Idaho National Engineering and Environmental Laboratory

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Integrated AMP-PAN, TRUEX, and SREX Flowsheet Test to Remove Cesium, Surrogate Actinide Elements, and Strontium from INEEL Tank Waste Using Sorbent Columns and Centrifugal Contactors

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ABSTRACT

Three unit operations for the removal of selected fission products, actinides, and RCRA metals (mercury and lead) have been successfully integrated and tested for extended run times with simulated INEEL acidic tank waste. The unit operations were ion exchange for Cs removal, followed by TRUEX solvent extraction for Eu (actinide surrogate), Hg, and Re (Tc surrogate) removal, and subsequent SREX solvent extraction for Sr and Pb removal.

Approximately 45 L of simulated INTEC tank waste was first processed through three ion exchange columns in series for selective Cs removal. The columns were packed with a composite ammonium molybdophosphate-polyacrylonitrile (AMP-PAN) sorbent. The ion exchange system was operated continuously for ~34 hours at 22 bed volumes per hour in the first two columns. The first two columns were each sized at a bed volume of 60 cm^3 and were operated to 100% breakthrough. The experimental breakthrough data were in excellent agreement with modeling predictions based on data obtained with much smaller (1.5 cm^3) columns. The third column (220 cm^3) was used for polishing and Cs removal after breakthrough of the up-stream columns. The Cs removal was >99.83% in the ion exchange system without interference from other species.

Most of the effluent from the ion exchange (IX) system was immediately processed through a TRUEX solvent extraction flowsheet to remove europium (americium surrogate), mercury and rhenium (technetium surrogate) from the simulated waste. The TRUEX flowsheet test was performed utilizing 23 stages of 3.3 cm centrifugal contactors. The TRUEX test was operated a total of 71.3 hours and processed ~41 L of the IX effluent using 1.5 L of TRUEX solvent with constant solvent recycle. The TRUEX solvent was recycled through the flowsheet an estimated 17 times. Greater than 99.999% of the Eu, 96.3% of the Hg, and 56% of the Re were extracted from the simulated feed and recovered in the strip and wash streams. Minor operational problems were encountered in the solvent wash section, where Hg precipitated as HgO; the problem did not require shutdown of the system. Flooding was never observed during the experiment. Over the course of the test, there was no detectable build-up of any components in the TRUEX solvent.

The raffinate from the TRUEX test was stored and subsequently processed several weeks later through a SREX solvent extraction flowsheet to remove strontium, lead, and Re (Tc surrogate) from the simulated waste. The SREX flowsheet test was performed using the same centrifugal contactors used in the TRUEX test after reconfiguration and the addition of 3 stages. Approximately 51 L of TRUEX raffinate was processed through the system during 77.9 hours of continuous operation with 1.5 L of SREX solvent and continuous solvent recycle. The SREX solvent was recycled through the system an estimated 45 times without measurable build-up of any components in the solvent. Approximately 99.9% of the Sr, >99.89% of the Pb, and >96.4% of the Re were extracted from the aqueous feed to the SREX flowsheet and recovered in the strip and wash sections. Operational problems such as flooding and precipitation were not encountered during the SREX test.

Approximately 41 L of simulated tank waste (based on the volume processed through the TRUEX flowsheet) was processed through the integrated flowsheet and resulted in 175 L of liquid high activity waste (HAW) and 219.6 L of liquid low activity waste (LAW). The HAW fraction would be evaporated, dried and subsequently vitrified for final disposal. Based on current baseline assumptions, including a maximum phosphate loading of 2.5 wt. % in the HAW glass, the flowsheet tested would result in the production 0.195 kg of glass per L of tank waste processed. The LAW fraction would be solidified (via evaporation and denitration) and subsequently grouted. The current baseline assumptions for grouting the LAW stream indicate 0.37 kg of grout would be produced per L of tank waste treated. Under these assumptions, treating the current inventory of ~5E+6 L (5,000 m^3) of tank waste would result in 375 m^3 of HAW glass and 1,135 m^3 of LAW Class A performance grout. The HAW glass volume could be significantly decreased by suitable TRUEX flowsheet modifications.

ACKNOWLEDGEMENTS

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INTRODUCTION

The Idaho Nuclear Technology & Environmental Center (INTEC), formerly known as the Idaho Chemical Processing Plant (ICPP), previously reprocessed spent nuclear fuel to recover enriched uranium. The radioactive raffinate stream from reprocessing was stored and subsequently solidified at 500°C in a fluidized bed calciner. Calcination converted the liquid wastes into a granular, free flowing solid (calcine). INTEC currently has approximately 4,200 cubic meters of radioactive calcine stored onsite in above ground stainless steel bins enclosed in a concrete vault.

A secondary acidic aqueous waste was generated during equipment decontamination between processing campaigns and from solvent cleanup activities. Currently, over 5 million liters of tank waste are stored in underground, concrete enclosed stainless steel tanks at INTEC. This liquid waste is not directly amenable to calcination due to its high sodium content, which causes agglomeration of the fluidized bed. Historically, the high sodium secondary wastes were blended with reprocessing raffinates; however, blending is no longer possible since all raffinate solutions have been calcined. The current inventory of high sodium tank wastes can be calcined if blended with non-radioactive aluminum nitrate; however, this reduces calciner throughput and results in increased calcine volume. The reduced throughput may prevent meeting the year 2012 deadline for emptying the tank farm, as agreed upon by DOE and the State of Idaho EPA.

Separation processes are being evaluated as alternatives to calcination for treating the remaining tank waste, and ultimately, for treating the (dissolved) calcine for final disposition. The large volume of calcine drives the economics of the separation processes, since the tank wastes are a small fraction of the total waste volume stored at INTEC. The goal of the separations processes is to remove fission products and actinides from the liquid wastes so the bulk of the treated waste meets NRC Class A low level waste (LLW) requirements as defined by 10 CFR 61.55. It is anticipated that the combined LLW raffinate streams resulting from the separation unit operations would be grouted for near surface disposal. The remaining high activity fractions containing transuranic (TRU) and fission products would be vitrified and disposed as high activity waste (HAW) glass. The economic benefit of utilizing separation processes is a significant reduction in the volume of HAW glass, which has very substantial disposal costs.

One viable treatment alternative for the remaining inventory of INTEC tank waste is known as the “full separation” option. This option consists of three integrated separation unit operations intended to collectively remove the major radionuclides and, as an unintentional benefit of process chemistry, certain RCRA metals are also removed from INTEC waste solutions:

(1) Ion exchange (IX) using an ammonium molybdophosphate (AMP)-polyacrylonitrile (PAN) composite sorbent (referred to as AMP-PAN) to remove cesium.

(2) The Transuranic Extraction (TRUEX) solvent extraction process to remove the actinides, mercury, and technetium.
(3) The Strontium Extraction (SREX) solvent extraction process to remove Sr, Pb, and Tc.

AMP is a well-known inorganic cesium precipitant in acidic media, but due to small particle size (resulting in poor hydrodynamic properties of column operation), is not alone suitable for use in columns as an ion exchange media. Recently, Czech scientists have developed an engineered form of AMP by forming a composite of AMP and PAN [1,2]. Numerous experiments at the Czech Technical University and Idaho National Engineering and Environmental Laboratory (INEEL) using simulated and actual tank waste and solutions of dissolved calcine have demonstrated AMP-PAN has a high capacity and selectivity for cesium from these acidic waste solutions [3,4,5]. The high selectivity of AMP-PAN for Cs, relative to bulk components in the tank waste, makes AMP-PAN an excellent choice for cesium removal. Elution of cesium and regeneration/reuse of the sorbent is possible, but this requires the use of concentrated ammonium salts (such as NH_4Cl or NH_4NO_3) which have undesirable material handling or safety characteristics. AMP is readily dissolved from the PAN support with caustic solutions (e.g. 6 to 10 bed volumes of 1M NaOH) [4,6], which may provide a convenient means to separate the organic substrate (PAN) from the Cs laden inorganic (AMP) for subsequent processing options.

The Transuranic Extraction (TRUEX) process, developed by Horwitz, Schulz, and many others, is the most mature actinide separation technology for INTEC acidic tank waste [7]. The active extractant used in the TRUEX process solvent is octyl(phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide (CMPO). Tributylphosphate (TBP) is added to the solvent as a phase modifier to prevent third phase formation. Isopar $L^{®}$, a paraffinic hydrocarbon, is used as the diluent. The TRUEX process has demonstrated effective removal of actinides from actual tank waste using centrifugal contactors to well below the 10 nCi/g NRC Class A LLW TRU requirements [7,8]. Previous testing also indicates the TRUEX solvent effectively removes mercury from tank waste and the sodium carbonate wash cycle back extracts the mercury, which is added back to the LLW fraction [9,10].

The Strontium Extraction (SREX) process, developed by Horwitz *et. al.* [7], has been shown to remove strontium, lead, and technetium from INTEC tank waste [13]. The active extractant used in the SREX solvent is 4',4'(5')-di-(tert-butyldicyclohexo)-18-crown-6 (DtBuCH18C6). TBP is added as a phase modifier and the diluent is Isopar $L^{®}$, as used in the TRUEX solvent. SREX flowsheet performance has been previously developed and demonstrated at INTEC using both actual and simulated tank wastes and dissolved calcine solutions [8,11,12,13].

It is anticipated that the high activity waste (HAW) resulting from the full separation process would be vitrified and disposed of as a glass waste form in a deep geological repository. Based on the current assumptions, the HAW fraction will be evaporated, after which glass frit would be added to form slurry feed to a melter. Based on the available data, waste loading estimates for the HAW fraction for the full separations flowsheet tested here will be limited by a maximum of 2.5 wt. % phosphate (as P_2O_5) composition in the glass [14]. The phosphate in the HAW fraction predominately came from the use of HEDPA in the actinide strip solution from the TRUEX process. Alternative strip solutions or strip compositions could be used to avoid or minimize this problem. As a point for comparison, direct vitrification of the tank waste is expected to result in 0.32 kg of glass per liter of tank waste processed [14]. Separation options must significantly reduce the quantity of HAW glass generated if these options are to be economically feasible.

