INEEL/CON-99-00092
PREPRINT

Demonstration of a Universal Solvent Extraction Process for the Separation of Cesium and Strontium from Actual Acidic Tank Waste at the INEEL

IDAHO NATIONAL ENGINEERING & ENVIRONMENTAL LABORATORY

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August 29, 1999 – September 3, 1999

Global '99

LOCKHEED MARTIN

DEMONSTRATION OF A UNIVERSAL SOLVENT EXTRACTION PROCESS FOR THE SEPARATION OF ACTINIDES, CESIUM, AND STRONTIUM FROM ACTUAL ACIDIC TANK WASTE AT THE IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

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ABSTRACT

A universal solvent extraction process is being evaluated for the simultaneous separation of Cs, Sr, and the actinides from acidic high-activity tank waste at the Idaho National Engineering and Environmental Laboratory (INEEL) with the goal of minimizing the high-activity waste volume to be disposed in a deep geological repository. The universal solvent extraction process is being developed as a collaborative effort between the INEEL and the Khlopin Radium Institute in St. Petersburg, Russia. The process was recently demonstrated at the INEEL using actual radioactive, acidic tank waste in 24 stages of 2-cm diameter centrifugal contactors located in a shielded cell facility.

With this testing, removal efficiencies of 99.95%, 99.985%, and 95.2% were obtained for ^{137}Cs, ^{90}Sr, and total alpha, respectively. This is sufficient to reduce the activities of ^{137}Cs and ^{90}Sr to below NRC Class A LLW requirements. The total alpha removal efficiency was not sufficient to reduce the activity of the tank waste to below NRC Class A non-TRU requirements. The lower than expected removal efficiency for the actinides is due to loading of the Ph_2Bu_2CMPO in the universal solvent with actinides and metals (Zr, Fe, and Mo). Also, the carryover of aqueous solution (flooding) with the solvent exiting the actinide strip section and entering the wash section resulted in the recycle of the actinides back to the extraction section. This recycle of the actinides contributed to the low removal efficiency. Significant amounts of the Zr (>97.7%), Ba (>87%), Pb (>98.5%), Fe (6.9%), Mo (19%), and K (17%) were also removed from the feed with the universal solvent extraction flowsheet.

I. INTRODUCTION

The Idaho Nuclear Technology and Engineering Center (INTEC), formerly known as the Idaho Chemical Processing Plant (ICPP), is the only facility storing high-activity waste at the INEEL. Approximately five million liters of aqueous acidic high-activity waste, known as sodium-bearing waste (SBW), are currently on inventory at the INTEC. This waste was derived primarily from solvent washing operations in the uranium recovery process and equipment decontamination activities. The INTEC is no longer recovering uranium; therefore, waste from this process is no longer being generated.

However, waste generation from decontamination activities and daily plant operations is continuing.

The use of a single process to remove the desired radionuclides, as opposed to a combination of different unit operations that remove these same radionuclides, evolved from previous collaborative work with scientists from the Khlopin Radium Institute in St. Petersburg, Russia.[1] The possibility of using a universal solvent containing chlorinated cobalt dicarbollide with polyethylene glycol (PEG) to remove cesium and strontium, and a carbamoylmethyl phosphine oxide derivative to remove the TRU's was discussed early in FY-95. A proposal to investigate such a solvent was submitted to and accepted by the Environmental Management (EM-50) Efficient Separations and Processing CrossCutting Program. A process based on a universal solvent may provide a more simple and cost effective method for waste treatment than a method that utilizes two or three separate processes. Batch contact testing of the universal solvent was performed in 1997 using actual INTEC SBW and a countercurrent flowsheet test using 26 stages of 3.3-cm diameter centrifugal contactors and simulated tank waste was performed in 1997, both with very positive results.[2] Based on this testing, a countercurrent flowsheet was developed for demonstration using actual INTEC SBW in a centrifugal contactor pilot plant located in a shielded cell facility. This paper will discuss the results of this flowsheet testing.

II. EXPERIMENTAL

Based on the results of universal solvent development studies performed at the Khlopin Radium Institute and at the INEEL, a flowsheet was developed and recommended for countercurrent flowsheet testing in 2.0-cm diameter centrifugal contactors. This flowsheet, as shown in Figure 1, consists of eight stages of extraction, two stages of scrub, six stages of Cs/Sr strip, three stages of actinide strip, and five stages of solvent wash.

