DEVELOPMENT AND DEMONSTRATION OF THE UNIVERSAL SOLVENT EXTRACTION PROCESS FOR THE SIMULTANEOUS SEPARATION OF CESIUM, STRONTIUM, AND THE ACTINIDES FROM ACTUAL HIGHLY RADIOACTIVE WASTE

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ABSTRACT

The Universal Solvent Extraction (UNEX) process is being developed 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 UNEX process is being developed as a collaborative effort between the INEEL and the Khlopin Radium Institute in St. Petersburg, Russia. The UNEX solvent consists of chlorinated cobalt dicarbollide for the extraction of Cs, polyethylene glycol for the extraction of Sr, and a carbamoylmethyl phosphine oxide derivative for the extraction of the actinides. 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. The flowsheet consisted of ten stages of extraction, two stages of scrub, six stages of strip, and six stages of solvent wash. With this testing, removal efficiencies of 99.4%, 99.995%, and 99.96% were obtained for ^{137}Cs, ^{90}Sr, and total alpha, respectively. This is sufficient to reduce the activities of ^{137}Cs, ^{90}Sr, and the actinides in a grout waste form to below NRC Class A LLW requirements. Significant amounts of the Ba (99%), Pb (98.6%), Zr (84%), Mo (32%), K (28%), and Fe (8%), were also removed from the feed with the universal solvent extraction flowsheet. Approximately 0.1% of the Na and Al were extracted and exited with the high-activity waste strip product. Operational problems such as flooding or precipitate formation were not observed during testing.

INTRODUCTION

Separation processes are being evaluated for the treatment of 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 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. Nearly 5 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.

SBW is stored in underground stainless steel tanks. A Notice of Noncompliance was filed in 1992 by the State of Idaho Department of Health and Welfare and the Region 10 EPA contending that the tanks did not meet secondary containment requirements set forth in Title 40, Part 265.193 of the Code of Federal Regulations. A recent agreement between the State of Idaho, Department of Energy, and United States Navy, known as the 1995 Settlement Agreement, establishes that the SBW must be out of the tanks by 2012. Separation processes are being evaluated as alternatives to remove the waste from the tanks by 2012, while at the same time, treat the waste for permanent disposal.

The INTEC has historically blended SBW with fuel reprocessing raffinates and solidified it in a fluidized bed calciner operated at 500 ^{o}C. However, all the fuel reprocessing raffinates were depleted by calcination in 1993, which eliminated the option of blending these two wastes. SBW cannot be calcined by itself because the high sodium content causes bed agglomeration in the calcination vessel. SBW can be blended with non-radioactive aluminum nitrate as another option for calcining the waste and removing it from the tanks by 2012. However, this option increases the calcine volume, and the resulting calcine would still require further treatment before final disposal. Additionally, proposed legislation and permitting requirements would require extensive modifications to the calcination facility prior to operation beyond April of 2000. Separations are being developed for the treatment of SBW, as well as an option for treating INTEC high-activity calcined waste. Therefore, calcined waste may eventually be treated by separating the radionuclides from the inert components of the waste.

Several separation technologies have been demonstrated at the INEEL using actual SBW (1). The TRUEX process has been demonstrated to efficiently remove the transuranic (TRU) elements from the waste in a 2-cm centrifugal contactor pilot-plant (2,3). Likewise, strontium removal has been demonstrated in the 2-cm centrifugal contactor pilot-plant using the SREX process (4,5). Cesium removal has been demonstrated in small ion exchange columns (1 - 1.5 cm^3) loaded using either potassium hexacyanoferrate, crystalline silicotitanates, or ammonium molybdophosphate (6). Finally, collaborative testing efforts between Russian scientists from the V. G. Khlopin Radium Institute in St. Petersburg, Russia, have resulted in the successful demonstration of the chlorinated cobalt dicarbollide process with and without polyethylene glycol (PEG) for the removal of cesium and strontium (with PEG) from INTEC SBW (7,8). These collaborative tests have also resulted in the successful demonstration of a phosphine oxide process for removing TRU's from SBW (8).

