65 hours. In a process plant with 3785 L (1000 gal) of solvent and a solvent flow rate of 25.0 L/min (6.6 gpm), the solvent will be recycled every 2.53 hours. Thus, the 42 solvent cycles after 2.94 d of operation in the minicontactor test reported here is equivalent to the solvent recycle for 4.49 d of plant operation.

V. SUMMARY AND CONCLUSIONS

A caustic-side solvent extraction process to remove cesium from SRS high-level waste was tested for three days in a 33-stage 2-cm centrifugal contactor. This multi-day demonstration of CSSX flowsheet was done using an average SRS simulant for the waste feed. We achieved and maintained the two key process goals: (1) the cesium was removed from the waste with decontamination factors greater than 40,000 and (2) the recovered cesium was concentrated by a factor of 15 in dilute nitric acid. In the multi-day test, 1.4 L of solvent was recycled a total of 42 times used while processing 180 L of SRS simulant. The cesium had an average decontamination factor of 159,000 and an average concentration factor of 14.9.

The process had to be shutdown twice. Both times it was restarted easily. The first time, we had to replace a feed pump. This did not cause in any change in the process, which was restarted at essentially steady-state conditions. The second time, with 1 hour and 20 minutes remaining until the end of the test, solids from the SRS simulant partially plugged the rotor where the simulant was being fed, forcing some of the alkaline aqueous phase into the scrub section and making the first strip stage alkaline as well. This destroyed the ability of the strip section to remove cesium from the solvent. After the solids were dissolved out of the contactor rotors and the high cesium concentration was cleared from the strip section and the solvent recycle tank, the test was restarted and continued until the SRS simulant was gone. In the three samples taken after recovering from the solids problem, the system was found to have the same high decontamination factor as before.

This multi-day test showed the ability of the solvent to be used for at least 42 solvent recycles, the ability to recover from process upsets, and the importance of keeping solids out of the contactor. After 42 solvent cycles, there was no indication that the solvent had lost any of its ability to remove and concentrate cesium.

ACKNOWLEDGMENTS

This work was supported by the Office of Environment Management of the U.S. Department of Energy through (1) the Office of Project Completion and (2) the Tank Focus Area of the Office of Science and Technology under Contract W-31-109-Eng-38 with Argonne National Laboratory, managed by the University of Chicago.

The authors wish to acknowledge the help of John Falkenberg, Frank Markun, Del Bowers, Kevin Quigley, Manuel Almazan, Allen Bakel, Jim Byrnes, Jeff Emery, Joe Hirsch, and John Basco, all of the ANL Chemical Technology Division, in preparing for and carrying out the test. Test observers were Leon Klatt of ORNL and Seth Campbell of WSRC. Bob Finch of the ANL Chemical Technology Division did the XRD analysis.

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