Flowsheet testing was performed using 2.0-cm diameter centrifugal contactors designed and manufactured by Argonne National Laboratoy. The 2.0-cm Centrifugal Contactor Pilot Plant consists of 24 stages of 2.0-cm diameter centrifugal contactors, feed and receiving vessels, feed pumps, and an air purge system for the contactor bearings. The aqueous and organic feed pumps and feed vessels were located inside the shielded cell. The remaining feed pumps and feed vessels were located outside the cell. All of the feed pump controllers were located outside the cell. Non-radioactive solutions used for the flowsheet testing were pumped to the centrifugal contactors through penetrations in the cell wall. The centrifugal contactors operate at a rotor speed of 3,600 rpm.

Approximately 1.6 liters of actual INTEC waste solution, obtained from tanks WM-183 and WM-185 in 1997 was used as feed solution for the flowsheet testing. A 50:50 vol. % mixture of waste from these two tanks was used. The feed solution was filtered through a 0.45 micron filter. The chemical composition of the WM-183/185 waste is shown in Table 1. For the flowsheet demonstration with tank waste, 18 mL of 10 *M* HF was added to the 1.6 L of feed to complex the Zr in the feed and minimize the extraction of Zr. Upon adding the HF to the feed, a white precipitate was immediately noticed. The precipitate re-dissolved in the feed after several minutes of shaking the feed bottle. The feed was then re-filtered through a 0.45 micron filter and sampled. Analyses indicate that the composition did not change significantly as a result of the adjustment and filtering.

Table 1. WM-183/185 tank waste composition.

Component	WM-183/185	Component	WM-183/185
Acid (*M*)	1.55	Na (*M*)	1.14
Al (*M*)	0.68	NO_3 (*M*)	4.38
B (*M*)	0.016	Zr (*M*)	0.0054
Ba (*M*)	3.4E-05	Alpha(nCi/g)	473
Ca (*M*)	0.049	^{241}Am (nCi/g)	54
Cr (*M*)	0.011	^{134}Cs (Ci/m^3)	0.16
F (*M*)	0.13	^{137}Cs (Ci/m^3)	185
Fe (*M*)	0.038	^{238}Pu (nCi/g)	343
Pb (*M*)	0.0016	^{239}Pu (nCi/g)	71
Hg (*M*)	0.0041	^{99}Tc (Ci/m^3)	0.034
Mo (*M*)	0.012	^{90}Sr (Ci/m^3)	181
K (*M*)	0.15	U (g/L)	0.087

The flowsheet demonstration was performed as follows. Each of the centrifugal contactors was filled with 15 mL of process solution by pumping the appropriate solution into each stage through the overflow ports. One molar HNO_3 was used for the stages in the extraction section.The centrifugal contactor motors were then started at 3,600 rpm. Solvent flow was established. When solvent began exiting contactor stage