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 the previous collaborative work with the Russian scientists. The possibility of using a universal solvent containing chlorinated cobalt dicarbollide with 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 Department of Energy Office of Science and Technology 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, a countercurrent flowsheet test using 26 stages of 3.3-cm diameter centrifugal contactors and simulated tank waste was performed in 1997, and a countercurrent flowsheet test using 24 stages of 2.0-cm diameter centrifugal contactors and actual tank waste was performed in 1998, all with very positive results (9-11). The flowsheet demonstration performed with actual waste in 1998 resulted in removal efficiencies of 99.9% and 99.99% for Cs and Sr, respectively. However, the removal efficiency obtained for the actinides was only 96%. This low removal efficiency was attributed to loading of the Ph_2Bu_2CMPO extractant in the universal solvent with metals such as Zr, Fe, and Mo. Based on these results, further testing was performed at the Khlopin Radium Institute and a modified flowsheet was developed to suppress the extraction of metals. This flowsheet was demonstrated using actual INTEC SBW in a centrifugal contactor pilot plant located in a shielded cell facility.

This document reports the results of the Universal Extraction (UNEX) process flowsheet demonstration with SBW. Distribution coefficients and removal efficiencies of the actinides, ^{137}Cs, ^{90}Sr, and some of the non-radioactive elements are reported.

EQUIPMENT DESCRIPTION

The flowsheet demonstration was performed using 2-cm diameter centrifugal contactors installed in an INEEL Remote Analytical Laboratory (RAL) shielded hot cell. The centrifugal contactors, as shown in Figure 1, consist of 24 stages of 2-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 were designed and fabricated by Argonne National Laboratory. The centrifugal contactors were designed specifically for operation of the TRUEX process with INTEC SBW. The contactors were modified at the INEEL for remote installation and operation in the RAL hot cell.

When testing with actual waste solutions, Treatability Study constraints limit the amount of actual waste which can be used to 2.0 Kg's. 2-cm centrifugal contactors are ideal for performing countercurrent flowsheet tests with small volumes of waste solution. These centrifugal contactors have a maximum total throughput of approximately 40 mL/min with the TRUEX solvent and nitric acid solution. A full-scale process for treating the entire SBW inventory in one to two years would require 10-cm to 15-cm centrifugal contactors.

The 2-cm centrifugal contactors do not have provisions for sampling the aqueous and organic solutions exiting individual stages during operation. The aqueous raffinate, strip product, wash effluent, and solvent recycle streams were sampled by routing the solution draining to a receiving vessel into a sample bottle during the actual flowsheet test. Individual stage samples were taken by draining the contactor stages after shutdown.