Grouting of the LLW fraction is the anticipated and preferred method for disposal. The current preferred option would be near surface disposal in an approved landfill, which would likely require a “performance grout” (a grout that meets not-yet-promulgated waste acceptance criteria, but likely includes performance measures of strength and leaching characteristics for RCRA constituents and radionuclides). This disposal option would likely require a grout meeting NRC Class A LLW criteria (as a minimum) for the radionuclides. An alternative disposition option for low activity waste (LAW) grout would be at the Waste Isolation Pilot Plant (WIPP). WIPP was designed for disposition of contact-handled TRU waste and likely would not require a “performance grout” (e.g., a solid that meets the no-free liquid and other requirements of the WIPP Waste Acceptance Criteria). Since the full separation option would includes removal of the TRU elements, it is unlikely a final LAW grout could be sent to WIPP, and therefore a “performance grout” and NRC Class A limits are likely required. Primarily due to the above considerations, development efforts at the INEEL have focussed on a number of potential grout formulations, which depend largely on the disposition route of the final product [15]. The current baseline for the full separation option would include concentration and denitration of the LAW fraction to convert the liquid stream to solids comprised largely of metal oxides. The grout ingredients (Portland cement, slag, fly ash and water) would be mixed with the LAW solids, poured into drums, and allowed to cure [15]. This formulation allows for 30 wt % waste loading in the grout, which has a cured density of 1,826 kg/m^3 and a 28 day compressive strength of 1,600 psi [15]. Using this formulation, 0.21 L of grout is produced per liter of LAW [15]. Initial material balance calculations indicate there are approximately 5 L of LAW produced per liter of SBW treated in full separations. Therefore, the full separations flowsheet developed for the treatment of INEEL tank waste must reduce the activity in the LAW raffinate to approximately 110% of the NRC Class A LLW limits in order for the resulting grout to meet these limits. The NRC Class A limit, estimated limit in the LAW stream (to achieve a Class A grout), typical tank waste concentration, and estimated removal efficiencies are indicated in Table 1 for tank waste.

This report provides the results of integrated flowsheet testing of the full separation option with simulated tank waste solution using AMP-PAN ion exchange columns and centrifugal contactors for the TRUEX and SREX operations. The pilot plant equipment, including the columns and centrifugal contactors, was assembled at INTEC in building CPP-1634, which is a non-radioactive area. A simulated tank waste solution (~45 L) was prepared to emulate the average chemical composition of actual INTEC tank waste. Non-radioactive europium was spiked into the simulant as a surrogate for americium to gauge the performance of the TRUEX solvent with respect to actinide behavior. Stable cesium and strontium were spiked into the simulant (at greater than 10 times actual tank concentrations to allow analytical detection) to establish removal data for AMP/PAN and SREX flowsheet testing, respectively. Additionally, rhenium was added to the simulated waste (as perrhenate) as a surrogate for technetium.

Table 1. Characteristics of the LAW fraction.

Component	NRC Class A Limit	LAW Limit [a]	Typical Conc. In Tank Waste	Required Removal Efficiency
Cs-137	1.0 Ci/m^3	1.1 Ci/m^3	40 Ci/m^3	97.3%
Sr-90	0.04 Ci/m^3	0.044 Ci/m^3	40 Ci/m^3	99.9%
TRU	10 nCi/g	11 nCi/g	1200 nCi/g	99.1%

[a] Activity in the LAW stream necessary to insure the final grout meets NRC Class A limits.

PURPOSE AND SCOPE

The purpose of this work was to evaluate the performance of the integrated flowsheet with simulated tank waste to support the full separation option for the upcoming High Level Waste Environmental Impact Statement (EIS). The scope of this work included the use of ion exchange (IX) columns in series containing AMP-PAN sorbent for cesium removal. The effluent from the IX process was used as feed in an extended (~80 hour) TRUEX flowsheet test in centrifugal contactors to evaluate Eu (as an Am surrogate) and Hg removal. Finally, the raffinate collected from the TRUEX flowsheet operation was used to feed an extended (~80 hour) SREX flowsheet test in centrifugal contactors to evaluate the efficiency of Sr and Pb removal. The scope of this experimental program included:

1) Integrating the three unit operations (IX-TRUEX-SREX) performed in series to evaluate and/or confirm the behavior of targeted species and numerous waste matrix components in the integrated flowsheet.

2) Increasing the scale of the IX columns over those used in earlier tests to evaluate the associated effect on cesium removal and obtain complete breakthrough curves for two of the AMP-PAN columns. Effluent Cs concentrations was to be maintained at low values using a third, polishing column in series with the first two columns.

3) Extending the run times used in the solvent extraction flowsheets to identify potential problems with recycle of the TRUEX and SREX organic solvents occurring from solvent degradation or long term build-up of materials in the organic phases.

4) Evaluating the feasibility and testing optimal conditions under which the TRUEX flowsheet could be used for mercury removal from the simulated tank waste and stripping of the extracted Hg in the carbonate wash stream.

5) Evaluating of the effect of any residual Hg in the TRUEX process raffinate on the SREX process.

6) Evaluating the feasibility and conditions for lead removal and stripping in the SREX flowsheet.

7) Obtaining experimental data to establish concentrations and volumes of the composite HAW and LAW streams resulting from combination of individual product streams from the three unit operations.

EQUIPMENT DESCRIPTION

AMP-PAN IX Columns

Three borosilicate glass columns and accessories were obtained from the ACE Glass Company for AMP-PAN testing. All columns were 2.54 cm inside diameter. The first two columns were filled with AMP-PAN to a bed height of 11.8 cm (60 cm^3 bed volume) and the third column was filled to a bed height of 43.4 cm (220 cm^3 bed volume). The columns were sized so that the first two columns would reach complete cesium breakthrough and the third column would function as a polishing column to remove the cesium following breakthrough of the second column. Sintered glass frits (2.54 cm od) were inserted in the top and bottom of the columns to prevent AMP-PAN from escaping through or plugging the column outlet during testing**.** Hollow Teflon plugs were screwed into the threaded ends of the columns to support the glass frit and provide the means to attach necessary valves and tubing to the glass columns. Three-way valves were attached to the bottom of the columns to allow for sample collection during operation. Pressure gauges were placed on inlet and outlet lines to monitor differential pressure of the columns.

The feed was pumped from the feed vessel to the columns using a valveless metering pump. Tygon® tubing was used to route the solution from the feed vessel through the pump, then columns, and finally to the collection vessel. Tygon® tubing was attached to equipment using appropriate swagelok fittings. Solution was fed to the columns in series, beginning with the two 60 cm^3 columns and then the 220 cm^3 column. The test setup is pictured during operation in Figure 1.

TRUEX/SREX Solvent Extraction

Flowsheet testing was performed using centrifugal contactors installed in building CPP-1634. The contactor setup consists of twenty-six available stages, reagent feed and receiving vessels, and feed pumps with associated controllers. The 3.3 cm centrifugal contactor mockup is pictured in Figure 2.

The 3.3 cm contactors were designed and fabricated in Moscow, Russia by the Research and Development Institute of Construction Technology (NIKIMT). Table 2 lists the operating specifications of the contactors. A total of 26 contactors were obtained from NIKIMT for testing purposes. Each stage can be operated independently allowing numerous combinations for changing flowsheet configurations.

Solutions are fed to the contactors using valveless metering pumps with controllers. Surge suppression tubes, 4 inches long and 1 inch in diameter, were placed on the pump outlets to dampen solution flow fluctuations. Flow rates were adjusted by controlling pump speed using a ten-turn potentiometer or by manual adjustment of the piston stroke length and/or a combination of the two. Once solutions enter the contactors, flow through the equipment is by gravity, i.e., the solutions in the contactors are not under pressure. The product solutions from the contactors drain by gravity to the product vessels.

The 3.3 cm centrifugal contactors do not have provisions for sampling aqueous or organic streams exiting from individual stages. Aqueous raffinate, aqueous strip, and solvent recycle streams were sampled by periodically routing the solution draining to the appropriate receiving vessel into a sample bottle.

Figure 1. Ion exchange equipment used for AMP-PAN testing in the integrated flowsheet.

Table 2. Operating specifications for the 3.3 cm contactors.

Size	3.3-cm rotor diameter
Mixing Chamber Volume	22 mL
Separating Chamber Volume	32 mL
Overall Dimensions:	
Length	105 mm (4.13 in)
Width	132 mm (5.2 in)
Height	286 mm (11.26 in)
Volumetric Capacity	25 L/hr (for the system TBP-kerosene/2 M HNO_3)
Mass	5 kg per stage (includes electric motor)
Motor	160-180 volt, 50-60 Hz, 0.04 kW (Russian Design)
RPM	2700 rpm (slightly adjustable)
Material of construction	12X18H10T stainless steel (Russian designation)
Inlet and outlet ports	3/8 in. o.d. tubing
Configuration	Single stage units, which can be configured as desired. Stages connected using U-tubes.

Figure 2. Centrifugal contactors used for the TRUEX and SREX solvent extraction flowsheets.

METHODOLOGY/EXPERIMENTAL PROCEDURE

Simulated Tank Waste Composition

A large volume of tank waste was necessary to accommodate the extended run times in the solvent extraction unit operations. For this reason simulated waste was used in lieu of actual tank waste (there are volume restrictions on the use of actual waste related to RCRA treatability studies). Approximately 50 liters of simulated tank waste was prepared from stock chemical reagents. The simulated waste was prepared to represent the average composition of actual tank waste currently stored at INTEC. Compositions of both the average actual, and simulated waste solutions are presented in Table 3. Non-radioactive europium was added to the makeup as a surrogate for americium to evaluate the performance of the TRUEX process with respect to actinide behavior. Stable cesium and strontium were also added to increase their respective concentrations in the feed providing better analytical results to more accurately calculate removal data for IX and SREX unit operations.

Past experience indicates that precipitate formation, typically $NaNO_3$, occurs in freshly prepared tank waste simulant. The precipitate is filtered from the simulant after preparation and prior to experimental testing. Once the solution is filtered, it is typically stable for several months of storage at room temperature in the laboratory. Precipitate formation from the simulated feed has been observed to increase as temperature decreases. The feed used in these tests was prepared, allowed to settle overnight, and partially filtered the next day through a 5 μm filter. The solution was allowed to stand overnight and the entire volume was re-filtered through a new 25 μm filter the following day.

Table 3. Composition of actual and simulated INTEC tank waste.