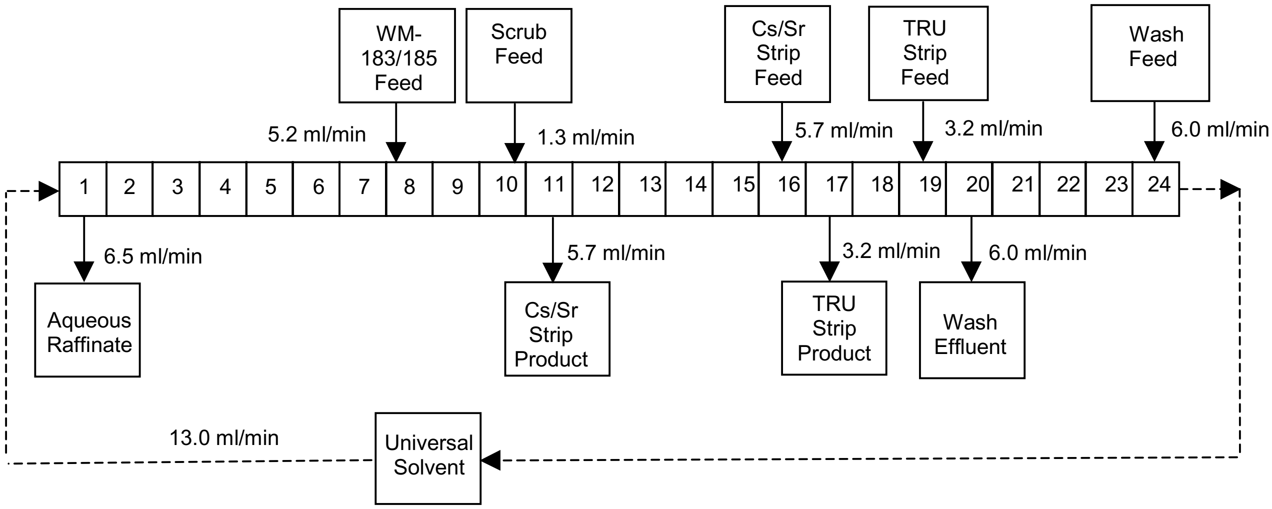

Figure 1. Flowsheet for demonstration of the universal solvent extraction process with WM-183/185 tank waste.

24, aqueous solution flows were established. One molar HNO_3 was used in place of the WM-183/185 feed for the startup. Approximately the first 50 mL of solvent to exit the contactors was collected separately in case it picked up contaminants from previous flowsheet testing. Thirty and sixty minutes after the start of the aqueous flows, samples of the raffinate and Cs/Sr strip product streams were taken in order to determine radioactive contamination levels present at the start of the testing. One hour after the start of the aqueous flows, WM-183/185 flow was established. Samples were taken from the raffinate and strip product streams at intervals of 95, 140, and 185 minutes after actual waste solution flow was established (Time = 0). Samples of all effluent streams were taken 230 minutes after the start of actual waste feed. The contactors were then shut down by simultaneously stopping the contactor motors and feed pumps. Each stage remains approximately at steady-state operating conditions with this type of shutdown. This allowed aqueous and organic samples to be taken from each stage and, therefore, distribution coefficients to be determined for any of the 24 stages.

III. RESULTS

A. Contactor Operation

Throughout the run, small quantities of organic were observed in the aqueous samples of the Cs/Sr strip product and actinide strip product streams. It is estimated that the quantity of organic in the samples was 1% to 2% of the total volume. Flooding was not observed in the raffinate or wash product streams. Precipitate formation was not observed in any of the samples taken during operation or in the contactors after shutdown.

B. Compositions at Shutdown

The percentages of total alpha, ^{137}Cs, ^{90}Sr, ^{238}Pu, ^{239}Pu, ^{241}Am, ^{154}Eu, ^{99}Tc, Ba, Zr, Fe, Pb, Hg, Mo, Na, and K in each of the effluent streams and the overall material balance for each component are given in Tables 2 and 3. Distribution coefficients were calculated for ^{137}Cs, ^{90}Sr, ^{241}Am, and ^{154}Eu on various stages. The resulting distribution coefficients are given in Table 4. A discussion of the behavior of each component follows.

1. Cesium. The ^{137}Cs activity was reduced from 6.78E+06 Bq/mL in the feed to 3.56E+04 Bq/mL in the aqueous raffinate immediately prior to shutdown. This corresponds to a removal efficiency of 99.34%. However, evaluation of all of the ^{137}Cs data indicate the aqueous raffinate sample taken immediately prior to shutdown was contaminated. Specifically:

- The aqueous raffinate sample and the stage one aqueous sample taken after shutdown should be approximately the same activity since the contactors were shutdown after taking the raffinate sample and the contactors remain approximately at steady state conditions during shutdown. The stage one ^{137}Cs activity was 2.41E+03 Bq/mL, which is much lower than the raffinate activity.

Table 2. Percentage of radionuclides in the effluent streams for the flowsheet demonstration with WM-183/185 tank waste.