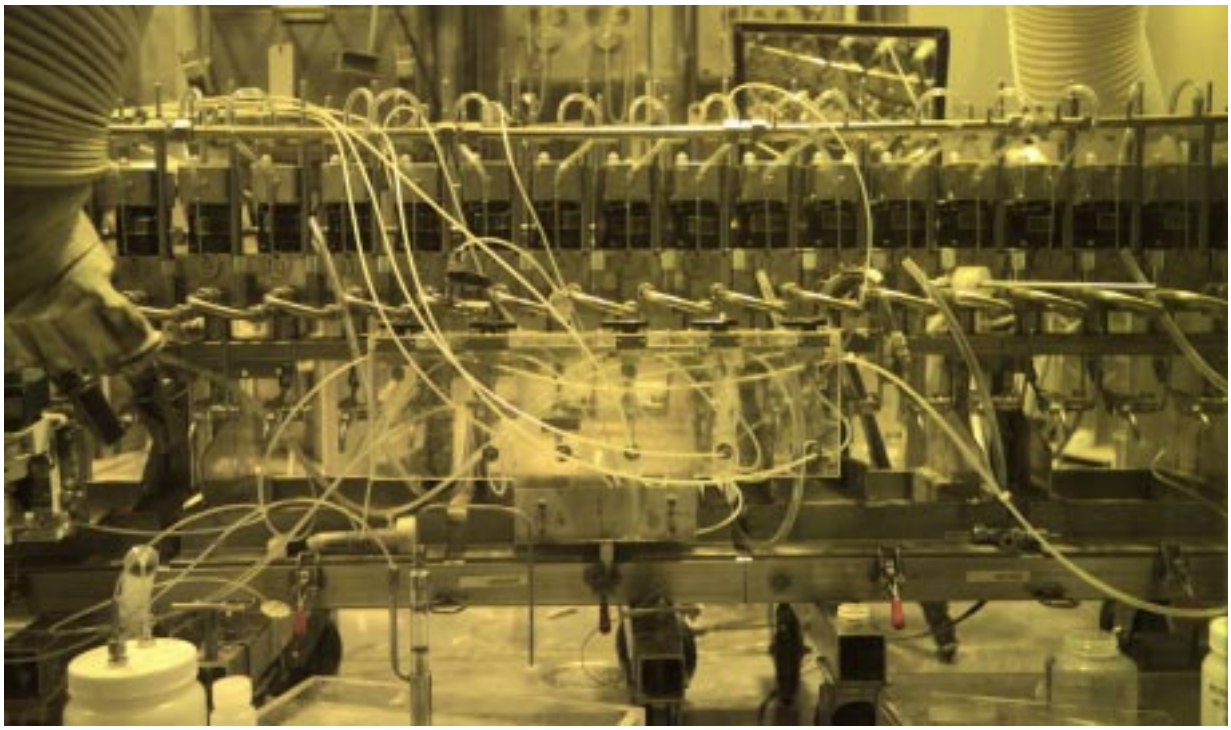

Figure 1. 2-cm Centrifugal Contactors Installed in a Shielded Cell Facility.

METHODOLOGY/EXPERIMENTAL PROCEDURE

UNEX Solvent

The UNEX process solvent used in all testing is a "universal extraction mixture" which was developed to remove all of the radionuclides of concern, both fission product and actinides, in a single, solvent extraction unit operation. The solvent is designed to be the heavy phase, i.e., it has a specific gravity greater than the aqueous solutions. The solvent consists of 0.08 M chlorinated cobalt dicarbollide, 0.5 vol. % polyethylene glycol-400 (PEG-400), and 0.02 M diphenyl-N,N-dibutylcarbamoyl phosphine oxide (Ph_2Bu_2CMPO) in a polar diluent.

The UNEX solvent was recycled continuously during testing. Based on the solvent feed rate of 8 mL/min and 315 minutes of solvent feed, the solvent was recycled a total of 2.5 times. Of the total 315 minutes of solvent feed, 255 minutes was with WM-185 feed. This corresponds to the solvent being recycled 2.0 times while operating with WM-185 feed.

Tank Waste

Approximately 0.9 liters of actual INTEC SBW, obtained from tank WM-185 in 1999 was used as feed solution for the flowsheet testing. Currently, approximately 5.0 million liters of SBW are stored in six tanks. The composition in each tank varies; however, the composition of the solutions in tank WM-185 are representative of the solution in all the tanks. The chemical composition of the average composition of the current SBW inventory is shown in Table I. For the flowsheet demonstration with WM-185 tank waste, 90 mL of 5.2 *M* HF was added to 810 mL of feed. The feed is diluted to reduce the concentration of metals, thus decreasing the solvent

Table I. Adjusted WM-185 Tank Waste And Average SBW Tank Waste Compositions.

Component	Adjusted WM-185	Average SBW	Component	Adjusted WM-185	Average SBW
Acid (*M*)	1.45	1.59	Na (*M*)	1.06	1.9
Al (*M*)	0.51	0.64	NO_3 (*M*)	---	5.07
B (*M*)	---	0.018	Zr (*M*)	0.010	0.002
Ba (*M*)	4.9E-05	6.1E-05	Alpha (nCi/g)	525	369
Ca (*M*)	0.050	0.054	^{241}Am (nCi/g)	50	90
Cr (*M*)	---	0.003	^{134}Cs (Ci/m^3)	---	1.1
F (*M*)	---	0.070	^{137}Cs (Ci/m^3)	88	41
Fe (*M*)	0.021	0.022	^{238}Pu (nCi/g)	435	245
Pb (*M*)	0.0018	0.0012	^{239}Pu (nCi/g)	53	35
Hg (*M*)	0.0037	0.0011	^{99}Tc (Ci/m^3)	0.031	---
Mo (*M*)	0.0002	0.0006	^{90}Sr (Ci/m^3)	75	38
K (*M*)	0.14	0.206	U (g/L)	---	0.12

loading of the Ph_2Bu_2CMPO. Fluoride is added to the feed to complex the Fe and Zr, thus minimizing the extraction of Fe and Zr. The composition of the adjusted WM-185 feed is shown in Table I.