	Average Tank Composition	Composition of Simulated Waste			Average Tank Composition	Composition of Simulated Waste	
Element	(*M*)	(*M*)	(mg/L)	Element	(*M*)	(*M*)	(mg/L)
Acid	1.97	1.55	---	NO_3	5.92	---	N/A [a]
Al	0.63	0.54	1.41E+04	Ni	0.002	0.002	107.6
B	0.016	0.018	194.7	Pb	0.002	0.001	191.7
Ca	0.046	0.045	1797	PO_4	0.016	N/A	N/A
Cl	0.034	0.041	1450	Sr	1.35E-05	0.001	86.85
Cs	2.89E-05	0.001	130	SO_4	0.051	N/A	N/A
Cr	0.004	0.005	247.3	Zr	0.006	0.0014	130.7
F	0.067	0.259	4930	Eu	---	0.0070	1067
Fe	0.022	0.023	1282	Re	---	0.0036	667.4
Hg	0.0016	0.0020	395	Ba	5.65E-05	0.00011	15.00
K	0.19	0.118	4600	Ag	2.25E-05	4.12E-05	4.446
Mn	0.013	0.008	426.2	Se	2.56E-05	2.87E-05	2.266
Mo	6.55E-04	0.0012	119.5	Ru	3.01E-05	1.94E-05	1.966
Na	1.82	1.24	2.84E+04	Rh	---	<1.00E-06	<0.10
Cl/Hg (molar)	21.3	20.8	---	Pd	4.75E-06	<6.31E-07	<0.07

[a] N/A = not analyzed.

Flowsheet Configuration

Based on the results of previous tests with tank wastes, a flowsheet was proposed for integrated testing of the three operations in series as indicated in Figure 3. The AMP-PAN IX system was placed first in the series since early cesium removal (primary γ-emitter) could result in a potential reduction of shielding requirements for the downstream operations. The AMP-PAN is also effective at the composition of as-received tank wastes. Thus, the IX columns would process minimal waste volumes for Cs removal, i.e., placing the IX columns downstream of the TRUEX and/or SREX process would require larger volumes of waste be processed through the IX system due to dilution from the scrub section(s) of the solvent extraction flowsheets. Finally, there is no net increase in the effluent volume from the IX system. The TRUEX flowsheet was ordered second in the integrated flowsheet due primarily to consideration of mercury interactions in SREX. The TRUEX process has been demonstrated to effectively remove mercury present in the tank waste through extraction and subsequent stripping in the carbonate wash stream. The SREX process solvent also effectively extracts mercury from the tank wastes; however, there is currently no known method to re-extract Hg from the SREX process solvent. Consequently, mercury is extracted from the waste in the SREX process and builds up in the solvent, ultimately degrading the process efficiency with regard to Sr removal. This potential problem is largely overcome by placing the TRUEX process in front of the SREX process and extracting the Hg prior to SREX processing. Experimental work is continuing further elucidate the SREX/Hg interaction. Once Hg stripping from the SREX process is viable, it may be advantageous to order the solvent extraction processes in reverse order (SREX then TRUEX) from the standpoint of a further reduction in shielding requirements for the TRUEX portion of the integrated flowsheet.

The proposed disposal routes of the product streams (LAW or HAW) are indicated in Figure 3. The compositions of the input streams to the solvent extraction flowsheets are also shown. Due to time constraints, the flowsheet in Figure 3 was not optimized with respect to impacts on LAW and HAW immobilization. Instead, the conditions tested were those known to work with regard to separation flowsheet performance. A major emphasis in the test was to integrate the unit operations, including LAW and HAW treatments, for a first time evaluation of effects on the overall process. However, the immobilization tests on the separation products were never performed, primarily due to time constraints and funding limitations.

Notable, non-optimal conditions employed in the tests include the HEDPA concentration in the TRUEX strip and ammonium citrate in the SREX Pb strip. The 0.04 *M* HEDPA concentration used in the TRUEX strip feed is a factor of 4 to 10 times too high from the standpoint of final HAW volume since phosphate significantly impacts waste loading in glass. It is also anticipated that citrate would have deleterious effects on LAW grouting operations since it inhibits solidification. Further development efforts in these areas (at a minimum) are required to optimize the separation processes with regard to the overall flowsheet.

AMP-PAN Sorbent

The AMP-PAN sorbent material was procured from Dr. Ferdinand Šebesta of the Czech Technical University and used as received (wet). The first two columns were filled with AMP-PAN/SF02 (0.3-0.6 mm particle diameter) and the third column was filled with an older batch of AMP-PAN material (0.4-0.85 mm particle diameter). The SF designator denotes that the AMP-PAN was dried after formation, improving mechanical properties, then rehydrated, while the AMP-PAN without the SF designator was stored in solution since production. The AMP-PAN/SF sorbent is composed of 85.7 wt% AMP and 14.3 wt% PAN.

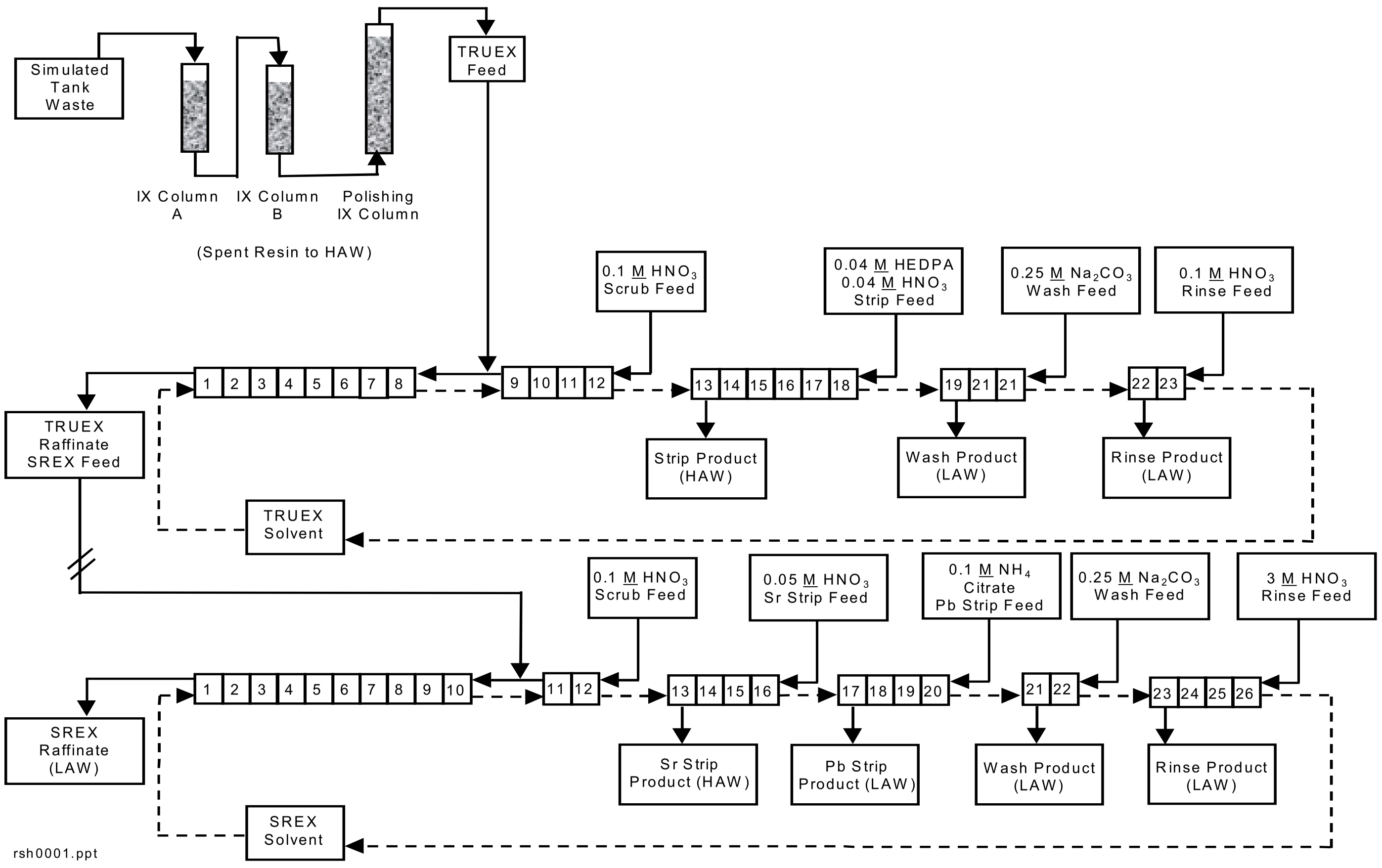

Figure 3. Integrated flowsheet for testing with simulated tank waste.

TRUEX Solvent

The composition of the TRUEX solvent used in these tests was 0.2 *M* CMPO and 1.4 *M* TBP in Isopar L , a branched chain paraffinic hydrocarbon. Two liters of fresh solvent was prepared by the INTEC Quality Control (QC) Laboratory. Approximately three weeks prior to the TRUEX flowsheet test, the entire volume of TRUEX solvent was washed with 0.25 *M* Na_2CO_3 followed by acid rinsing with 0.1 *M* HNO_3 in the 3.3 cm centrifugal contactors. The solvent cleanup utilized 3 stages of carbonate wash (O/A = 5) and 2 stages of acid rinse (O/A = 3). The purity and composition of the washed TRUEX solvent were subsequently tested by determination of the extraction distributions of Am-241 at various HNO_3 concentrations, a procedure previously developed as a quality control measure for the TRUEX solvent [16]. The test was completed 4 days prior to initiating the TRUEX flowsheet experiment. The composition and purity were acceptable and the solvent was subsequently used in the TRUEX flowsheet test.

SREX Solvent

The composition of the SREX solvent used in the integrated flowsheet test was 0.15 *M* 4',4'(5')-di-(tert-butylcyclohexo)-18-crown-6 and 1.5 *M* TBP in Isopar L . Two liters of SREX solvent were freshly prepared by the INTEC QC Laboratory. The crown ether extractant was obtained from Eichrom Industries, Darien IL and was used as received. Approximately two days prior to the SREX flowsheet test, the entire volume of SREX solvent was washed with 0.25 *M* Na_2CO_3 followed by acid rinsing with 3.0 *M* HNO_3 in the 3.3 cm centrifugal contactors. The solvent cleanup utilized 2 stages of carbonate wash (O/A = 5) and 4 stages of acid rinse (O/A = 2). The purity and composition of the washed SREX solvent were then confirmed based on the extraction distribution of Sr-85 from 3 *M* HNO_3, a procedure previously developed as a quality control measure for the SREX solvent. The distribution of Sr from 3 *M* HNO_3 was $D_{Sr} \geq 4$, and indicated the solvent extraction properties were acceptable. The solvent was subsequently used in the SREX flowsheet test.