Effluent	^{137}Cs	^{90}Sr	Alpha	^{241}Am	^{238}Pu	^{239}Pu	^{154}Eu	^{99}Tc
Raffinate	0.051%	0.015%	4.81%	44.9%	2.5%	2.5%	62.8%	108.9%
Cs/Sr Strip	85.6%	81.0%	0.51%	0.02%	0.01%	0.01%	---	11.7%
Actinide Strip	0.14%	12.0%	83.4%	40.2%	98.2%	98.6%	38.0%	0.02%
Wash	0.03%	0.003%	0.01%	0.06%	0.001%	0.0008%	0.04%	---
Solvent	0.004%	0.009%	0.95%	4.8%	0.81%	0.56%	6.0%	0.01%
Material Balance	85.8%	93.1%	88.7%	103.6%	100.6%	101.1%	100.8%	120.6%

Table 3. Percentage of metals in the effluent streams for the flowsheet demonstration with WM-183/185 tank waste.

Effluent	Ba	Fe	Hg	K	Mo	Na	Pb	Zr
Raffinate	<12.8%	94.5%	108.0%	37.8%	34.7%	52.5%	<1.5%	2.3%
Cs/Sr Strip	<5.8%	0.17%	0.13%	17.2%	18.7%	1.2%	2.6%	76.7%
Actinide Strip	206%	6.6%	0.02%	0.01%	<0.12%	0.01%	95.9%	30.0%
Wash	<0.48%	0.09%	0.01%	0.004%	<0.02%	0.002%	0.09%	0.03%
Solvent	18.3%	2.4%	0.02%	0.06%	---	0.20%	< 1.9	<0.57%
Material Balance	206% - 225%	101.4%	109.0%	55.0%	53.6%	53.7%	98.6% - 100.1%	109.0%

- The raffinate samples taken throughout the run ranged from 2.76E+03 Bq/mL to 4.38E+03 Bq/mL (the raffinate sample taken 45 minutes prior to the final raffinate sample had an activity of 2.76E+03 Bq/mL).

- The aqueous raffinate was collected in a container during the demonstration. At the end of the test the contents were mixed, sampled, and submitted for analysis in order to obtain an average raffinate activity for the run. The results of the analysis were then corrected for dilution due to startup (65 minutes of operation with non-radioactive feed and 255 minutes of operation with WM-183/185 feed). The average raffinate activity for ^{137}Cs was 2.37E+03 Bq/mL, which is in agreement with the stage one sample and the raffinate samples taken throughout the testing.

- The distribution coefficients obtained in the extraction section ranged from 1.2 to 1.8. These distribution coefficients were used in conjunction with the Generic TRUEX Model (GTM) to model the extraction section of the flowsheet. With the experimental distribution coefficients and an efficiency of 94% the raffinate activity for ^{137}Cs is expected to be 2.40E+03 Bq/mL (stage one aqueous activity). Under these condition the aqueous stage activities are in good agreement with the predicted activities as shown in Figure 2.

Table 4. Distribution coefficients from the flowsheet demonstration with WM-183/185 tank waste.

Stage		D_{Cs-137}	D_{Sr-90} [a]	D_{Eu-154}	D_{Am-241}
Extraction	1	1.5	1.7	3.0	2.7
	2	1.5	0.9	0.56	0.65
	3	1.4	2.1	0.33	0.37
	4	1.3	1.6	0.20	0.22
	5	1.2	1.3	0.15	0.15
	6	1.3	1.8	0.095	0.14
	7	1.4	1.2	0.016	---
	8	1.8	3.0	0.01	---
Scrub	9	0.87	---	---	---
	10	1.5	---	---	---
Cs/Sr Strip	11	0.16	---	---	---
	12	0.17	---	---	---
	13	0.18	---	---	---
	14	0.18	---	---	---
	15	0.17	---	---	---
	16	0.17	---	---	---
Actinide Strip	17	0.16	---	0.008	0.018
	18	0.20	---	0.001	---
	19	0.17	---	0.001	---
Wash	20	0.28	---	157	---
	21	0.46	---	38	---
	22	0.53	---	47	---
	23	0.78	---	45	---
	24	0.85	---	---	---

a. Organic phase activity was calculated based on material balance.