Flowsheet Demonstration With WM-185 Tank Waste

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 the 2-cm diameter centrifugal contactors using actual waste solution. This flowsheet, as shown in Figure 2, consists of ten stages of extraction, two stages of scrub (0.3 *M* citric acid in 0.1 *M* HNO_3), six stages of strip (carbonate solution with a proprietary composition), and six stages of solvent wash (2.0 *M* HNO_3). In the extraction section, the Cs, Sr, and actinides are extracted from the waste feed. In the scrub section, extracted metals such as Zr and Fe are back-extracted from the UNEX solvent. The radionuclides are recovered from the UNEX solvent in the strip section. Finally, the solvent is washed with nitric acid to remove any entrained carbonate solution from the strip section prior to recycle of the solvent to the extraction section.

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 1, aqueous solution flows were established. One molar HNO_3 was used in place of the WM-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. The process then continued to operate without recycle of the

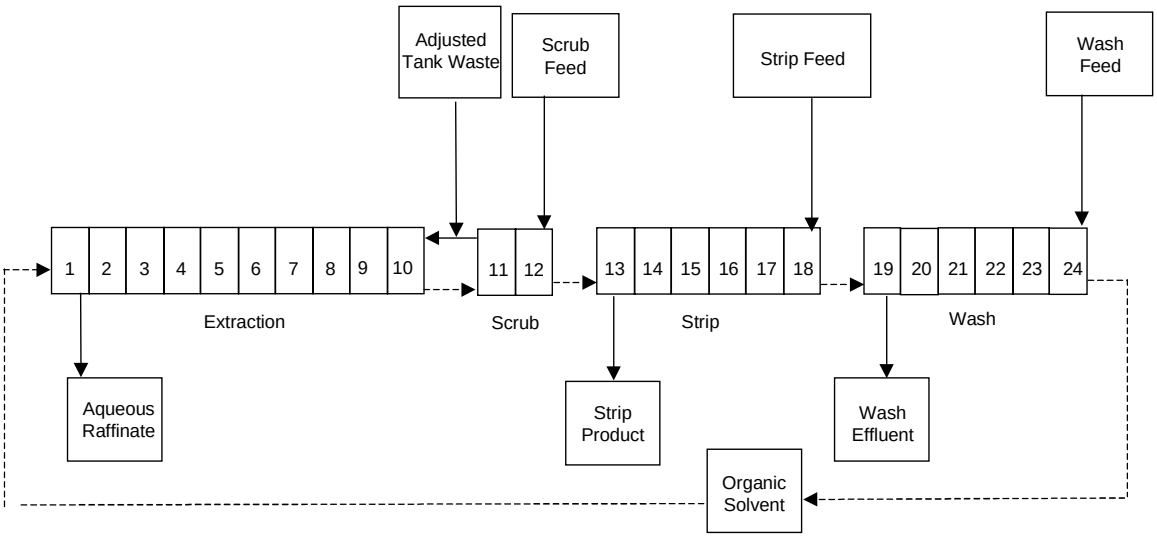

Figure 2. UNEX Process Flowsheet

solvent. Twenty minutes after the start of the aqueous flows, WM-185 flow was established. Samples were taken from the raffinate 60 and 80 minutes after the start of WM-185 feed. At this time recycle of the solvent was initiated and the raffinate, strip product, wash effluent, and solvent effluents were sampled at intervals of 30 minutes. Final samples of all effluent streams were taken 255 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.