Analytical

The samples taken during the integrated test were submitted for analysis at the INTEC Analytical Laboratory. Al, Ag, B, Ba, Ca, Cr, Fe, Mn, Ni, Pb, Pd, Re, Rh, Se, Sr, and Zr analyses were performed using Inductively Coupled Plasma Emission Spectroscopy (ICP-ES). Sodium, Cs, and K analyses were performed using Atomic Absorption Spectroscopy. Mercury was determined by fluorescence spectroscopy. Samples of the TRUEX or SREX solvents could not be directly analyzed. Consequently, the organic samples from the TRUEX test were stripped with 0.25 *M* HEDPA in 0.05 *M* HNO_3 at an O/A ratio of 0.2 to quantitatively back-extract all metals with the exception of Hg and a portion of the Re into an aqueous solution. The resulting aqueous phase was analyzed for all metals except Hg. The organic phase, after contact with the HEDPA strip solution, was contacted with 0.25 *M* Na_2CO_3 at an O/A of 0.2 to back-extract Hg and the remaining Re from the organic phase. The carbonate samples were analyzed for Hg and Re. Analyses of the SREX organic samples were similarly performed by back-extraction of the SREX solvent with 0.1 *M* ammonium citrate at an O/A of 0.2. The citrate stripping procedure was conducted twice on each organic sample and the two aqueous fractions combined for analysis. The back extraction of Hg from the SREX solvent is difficult; however, several of the SREX organic samples were stripped twice with 0.1 *M* sodium EDTA at an O/A of 0.2 and the resulting aqueous phases combined and submitted for mercury analysis.

RESULTS AND DISCUSSION

Cesium Ion Exchange

The cesium ion exchange test ran for ~34 hours and processed ~ 45 L of simulated tank waste through the AMP-PAN columns. The flowrate through the system was ~22 bed volumes/hour, based on the first two columns, for the duration of the test. Minor operational problems were encountered during the startup. The columns were initially all configured for up flow. Small voids were observed in the first two columns upon starting the feed pump with minor fluidization of the AMP-PAN in the voids. The system was shut down and the first and second columns were re-configured for down flow to eliminate the voids observed in the bed. The final, polishing column was left in an up flow configuration.

During the test the pressure on the feed pump outlet gradually increased until approximately twenty hours into the run, when the feed pressure exceeded the capacity of the instrument (15 psig). A white, gelatinous precipitate was observed at the entrance (top) of columns A and B. Inspection of the feed tank confirmed precipitate formation in the feed solution. The increased pressure drop through the system was attributed to transfer of the precipitate from the bottom of the feed vessel. The system was shut down for approximately 5 minutes and the pressure gauge in the feed line was replaced with a 60 psig (maximum) gauge. After re-starting the feed pump, the feed pressure held steady at 20 – 21 psig for the duration of the experiment. The remaining feed solution was continuously re-circulated for several hours through a 5 micron filter using a masterflex pump while the ion exchange system was operating. The on-line filtration appeared to remove the precipitate from the feed solution.

Past experience indicates that precipitate formation, typically $NaNO_3$ with minor amounts of $Al(NO_3)_3$ and metal phosphates, occurs in freshly prepared tank waste simulant. The precipitate is filtered from the simulant after preparation and prior to experimental testing. Once the solution is filtered, it is typically stable for several months at room temperature. Precipitate formation from the simulated feed has been observed to increase as temperature decreases. The feed used in these tests was prepared, allowed to settle overnight, and partially filtered the next day through 5-μm filters. The solution was allowed to stand overnight and the entire volume was re-filtered through a new 25 micron filter the next day. The ion exchange portion of the test was started ~4 days later and solids were not observed in the simulated feed solution at the time of startup. It is postulated that lower temperatures in the laboratory during the ion exchange testing (during the night) facilitated the additional precipitate formation in the feed solution.

Effluent samples were taken for Cs analysis from columns A and B hourly for the first 15 hours of operation, followed by three samples of the effluents taken every 3 hours. Subsequent samples were taken every 5 hours until the end of the IX test. The full gamut of metals analyses were requested on the column A and B samples taken at 1, 7, and 12 hours. Cesium breakthrough results are shown in Figure 4, where the ratio of cesium concentration in the column effluent (C) divided by the cesium concentration in the feed (Co) is plotted versus bed volumes of solution processed. A second order kinetic equation [3] was used to predict cesium effluent concentration as a function of the volume of feed processed (at a given feed concentration). Using the cesium concentration in the feed (130 mg/L) and referring to an AMP-PAN isotherm previously obtained [3], a maximum equilibrium cesium capacity of approximately 64 mg Cs per gram AMP is obtained. Using the mass transfer coefficient derived from AMP-PAN testing in 1.5 cm^3 columns, breakthrough curves for the two 60 cm^3 columns were predicted [3]. The predicted breakthrough curves are also shown in Figure 4 and indicate excellent agreement with the experimental data. Scale-up of the AMP-PAN ion exchange process from 1.5 cm^3 columns to 60 cm^3 columns was accurately predicted using the kinetic model, even though cesium feed concentrations varied by greater than an order of magnitude between the different column tests.

Fifty-percent cesium breakthrough was observed at approximately 280 bed volumes for column A and 525 bed volumes for column B. It is important to note that complete cesium breakthrough of column A occurred before 1% cesium breakthrough occurred in column B. This is significant since it is indicated that complete loading of the first column, in a two column series arrangement, can be realized while maintaining the desired Cs removal, thus minimizing the total amount of sorbent that is required.

Total dynamic capacity (DC) was calculated using the following relationship:

$$\mathrm{DC} = \frac{\int_0^v (Co - C)dv}{\mathrm{M}}$$

Where:

v = volume at a specified breakthrough, Liters
Co = initial Cs concentration, g/L
C = Cs concentration in the column effluent, g/L
M = mass of sorbent, grams (dry weight)

Numerical integration of the above equation over the volume of feed processed at 50% and 100% breakthrough yields the dynamic capacity for each column (shown in Table 4). The value of C in the equation is obtained from the kinetic model.

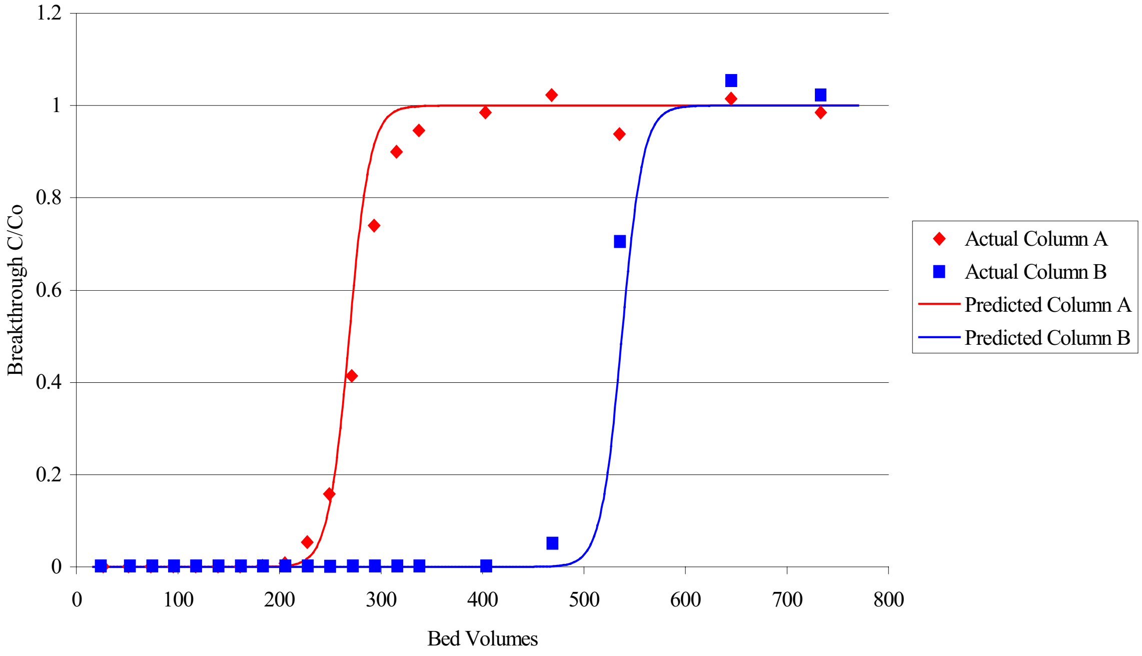

Figure 4. Cesium breakthrough curves for the AMP-PAN IX columns A and B. (Feed 130 mg/L Cs, 60 mL bed volume per column, 1.32 L/hr flowrate).

Table 4. Dynamic capacities (DC) of AMP-PAN columns.

	DC at 50% Breakthrough mg Cs/g AMP-PAN	DC at 100% Breakthrough mg Cs/g AMP-PAN
Column A	62.2	64.0
Column B	63.1	64.0

Dynamic capacities at 100% breakthrough agree well with the AMP-PAN equilibrium isotherm, indicating that the sorbent is at or very near equilibrium. Dynamic capacities at 50% breakthrough are very close to the equilibrium isotherm capacity, indicating that nearly all of the AMP-PAN sorption capacity can be realized.

Breakthrough data were not obtained for the third, polishing column. However, a composite sample of the effluent from the IX portion of the test (used as feed to the TRUEX process) indicated a Cs concentration of 0.219 mg/L. This represents a cumulative breakthrough of C/Co = 0.168%, or an overall removal efficiency of 99.83%. Previous ion exchange testing performed at the INTEC with simulated tank waste indicated that some simulant components may interfere with the atomic absorption analysis of cesium and result in a bias toward higher cesium concentrations than actual [17]. Analytical results from this test show cesium concentrations from Column A effluent range from 0.15-0.16 mg/L from 0 to 160 bed volumes, and from Column B effluent range from 0.19-0.3 mg/L from 0-400 bed volumes. Due to the high affinity AMP-PAN has for cesium, it is highly unlikely that this level of cesium passed through both columns early in the test. Because the exact cesium analytical bias is unknown, the results are reported as >99.83%.