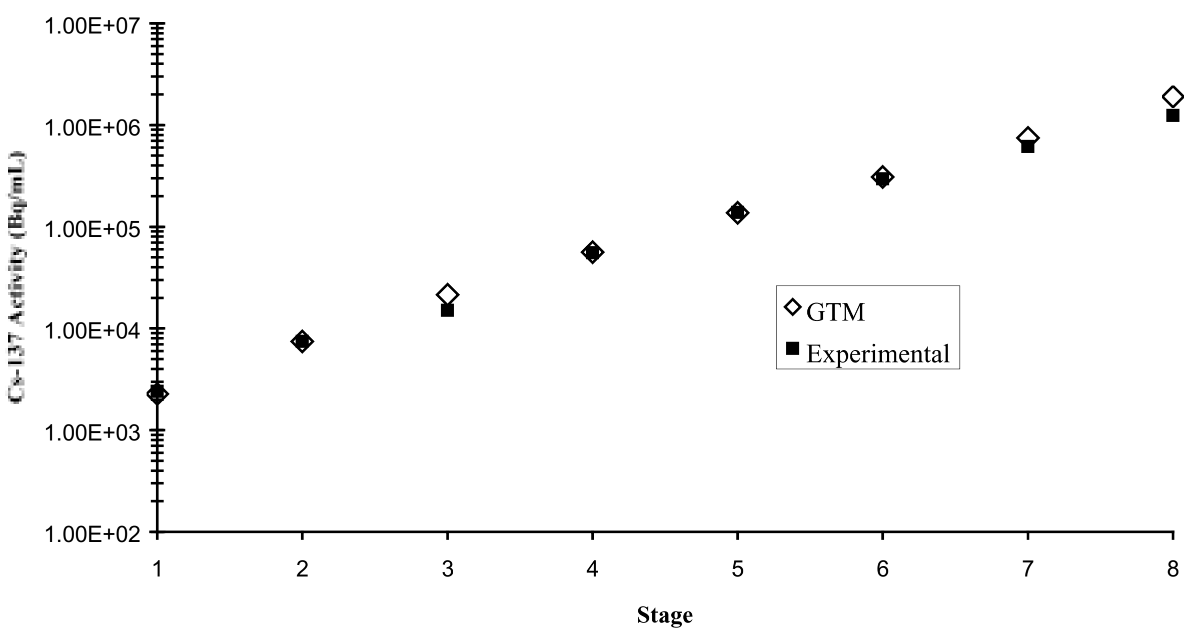

Figure 2. Comparison of experimental data with GTM data at 94% efficiency in the extraction section.

Distribution coefficients for ^{137}Cs ranged from 1.2 to 1.8 in the extraction section and were approximately 0.17 in the Cs/Sr strip section. The ^{137}Cs that extracted was primarily stripped in the Cs/Sr strip section with only 0.17% of the ^{137}Cs stripped in the actinide strip and solvent wash sections. The strip and wash sections removed 99.996% of the extracted ^{137}Cs from the universal solvent.

2. Strontium. The ^{90}Sr activity was reduced from 6.62E+06 Bq/mL in the feed to 800 Bq/mL (0.022 Ci/m^3) in the aqueous raffinate 45 minutes prior to shutdown. This corresponds to a removal efficiency of 99.985% which is sufficient to reduce the ^{90}Sr activity of the WM-183/185 waste below the NRC Class A LLW criteria of 0.04 Ci/m^3. The extracted ^{90}Sr was primarily removed in the Cs/Sr strip section with 12% of the ^{90}Sr removed in the actinide strip section. Distribution coefficients for ^{90}Sr ranged from 0.9 to 3.0 in the extraction section. The strip and wash sections of the flowsheet removed 99.99% of the extracted ^{90}Sr from the universal solvent.

3. Actinides. The total alpha activity was reduced from 2.15E+04 Bq/mL in the feed to 821 Bq/mL (18.1 nCi/g) in the aqueous raffinate 45 minutes prior to shutdown. This corresponds to a removal efficiency of 95.2% which is not sufficient to reduce the actinide activity of the WM-183/185 waste below the NRC Class A non-TRU criteria of 10 nCi/g. Removal efficiencies of 55.1% and 97.5% were obtained for ^{241}Am and ^{238}Pu, respectively. The actinide strip section removed most of the extracted ^{241}Am and ^{238}Pu, with only 0.01% stripped in the Cs/Sr strip section.