RESULTS AND DISCUSSION

The percentages of ^{137}Cs, ^{90}Sr, total alpha, ^{241}Am, ^{238}Pu, ^{239}Pu, ^{154}Eu, ^{99}Tc, Al, Ba, Ca, Ce, Fe, Hg, K, Mo, Na, Nd, Pb, and Zr in each of the effluent streams and the overall material balance for each component are given in Tables II and III. It should be noted that the percentages of components in the solvent are not included in the overall material balance since the solvent was recycled during testing. Distribution coefficients were calculated for total alpha, ^{137}Cs, ^{90}Sr, ^{241}Am, ^{238}Pu, ^{239}Pu, and ^{154}Eu on various stages. A discussion of the behavior of each component follows.

Cesium

The ^{137}Cs activity was reduced from 3.24E+06 dps/mL in the feed to 1.44E+04 dps/mL (0.39 Ci/m^3) in the aqueous raffinate immediately prior to shutdown. This corresponds to a removal efficiency of 99.4%. A grout formulation has been developed for immobilization of the low activity waste from separations processes (12). This formulation consists of concentrating and neutralizing the waste followed by the addition of grout additives. The removal efficiency of 99.4% is sufficient to result in a ^{137}Cs activity in this grout waste form which is below the NRC Class A LLW criteria of 1.0 Ci/m^3. The somewhat low ^{137}Cs removal efficiency was due to low

Table II. Percentages Of Radionuclides In The Effluent Streams For The Flowsheet Demonstration With Tank Waste.

Effluent	^{137}Cs	^{90}Sr	Alpha	^{241}Am	^{238}Pu	^{239}Pu	^{154}Eu	^{99}Tc
Raffinate	0.57%	0.0052%	0.040%	0.0002%	0.006%	0.002%	0.42%	81.2%
Strip	100.6%	108.1%	100.4%	105.6%	96.9%	103.9%	78.6%	<0.14
Wash	0.006%	0.0003%	0.001%	---	---	---	0.015%	---
Solvent	0.005%	0.005%	0.02%	0.2%	0.005%	0.0006%	0.075%	0.013%
Material Balance	101.2%	108.1%	100.4%	105.8%	96.9%	103.9%	79.1%	81.3%

TableIII. Percentages Of Non-Radioactive Metals In The Effluent Streams For The Flowsheet Demonstration With Tank Waste.

Effluent	Al	Ba	Ca	Ce	Fe	Hg
Raffinate	108.2%	<1.05%	97.8%	<11.4%	74.7%	110.9%
Strip	0.14%	105.7%	9.9%	109%	8.3%	<1.2%
Wash	---	---	---	---	---	---
Solvent	0.022%	<3.9%	<0.12	<25.8%	0.44%	0.04%
Material Balance	108.4%	106.8%	107.7%	109% - 120%	83.0%	110.9%
Effluent	K	Mo	Na	Nd	Pb	Zr
Raffinate	74.6%	79.1%	108.0%	<2.4%	1.2%	13.0%
Strip	27.9%	31.7%	0.13%	112%	83.2%	68.6%
Wash	0.008%	---	0.01%	---	---	---
Solvent	0.15%	<15.6%	0.02%	<5.5%	<0.62%	0.09%
Material Balance	102.4%	110.8%	108.1%	112% - 114%	84.4%	81.6%

distribution coefficients which ranged from 0.68 to 1.3 in the extraction section. In the strip section distribution coefficients ranged from 0.11 to 0.20, resulting in 99.99% of the extracted Cs exiting in the strip product.

Strontium

The ^{90}Sr activity was reduced from 2.76E+06 dps/mL in the feed to 111 dps/mL (0.003 Ci/m^3) in the aqueous raffinate immediately prior to shutdown. This corresponds to a removal efficiency of 99.995% which is sufficient to reduce the ^{90}Sr activity in a grout waste form to below the NRC Class A LLW criteria of 0.04 Ci/m^3.

Distribution coefficients for ^{90}Sr ranged from 0.7 to 6.3 in the extraction section with an average value of approximately 3.0. In the strip section distribution coefficients for ^{90}Sr ranged from 2.9E-05 to 0.09. As a result, 99.9999% of the extracted ^{90}Sr exited in the strip product.