The percentages of specific components removed from the simulated feed solution by the AMP-PAN columns are listed in Table 5. Rhenium, barium, chromium, fluorine and nickel were all essentially not sorbed on the AMP-PAN. The small removal efficiencies shown in Table 5 are all within the uncertainty associated with the analytical methods. Zirconium results are inconclusive, the removal efficiency shown in Table 5 corresponds to the upper limit of analytical uncertainty, indicating that Zr was either not sorbed, or a small amount (<10%) of Zr was sorbed. The validity of the high Ag removal efficiency is also uncertain. Silver nitrate was added to the initial IX feed and the feed had been filtered prior to entering the IX columns. It is possible that Ag precipitated in the feed vessel or was nearly quantitatively removed by the AMP-PAN columns. Further testing is needed to determine if Ag is indeed sorbed by AMP-PAN.

Table 5. Percent removal of various components during the AMP-PAN IX test.

Component	Simulated Waste (IX Feed) (mg/L)	IX Effluent (TRUEX Feed) (mg/L)	% Removed
Cs	130	<0.219	>99.83
Ag	4.45	0.048	98.91
Re	667	659	1.3
Ba	15.0	14.7	2.1
Cr	247	238	3.6
F	4930	4490	8.9
Ni	108	104	3.5
Zr	131	117	11

TRUEX Solvent Extraction

Contactor Operation

The TRUEX solvent extraction portion of the test was designed to remove predominately the actinides and mercury from the simulated tank waste. The TRUEX test was operated a cumulative of 71.3 hours and processed 40.1 L of simulated feed. Note that the TRUEX portion of the flowsheet was operated simultaneously with the Cs removal portion of the integrated test for the duration of ion exchange run (~34 hours).

Startup and operation of the equipment proceeded as follows: The centrifugal contactor motors were started at 2700 rpm. Aqueous solution flows, with the exception of the simulated tank waste, were established in the contactors. A startup solution consisting of 1.5 *M* HNO_3, 0.56 *M* $Al(NO_3)_3$, and 1.2 *M* $NaNO_3$ was used in place of the simulated tank waste while solution flows were being established in the system. The organic (TRUEX) solvent flow was started after aqueous flows were established. When organic was observed entering stage 20, in the carbonate wash section, the simulated tank waste (raffinate from ion exchange) was started in place of the initial startup solution, marking time zero for the test.

During the course of the run, flooding was never observed in any of the effluent streams. At several points during the run, the contactors and feed pumps were simultaneously, momentarily shut down (<5 minutes), and stage 13 in the strip section and stage 19 in the carbonate wash section were opened and inspected for precipitates. As part of the inspection process, the solutions in the stages were drained into glass separatory funnels for visual inspection. The solutions were then returned to their respective contactor housings prior to restart of the system in order to minimize disruption of steady state operation. Precipitates were never observed in the strip stage. During visual inspection of the stage 19 contactor after approximately 4 hours of operation, a slight band of rust/orange colored precipitate was observed on the exterior of the contactor rotor. The solutions in the contactor indicated no visible signs of precipitates. Based on previous experience, the precipitate was HgO, which could potentially be eliminated by increasing the carbonate feed flowrate, thus decreasing the O/A and the mercury concentration in the carbonate wash section. The decision was made to continue the run under the previous conditions to determine if the precipitation problem would worsen or was a condition that had occurred during startup. The stages were rechecked several more times and the HgO precipitate on stage 19 appeared slightly worse. At ~20 hours into the run, the carbonate wash feed flowrate was increased slightly from 7.9 mL/min to 8.3 mL/min. The rotor was inspected several more times without noticeable difference in the amount of HgO deposited on the rotor. At ~40 hours into the run, the carbonate flowrate was again increased from 8.3 mL/min to 9.3 mL/min to determine if the HgO on the rotor would re-dissolve. This flowrate was maintained for the duration of the run with no discernable, visual difference in the amount of HgO present on the rotor. Aside from the slight HgO precipitation, other problems were not encountered during the test.

Product and feed flowrates during the run were determined by actual measurement of the effluent streams and also by calculation from feed tank depletion and product tank filling rates. The different methods of determining flowrates were used to establish a time-weighted average for each flowrate during the course of the run. The actual and target flowrates, as well as organic-to-aqueous (O/A) phase ratios are listed in Table 6. Note that the average carbonate flow rate reported reflects adjustment of the flows to eliminate/reduce the HgO precipitation problem

Table 6. Flowrates and O/A ratios for the TRUEX flowsheet test.

		Flowrate (mL/min)		O/A Ratio		Total Flow
Section	Phase	Desired	Actual	Desired	Actual	(mL/min)
All	Org.	6	6.04	---	---	---
Extraction	Aq.	14	13.23	0.43	0.46	19.27
Scrub	Aq.	4	3.85	1.5	1.57	9.89
Strip	Aq.	8	8.64	0.75	0.70	14.68
Carbonate Wash	Aq.	8	8.65	0.75	0.70	14.69
Acid Rinse	Aq.	1	0.97	6.0	6.2	7.01

encountered in the carbonate wash section. The actual flowrates shown in Table 6 are comparable to the target values and were used for all subsequent calculations.

Based on the initial volume of 1500 mL of TRUEX solvent, a run time of 71.3 hours, and the organic flowrate of 6.04 mL/min, the number of times the solvent was recycled through the centrifugal contactors was calculated to be 17.2 times.

Real Time Product Concentrations

The primary species of interest for evaluating TRUEX flowsheet characteristics were Eu, Fe, Re, and Hg. Samples of the aqueous raffinate and strip product were taken every half-hour for the first 3 hours for Eu, Fe, and Re analyses to determine when steady state conditions were achieved. Additionally, mercury analyses were performed on the aqueous raffinate samples to evaluate steady state conditions (Hg was not requested on the early strip product samples since past experience indicated mercury was not effectively stripped by HEDPA).

Beginning at 5 hours into the run, product samples of the aqueous raffinate, strip, carbonate wash, acid rinse, and organic were taken at 5 hour intervals for analysis of Eu, Fe, Re, and Hg. Additional metals analysis (Ag, Se, Ru, Pd, Al, B, Ca, Cs, Cr, K, Mn, Mo, Na, Ni, Pb, Sr, Zr, Rh, Cl, and F) were requested on the raffinate, strip, wash, and organic product samples taken at 20, 40, and 60 hours. The additional metals analyses were also requested on the rinse product sample taken at 40 hours. Samples were also taken of each product stream just prior to shutdown (~72 hours) and composite samples were obtained from the product tanks after concluding the experiment. Analyses of the composite samples provide an indication of “average” behavior and flowsheet performance for each of the species over the duration of the experiment. The entire array of metal analysis was requested for the final product and composite samples.

Aqueous Raffinate. The concentrations of Fe, Re, and Hg in the aqueous raffinate are shown as a function of time in Figure 5. Europium concentrations are not indicated since the reported results were below the analytical detection limits for all raffinate samples. It is apparent from the data in Figure 5 that the extraction section was operating at steady state, with regard to each of these elements, within the first five hours of testing. The solid lines represent the analytical results for the composite sample taken at the end of the run. There is excellent agreement between the steady state and composite sample analyses.

The time to reach steady state with regard to these elements was predominately a function of contactor size and flowrates. The total volumetric capacity (organic plus aqueous) of the 3.3 cm contactors is 25 L/hour. Based on the flowrates chosen for this test, the volumetric throughputs were 1.156 L/hr (19.27 mL/min) in the extraction section and 0.88 L/hr in the strip section. Had the throughputs used in the test been closer to the maximum capacity of the contactors, the time required to achieve steady state would have been substantially less, probably within the first 30 minutes of operation. The fact steady state was achieved at approximately the same time for each of the elements examined indicates that chemical kinetics of the extraction operation were not an issue.

The analytical results for other species of interest in the aqueous raffinate are listed in Table 7. The analysis of the feed solution to the TRUEX flowsheet is indicated for comparison. Analyses for the composite aqueous raffinate sample, which provide an realistic measure of the average composition for the duration of the test, are also indicated in Table 7 for comparison. Note that the percentage of each component extracted from the TRUEX feed is based on the composite (normalized) sample analysis (refer to Table A-9 in the Appendix). The important point is that the analytical results for the raffinate samples (Aq. Raff.) and composite sample (Aq. Raff. Comp) are very consistent, but typically lower than those of the feed. Lower values are expected due to dilution of the feed by ~30 vol. % from the introduction of the scrub solution. The analyses over time are very consistent for each of the species listed in Table 7, indicating that the extraction section was indeed operating at steady state with regards to each of these species. The results also indicate that Mo, Zr, Ru, and perhaps Rh and Cl were the matrix components extracted from the feed by the TRUEX process to any significant extent.

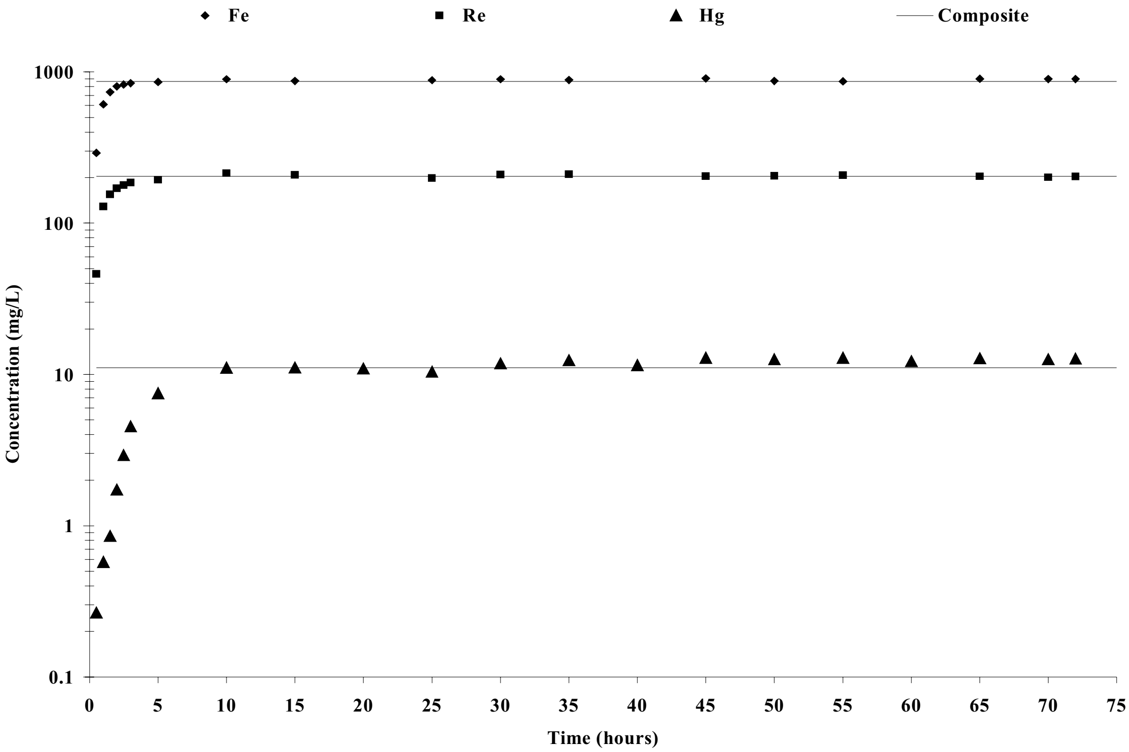

Figure 5. Aqueous raffinate approach to steady state.