Several unexpected results were obtained for the actinide analyses associated with the universal extraction flowsheet demonstration including:

- Much lower than expected overall removal efficiencies were obtained for total alpha (95.2%), ^{241}Am (55.1%), and ^{238}Pu (97.5%).
- The ^{241}Am extraction distribution coefficients were much lower than expected. They ranged from 0.65 to 0.14 on stages 2 through 6, progressively getting lower on each subsequent stage. Based on the results of the batch contact testing with WM-183/185, distribution coefficients of approximately 1.4 were expected. Distribution coefficients for ^{154}Eu were correspondingly low.
- A significant amount of actinide activity was recycled back to the extraction section with the solvent effluent even though the distribution coefficients in the actinide strip section were very low (0.018 for ^{241}Am). The ^{241}Am activity in the organic phase increased from 61 Bq/mL on stage 17 (actinide strip section) to 163 Bq/mL leaving stage 20 (wash section). This resulted in an ^{241}Am buildup in the solvent to 5% of the feed activity, which was recycled back into the extraction section. Similar behavior was observed for ^{154}Eu, with 10.7 Bq/mL on stage 19 (actinide strip section) and 856 Bq/mL on stage 20 (wash section) resulting in 7.4% of the ^{154}Eu feed activity exiting with the washed solvent.
- A buildup of ^{241}Am to 388% of the feed activity and a buildup of ^{154}Eu to 383% of the feed activity occurred in the universal solvent in the extraction section.

These results strongly indicate that flooding of aqueous solution in the organic phase occurred in the actinide strip section. Carryover of aqueous, actinide rich strip solution into the wash section resulted from this flooding. Once in the wash section, the actinides were extracted from the wash solution into the universal solvent ($D_{Eu\text{-}154}$ = 157). The actinides then remained in the universal solvent and were recycled back to the extraction section. Slight flooding was observed in the actinide strip product stream throughout the testing. Samples of the solvent exiting the actinide strip section could not be taken during operation so flooding could not be physically observed. Computer modeling using the GTM and the experimental distribution coefficients obtained for ^{154}Eu was performed. Results indicate that 12% carryover of the actinide strip solution into the wash section via the solvent would result in the observed increase of ^{154}Eu activity from the actinide strip section to the wash section. These results are consistent with the observation of the long disengagement time (>3 minutes) and cloudy organic phase in the actinide strip section of the batch contact testing. A similar recycle of ^{137}Cs and ^{90}Sr to the extraction section was not observed due to the low distribution coefficients in the wash section ($D_{Cs\text{-}137}$ = 0.28 to 0.85). With these low distribution coefficients, any ^{137}Cs and ^{90}Sr carried over to the wash section would remain in the aqueous phase and exit with the wash effluent stream.

Recycle of 5% of the WM-183/185 actinide activity back to the extraction section would have reduced the overall removal efficiency for the actinides to below the levels expected, but not to the extent observed. Furthermore, flooding in the actinide strip section does not account for the low distribution coefficients and the buildup of the actinides in the extraction section. Loading of the Ph_2Bu_2CMPO in the universal solvent with radionuclides and/or metals does account for these results. Distribution coefficients for ^{241}Am and ^{154}Eu were high on stage 1 of the extraction section (3.0 and 2.7, respectively). Therefore, the Ph_2Bu_2CMPO was not loaded as it exited the wash section and was recycled to the extraction section. The distribution coefficients on each subsequent stage decreased significantly, indicating the Ph_2Bu_2CMPO was becoming loaded with extracted species. With the actinide distribution coefficients high on stage one and lower on each subsequent stage, a buildup of the actinides will result in the universal solvent. A buildup of ^{241}Am to 388% of the feed activity and a buildup of ^{154}Eu to 383% of the feed activity was observed in this test.

The Ph_2Bu_2CMPO will extract actinides, Zr, Fe, and Mo. Results indicate that 97.7% of the Zr, 6.9% of the Fe, and 19% of the Mo were extracted. Extraction of these metals results in consumption of 100% of the Ph_2Bu_2CMPO based on the conservative assumption that two moles of Ph_2Bu_2CMPO are consumed per mole of Zr, Fe, Mo, and actinides.