Actinides

The total alpha activity was reduced from 2.33E+04 dps/mL in the feed to 7.2 dps/mL (0.16 nCi/g) in the aqueous raffinate immediately prior to shutdown. This corresponds to a removal efficiency of 99.96% which is sufficient to reduce the actinide activity in a grout waste form to below the NRC Class A non-TRU criteria of 10 nCi/g. Removal efficiencies of 99.999% and 99.994% were obtained for ^{241}Am and ^{238}Pu, respectively. In the strip section, 99.97%, 99.996%, and 99.9999% of the total alpha, ^{241}Am, and ^{238}Pu, respectively, were removed from the solvent.

The removal efficiency for total alpha was greatly improved as compared to previous flowsheet testing. It the previous testing, only 95% removal was obtained due to loading of the Ph_2Bu_2CMPO with Zr, Fe, and Mo. The following changes were made to the previous flowsheet, which reduced the loading of Ph_2Bu_2CMPO during this testing.

- The feed solution was diluted, thus decreasing the concentration of Zr, Fe, and Mo.
- An increased amount of fluoride was added to the feed solution to complex the Zr.
- A scrub solution consisting of 0.3 *M* citric acid was used instead of 0.1 *M* HF.

In the previous flowsheet testing it is estimated that nearly all of the Ph_2Bu_2CMPO was consumed from the extraction of Zr, Fe, and Mo. It is estimated that 52% of the Ph_2Bu_2CMPO was consumed with this modified flowsheet based on the assumption of three moles of Ph_2Bu_2CMPO consumed per mole of the extracted metal.

Technetium

Less than 0.14% of the ^{99}Tc was extracted from the WM-185 waste by the universal solvent and exited in the strip product. The overall material balance for ^{99}Tc was 81.3%. The wash effluent was not analyzed for ^{99}Tc; therefore, any ^{99}Tc in the wash effluent would increase the overall material balance. 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.

Aluminum, barium, calcium, cerium, iron, mercury, potassium, molybdenum, sodium, neodymium, lead, and zirconium
The effluent streams were analyzed for Al, Ba, Ca, Ce, Fe, Hg, K, Mo, Na, Nd, Pb, and Zr. Of these components, Ba, Ce, Nd, and Pb were nearly completely extracted. Significant amounts of Zr, Mo, K, and Fe were also extracted (87%, 32%, 28%, and 8%, respectively). The strip product analytical results indicate that 10% of the Ca was extracted and exited in the strip product. However, the raffinate analytical results indicate that 98% of the Ca remained in the raffinate. Aluminum, mercury, and sodium were essentially inextractable (<1% extracted).

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CONCLUSIONS AND RECOMMENDATIONS

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 99.4%, 99.995%, and 99.96% were obtained for ^{137}Cs, ^{90}Sr, and total alpha, respectively, with the flowsheet demonstration using WM-185 waste. This is sufficient to reduce the activities of ^{137}Cs, ^{90}Sr, and the actinides in a grout low-level waste form to below NRC Class A LLW limits. The actinides, ^{137}Cs, and ^{90}Sr were effectively removed from the universal solvent with the six stages of strip in the universal solvent flowsheet demonstration.

The effluent streams from the flowsheet demonstration were also analyzed for ^{99}Tc, Al, Ba, Ca, Ce, Fe, Hg, K, Mo, Na, Nd, Pb, and Zr. Ba, Ce, Nd, and Pb were nearly completely extracted. Significant amounts of Zr, Mo, K, Ca, and Fe were also extracted (87%, 32%, 28%, 10%, and 8%, respectively). Technetium, aluminum, mercury and sodium were essentially inextractable (<1% extracted).

Recommendations

Further development and testing of the universal solvent extraction process is recommended in order to optimize the flowsheet. Adjustments to the flowsheet should be made to minimize the flowrates of process solutions. Specifically, reduction of the O/A ratio in the extraction section, reduction of the strip flowrate and/or concentration, and minimization of the wash effluent flow via recycle should be investigated. Changes in these areas would decrease the overall amounts of high and low-activity liquid wastes generated by the process and potentially reduce the quantity of high- and low-activity waste forms generated. Additionally, applicability of the UNEX process to other waste streams, such as dissolved INTEC calcine or wastes at other DOE sites, should be evaluated.

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