Table 7. Analytical results (in mg/L) of several metals in the raffinate samples.

Sample	Al	Ag	B	Ba	Ca	Cr	K
Feed	14600	0.048	198	14.7	1870	238	5200
Aq. Raff. 20 hrs	10400	0.046	140	10.8	1350	169	3640
Aq. Raff. 40 hrs	10300	0.057	139	10.4	1330	172	3770
Aq. Raff. 60 hrs	10200	0.067	138	10.7	1330	167	3870
Aq. Raff. Composite	10200	0.065	136	10.7	1330	170	3520
Percent Extracted [a]	<1	<1 [b]	<1	<1	<1	<1	<1
	Mn	Mo	Na	Ni	Pb	Rh	Ru
Feed	427	121	29700	104	196	0.200	2.06
Aq. Raff. 20 hrs	305	2.92	29000	77.5	135	0.237	1.33
Aq. Raff. 40 hrs	302	3.06	20300	75.5	142	0.227	1.38
Aq. Raff. 60 hrs	300	2.90	19500	75.7	132	0.202	1.21
Aq. Raff. Composite	297	2.83	20000	76.1	129	0.246	1.31
Percent Extracted [a]	<1	97	<1	<1	<1	21 [b]	8
	Se	Sr	Zr	F	Cl		
Feed	2.31	93.0	117	4490	1540		
Aq. Raff. 20 hrs	1.70	65.8	60.1	3070	958		
Aq. Raff. 40 hrs	1.47	65.3	59.6	2750	910		
Aq. Raff. 60 hrs	1.56	64.6	58.9	3030	908		
Aq. Raff. Composite	1.62	63.9	59.1	4560	875		
Percent Extracted [a]	<10	<1	29	<1 [b]	10 [b]		

[a] Percentage extracted based on the normalized composite sample, refer to Appendix Table A-9.

[b] Material balance (non-normalized) was poor for this component (>±10%).

Strip Product. The concentrations of Eu, Fe, and Re in the strip product are shown as a function of time in Figure 6. Mercury is not included in Figure 6 since the concentration of Hg in the strip product was very low (≤1 mg/L) for the duration of the test. From the data in Figure 6, it is apparent that the stripping section was operating at steady state conditions, with regard to these elements, within the first five hours of the experiment. The solid lines represent the analytical results for the composite strip product sample taken at the end of the run. There is excellent agreement between the steady state and composite sample analyses.

The analytical results for the species removed from the feed in the extraction section and entering the strip section are listed in Table 8. The analyses for the feed solution to the TRUEX flowsheet and the composite aqueous raffinate sample are indicated in Table 8 for comparison. Note that the percentage of each component extracted from the TRUEX feed and recovered in the strip product is based on the composite (normalized) sample analysis (refer to Table A-9 in the Appendix). Note that analytical results for the strip product samples taken over time are consistent with the composite sample. These results confirm that the strip section was operating at steady state with regard to these components. Of the metals removed from the feed in the extraction section, Eu, Mo, and Zr were efficiently recovered in the strip section. Analytical results for Ru, Rh and Cl were reported as "less-than" values for the strip product samples taken as a function of time, indicating these components were not removed from the organic in the strip section. Note that the composite sample did contain rhodium, and although the material balance was extremely poor for this element (220%), >21.1% (essentially all of the extracted Rh) was recovered in the strip section. Only 0.26% of the Hg that was extracted from the feed was recovered with the strip product. The data indicate the strip section was operating very efficiently with regard to Eu, Mo, and Zr stripping.

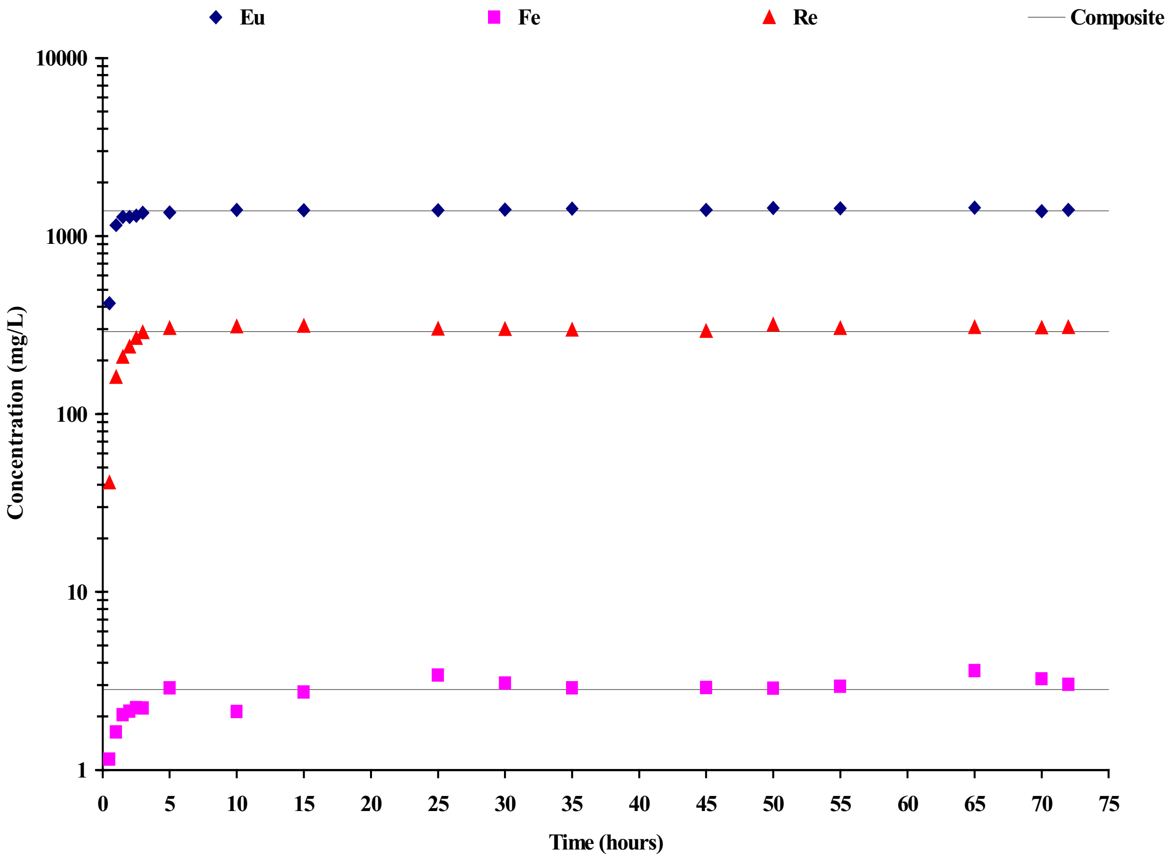

Figure 6. Strip product approach to steady state.

Table 8. Analytical results (in mg/L) of several metals in the strip product samples.

Sample	Eu	Fe	Re	Mo	Zr
Feed	1066	1297	659	121	117
Strip Product 20 hrs	1364	4.00	295	151	43.2
Strip Product 40 hrs	1384	2.60	300	141	43.9
Strip Product 60 hrs	1341	2.40	309	128	41.1
Strip Prod. Composite	1382	2.84	289	139	40.7
Percentage Extracted	>99.999	0.23	55.7	97	29
Percentage in Strip [a]	99.64	0.19	36.3	95	29

[a] Percentage in strip based on the normalized composite sample, refer to Appendix Table A-9.

Carbonate Wash Product. The concentrations of Hg and Re in the carbonate wash product are shown as a function of time in Figure 7. From the data in Figure 7, it is apparent that the carbonate wash section was never actually operating at steady state conditions with regard to Hg. This is understandable due to precipitation of HgO in the wash stages and also due to the manual increase in carbonate feed flowrate in an effort to prevent this precipitation. Recall that the carbonate flow was increased slightly at ~20 hours and again at ~40 hours into the run.

Whether steady state operation was actually achieved with regard to Re is also questionable. That there are not sharp breaks in the concentration profile indicates precipitation was not an issue with Re.

The solid lines represent the analytical results for the composite product sample taken at the end of the run. Although steady state was not demonstrated in the carbonate wash section, the composite sample appears representative of the average run composition for both Hg and Re. The ruthenium and chloride that extracted from the simulated feed were quantitatively recovered in the carbonate wash product. Based on the composite sample, 8.39% of the Ru in the feed (recall that 8.4% was extracted) was recovered in the wash product. Approximately 100% of the extracted chloride was recovered in the carbonate wash based on the composite sample. This is not surprising since the Hg extracts as the chloride species, $HgCl_2$, and is likely recovered as this species in the carbonate wash.