The end result of loading of the universal solvent with the actinides and metals, and a buildup of actinides in the extraction section is an increase in raffinate activity throughout the test. The activity of the actinides in the aqueous raffinate stream steadily increased from 394 Bq/mL to 1,110 Bq/mL as the test proceeded. A total alpha activity of 394 Bq/mL (8.7 nCi/g) in the raffinate near the start of the testing (65 minutes after the start of WM-183/185 feed) suggests that acceptable actinide removal can be accomplished if loading of the Ph_2Bu_2CMPO can be prevented. Also, alleviating the flooding in the actinide strip section would improve the actinide removal efficiency.

4. Technetium. Only 11.7% of the ^{99}Tc was extracted from the WM-183/185 waste by the universal solvent. The extracted ^{99}Tc was effectively stripped in the Cs/Sr strip section. It is important to note that the ^{99}Tc activities in INTEC tank wastes are anticipated to be below NRC Class A LLW requirements (0.3 Ci/m^3). Technetium removal is of concern due to its mobility, as TcO_4^-, in the environment. It would therefore be advantageous to be able to fractionate ^{99}Tc from the wastes.

5. Zirconium, barium, lead, iron, mercury, molybdenum, sodium, and potassium. The effluent streams were analyzed for Zr, Ba, Pb, Fe, Hg, Mo, Na, and K. Of these components, Zr, Ba, and Pb were nearly completely extracted. The Cs/ Sr strip section removed 76.7% of the Zr from the solvent, with the remainder removed in the actinide strip section. Less than 5.8% of the Ba was removed from the solvent in the Cs/Sr strip section. The majority of the extracted Ba was effectively removed from the solvent in the actinide strip section. Only 2.6% of the Pb was removed from the solvent in the Cs/Sr strip section, with the remainder being removed in the actinide strip section.

Significant amounts of Fe, Mo, and K were also extracted. Approximately 19% of the Mo, 7% of the Fe, and 17% of the K were extracted. The extracted Fe was removed from the solvent in the actinide strip section. The extracted K and Mo were removed in the Cs/Sr strip section. Very little of the Hg (0.2%) and Na (1.2%) were extracted by the universal solvent.

IV. CONCLUSIONS

The universal solvent extraction process, developed as a joint effort between the INEEL and the Khlopin Radium Institute, is a viable process for the separation of Cs, Sr, and the actinides from INTEC SBW. Overall removal efficiencies of 99.95%, 99.985%, and 95.2% were obtained for ^{137}Cs, ^{90}Sr, and total alpha, respectively, with the flowsheet demonstration using WM-183/185 waste. This is sufficient to reduce the activities of ^{137}Cs and ^{90}Sr to below NRC Class A LLW limits. The total alpha removal efficiency was not sufficient to reduce the activity of the WM-183/185 tank waste to below NRC Class A non-TRU requirements. The lower than expected removal efficiency for the actinides is due to loading of the Ph_2Bu_2CMPO in the universal solvent with actinides and metals (Zr, Fe, and Mo). Also, the carryover of aqueous solution (flooding) with the solvent exiting the actinide strip section and entering the wash section resulted in the recycle of the actinides back to the extraction section. This recycle of the actinides contributed to the low removal efficiency.

The effluent streams from the flowsheet demonstration with WM-183/185 tank waste were also analyzed for ^{99}Tc, Zr, Ba, Pb, Fe, Hg, Mo, Na, and K. Of these components, 12% of the ^{99}Tc, 97.7% of the Zr, >87% of the Ba, >98.5% of the Pb, 6.9% of the Fe, 0.1% of the Hg, 19% of the Mo, and 17% of the K were extracted.

Based on the analytical results for ^{137}Cs and computer modeling with the Generic TRUEX Model, the centrifugal contactors were operating at a stage efficiency of approximately 94% in the extraction section.

Slight flooding was observed in the Cs/Sr strip product and actinide strip product streams (1-2%). Also, analytical results indicate the solvent leaving the actinide strip section contained approximately 12% of the strip solution. Precipitate formation was not observed during the flowsheet demonstration or in the contactor stages after shutdown.

ACKNOWLEDGMENTS

This work was funded by the Office of Science and Technology (Efficient Separations & Processing Crosscutting Program), United States Department of Energy, and was supported by the DOE/MINATOM Joint Coordinating Committee for Environmental Restoration and Waste Management.

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