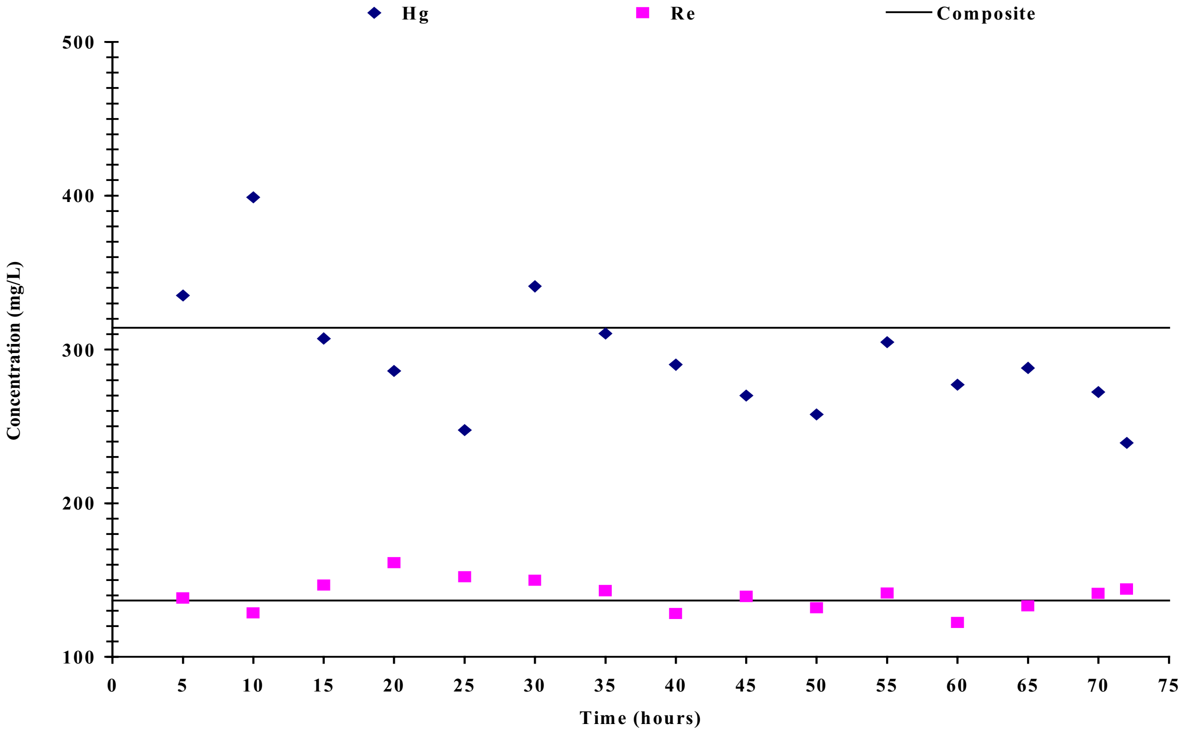

Figure 7. Concentrations of Hg and Re in the carbonate wash product as a function of time.

Acid Rinse Product. The concentrations of Fe and Hg in the rinse product are shown as a function of time in Figure 8. Iron and Hg were above the analytical detection limit in the time wise samples, while Eu and Re were consistently reported as below detection limits. Low metals concentrations in the rinse product are typical; the function of the acid rinse is simply to re-acidify the organic prior to recycling it to the extraction section. The composite sample analyses are not included in Figure 8 since the composite concentration of Hg (0.281 ppm) and Fe (3.207 ppm) in the composite were substantially higher than the time wise samples. Although the reason for this discrepancy between the composite and time wise samples is uncertain, it should be noted that the analytical results are all very close to the detection limits, which lends to the uncertainty associated with the analytical data. From the data in Figure 8, it appears that the acid rinse section was operating at steady state conditions.

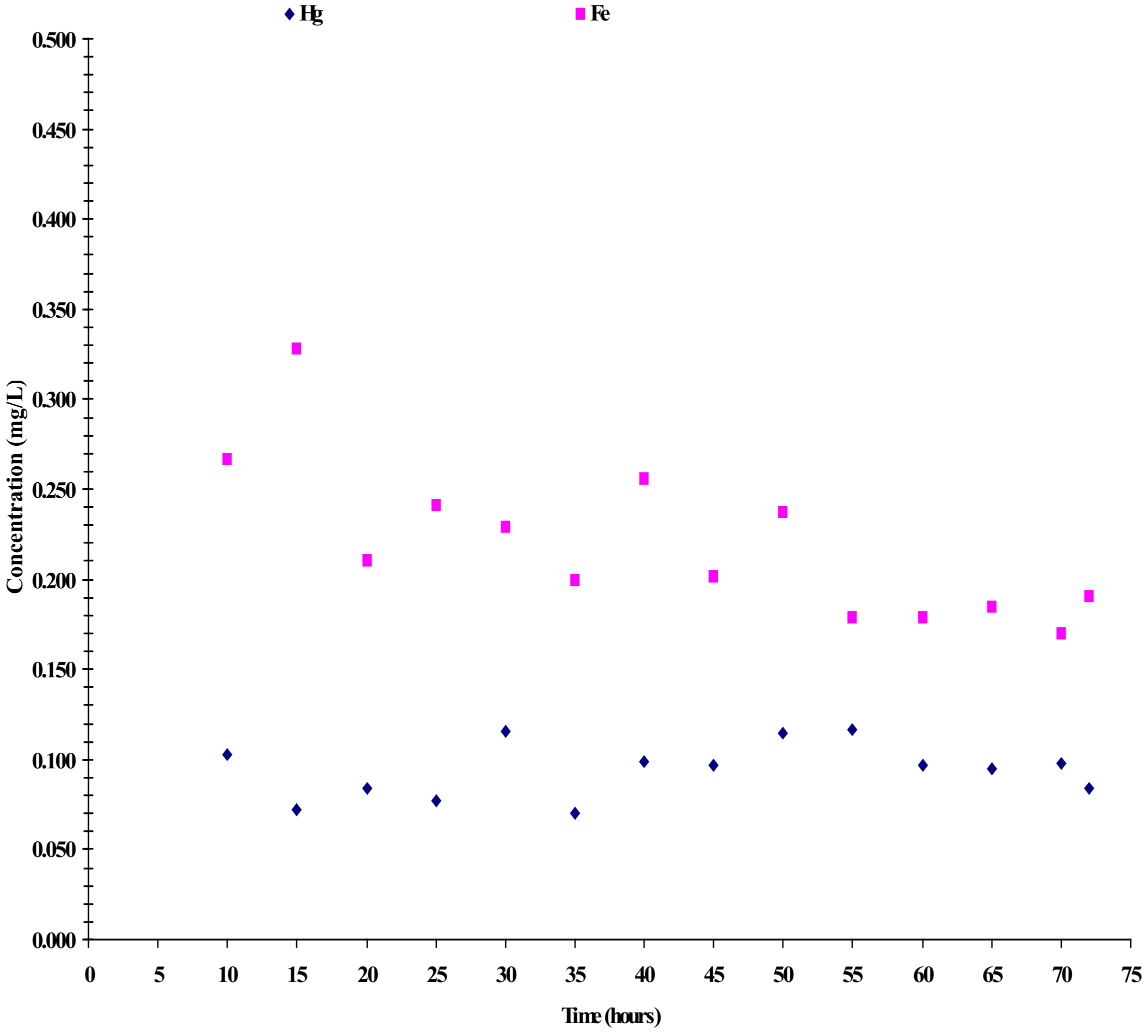

Figure 8. Concentrations of Hg and Fe in the rinse product as a function of time.

Organic Product. The concentrations of Hg and Fe in the organic product stream, as it exited from the acid rinse section, are shown as a function of time in Figure 9. Iron and Hg were present above the analytical limits of detection, Eu and Re were consistently reported as below detection limits. It is interesting to note that the Hg analysis for the samples taken at 20, 40, 60 hours, and the composite sample were consistently low relative to the other samples. The Fe results for the above mentioned samples were consistently high. While the reason for this discrepancy is uncertain, it should be noted that these samples were run in a different analytical batch since the full range of metals analysis were requested. Consequently, it is speculated that the discrepancy is likely attributable to analytical error. From the data in Figure 9, it appears that Hg and Fe concentrations were not increasing in the solvent as a function of time. An average of 1.7 mg/L Al, 3.8 mg/L B, 7.7 mg/L Ca, 0.06 mg/L Cr, 0.6 mg/L K, 1.5 mg/L Mo, 7.4 mg/L Na, and 2.6 mg/L Zr were detected for the three product samples taken at 20, 40, and 60 hours. The results for each of these elements were consistent in the three different samples. The data clearly indicate that none of these matrix components appear to have been building up in the solvent.

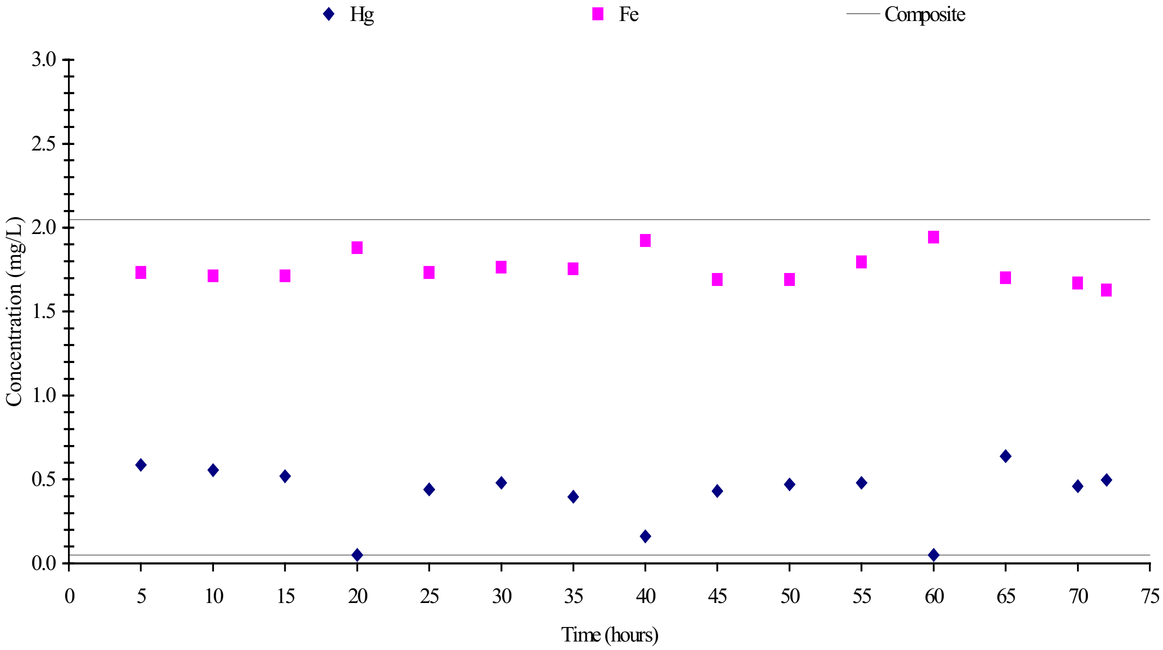

Figure 9. Concentrations of Hg and Fe in the organic product as a function of time.

Composite Product Concentrations and Stagewise Distribution Coefficients

An important piece of information from the extended flowsheet test is the composition of all final products from the TRUEX run. Composite samples of the product solutions were anticipated to be representative of the products that would be expected in full scale, continuous operation. Concentrations of the primary metals of concern, e.g., Eu, Fe, Re, and Hg, and the percentage of each component in the various composite products are indicated in Table 9. Average flowrates for the duration of the test, final total volumes, and the material balances for these components are also indicated. The stagewise distribution coefficients for these species were also calculated for each stage after shutdown and are presented in Table 10.

Europium. Based on the data in Table 9 for Eu, used as a surrogate for Am-241, >99.999% was extracted from the feed. Note that if Am-241 is removed from the actual tank wastes, the remaining actinides will also be removed since americium is the most difficult to extract (i.e., Am has a lower distribution coefficient than most actinides). Also, the concentration of Eu used in the simulated feed is orders of magnitude larger than the actinides and rare earth elements present in the actual wastes. The material balance of 106% for Eu is excellent, with most of the discrepancy attributed to the strip product sample. The distribution coefficients were very high for stages 5 through 8 in the extraction section. Distribution coefficients could not be determined on stages 1 through 4 since the Eu concentration in both the organic and aqueous phases were <0.04 mg/L for these stages. The distribution coefficient results indicate an excessive number of stages for Eu extraction; however, the required number of extraction stages was based on Hg removal (*vide infra*). The distribution coefficients were large enough in the scrub section ($D_{Eu} > 4$) to insure very little Eu scrubbing. Virtually all of the extracted Eu was recovered in the strip product (106% of the Eu in the feed), which is consistent with the low Eu distribution coefficients measured in the strip section ($D_{Eu} \leq 0.35$). Distribution coefficients in the carbonate wash section could not be evaluated since the results for these samples were reported as below detection limits, although a small amount of the Eu was detected in the carbonate wash product (4.41 mg/L, or 0.38%). These results indicate the effectiveness of the TRUEX process for actinide removal/recovery from the existing tank wastes currently stored at INTEC. With this high removal efficiency for the actinides, the LAW grout will be well below the 10 nCi/g NRC Class A LLW limits.

Table 9. Primary components and parameters of the composite product samples.

Component	Aqueous Feed	Aqueous Raffinate	Strip Product	Carbonate Wash	Acid Rinse	TRUEX Organic[a]	Material Balance[b]
Eu (ppm)	1066	<0.01	1382	4.41	0.034	0.04	
% per stream	---	<0.001	105.8	0.38	---	0.002	106%
Fe (ppm)	1297	867.1	2.84	0.16	3.21	2.05	---
% per stream	---	94.32	0.18	0.01	0.03	0.10	94.5%
Re (ppm)	658.5	203.8	289.4	136.7	0.72	<0.08	---
% per stream	---	43.66	35.83	19.16	0.01	<0.01	98.7%
Hg (ppm)	422.0	11.1	1.12	314	0.28	<0.05	---
% per stream	---	3.71	0.22	68.68	0.01	<0.01	72.6%
Final Volume[c] (L)	---	54.7	32.8	36.6	3.92	0.69	---
Flow (mL/min)	9.38	13.23	7.64	8.65	0.97	6.04	---

[a] Organic composition not included in the material balance since solvent was recycled.

[b] Calculated as a percentage of out/in.

[c] Does not account for samples removed during the course of the experiment or the volume remaining in the stages after shutdown.

Table 10. Measured stagewise distribution coefficients from the TRUEX test.

Section	Stage #	Eu	Fe	Re	Hg
Extraction	1	---	0.75	0.78	0.59
	2	---	0.92	0.67	0.51
	3	---	0.94	0.68	0.47
	4	---	0.96	0.65	0.50
	5	>36	0.93	0.73	0.72
	6	>35	0.92	0.76	0.82
	7	>31	0.92	0.80	1.3
	8	25	0.69	0.81	1.3
Scrub	9	14	0.12	2.4	7.7
	10	12	0.06	5.3	16
	11	7.6	0.03	12	31
	12	3.8	0.04	19	34
Strip	13	0.02	---	2.0	520
	14	<0.01	---	1.2	133
	15	<0.35	---	1.1	1000
	16	---	---	1.0	1250
	17	---	---	1.0	1360
	18	---	---	1.0	1150
Carbonate Wash	19	---	---	0.06	0.004
	20	---	---	0.06	9.4
	21	---	---	<0.4	23
Acid Rinse	22	---	---	---	0.9
	23	---	---	---	0.6

Iron. Iron in tank wastes has been observed to extract into the TRUEX solvent, but is effectively scrubbed with dilute nitric acid. The distribution data in Table 10 indicate that some iron extracted since D_{Fe} ~0.9 in the extraction section, and D_{Fe} <0.1 in the scrub section. The iron concentration in the organic phase exiting the scrub section was low, 2.3 mg/L, and the distribution data were meaningless for stages13 through 23. The distribution data are consistent with the results in Table 9, which indicate most of the Fe in the feed exited with the aqueous raffinate.

Rhenium. Rhenium was added to the simulated feed (as perrhenate, ReO_4^-) as a surrogate for technetium (as pertechnetate, TcO_4^-). The results in Table 9 indicate approximately 56% of the Re was extracted from the simulated waste. This is consistent with the distribution coefficients in Table 10, which indicates D_{Re} =0.65 to 0.81 in the extraction section and D_{Re} >2.4 in the scrub section. Approximately 36% of the Re was stripped with the HEDPA solution, consistent with the strip D_{Re} ~1. Roughly 19% of the Re exited with the carbonate wash stream, which was very effective at removing Re from the TRUEX solvent, as indicated by the D_{Re} of 0.06 to <0.4 in the carbonate wash stages. The material balance for Re was very good at 98.7%. It should be noted that the tank wastes currently stored at INTEC are below the limits of concern for Tc in Class A grout. However, since pertechnetate is extremely mobile in the environment, it is desirable to understand the behavior of this species for any treatment option.

Mercury. One of the goals in the TRUEX portion of the integrated test was to determine the efficiency of Hg removal from the simulated waste. To this end, the flowsheet was modified from previous tests to include more extraction stages and higher flowrates in the carbonate wash section to recover as much of the mercury as possible from the wastes. Mercury removal is important from the aspect that it is a RCRA regulated metal present in significant quantities in the tank wastes. Use of the TRUEX process to partition Hg into a relatively clean (low metals and radionuclide content) stream is viewed as an advantage if Hg must ultimately be removed and dispositioned in accord with RCRA regulations. Furthermore, mercury is extracted in the SREX process, but cannot currently be effectively stripped from the SREX solvent. Consequently, Hg tends to build up in the SREX solvent and would eventually poison the organic phase, impacting Sr extraction. Data from the composite samples indicate 96.3% of the Hg was extracted from the feed, which is consistent with the extraction section mercury distribution ratios of ~1. Very little of the Hg was scrubbed from the solvent as manifest from the available scrub section distributions of $D_{Hg} > 8$. The HEDPA strip solution was also ineffective at stripping Hg from the solvent, with distribution ratios of $D_{Hg} > 100$ in the strip section. A mere 0.22% of the mercury exited with the strip product. Table 9 indicates 68.7% of the mercury exited with the carbonate wash product. The overall material balance of mercury was quite low at 72.6%, consistent with the precipitation of HgO encountered in stage 19 of the carbonate wash section. The discrepancy in the overall Hg material balance is attributed to the error introduced by HgO precipitation.

An attempt was made to determine the mass of mercury that precipitated and remained on the contactor rotor and in the contactor housing of stage 19 during the course of the test. The stage 19 contactor was filled and soaked with known volumes of 1.1 *M* HNO_3 to dissolve the HgO precipitate. The contactor required three separate soaks with nitric acid before it visually appeared free of the orange HgO precipitate. The fractions were collected and submitted for Hg analysis. Based on the analytical results and volume of nitric acid used for dissolution, the total mass of mercury precipitated in stage 19 was calculated to be 1.96 g Hg (3510 mg/L Hg in 559 mL of 1.1 *M* HNO_3). Based on this analysis, an estimated 11.6% of the mercury in the feed was recovered from the precipitate in stage 19. Thus, accounting for the precipitated Hg recovered, the overall mercury material balance becomes (72.6% + 11.6% =) 84.2%, which is still somewhat low. This indicates that not all of the precipitated mercury was recovered or that analytical errors are associated with predominately the carbonate wash product, aqueous raffinate, and/or feed sample.

In general, the TRUEX portion of the flowsheet indicated relatively good Hg removal from the simulated tank waste. Further work is required to enhance mercury removal, if necessary, and to eliminate the precipitation of Hg in the carbonate wash section. Several more extraction stages and/or a slight modification in the extraction section O/A could be used to enhance the Hg extraction. Likewise, the O/A in the carbonate wash section could be altered to eliminate the HgO precipitation.

Other Matrix Components. The samples of the composite products were also analyzed for a variety of matrix metals, both micro and macro constituents in the simulated tank waste, to evaluate or further confirm their behavior in the TRUEX process. The results for these constituents, expressed in terms of percentage of each element in the different product streams relative to the feed composition, are listed in Table 11. Of the metals listed in Table 11, Mo, Ru, Zr, and perhaps Rh and Cl, were extracted from the feed to any measurable extent. Less than 1% of the remaining elements (Al, Ag, B, Ca, Cr, K, Mn, Na, Ni, Pb, Se, Sr, and F) were extracted from the feed. The percentage of Ag, Rh, F, and Cl extracted from the feed is relatively uncertain since the material balances for these species were typically very poor or marginal, at 190% for Ag, 220% for Rh, and 144% for F. Note that Pd was added to the initial tank simulant but was found at levels less than the analytical detection limits of <0.07 mg/L in all samples, indicating

Table 11. Percentage of other matrix components in the composite products.

Stream	Al	Ag	B	Ba	Ca	Cr	K
Aqueous Raffinate	98.56 (99.98)[a]	190.1 (100)	96.95 (100)	103.1 (99.97)	100.0 (99.92)	100.8 (99.96)	95.49 (99.97)
Strip Product	<0.0004 (0)	<49.6 (0)	<0.03 (0)	<0.06 (0)	0.006 (0.006)	0.010 (0.010)	0.001 (0.002)
Carbonate Wash	0.001 (0.001)	<56.2 (0)	<0.03 (0)	<0.17 (0)	0.050 (0.050)	0.011 (0.011)	0.002 (0.002)
Acid Rinse	0.022 (0.023)	<6.30 (0)	<0.04 (0)	0.03 (0.03)	0.021 (0.021)	0.024 (0.024)	0.021 (0.022)
Organic Product	0.012	<200	<1.6	<0.23	0.26	0.014	0.002
Mass Balance[b]	98.6	190	96.95	103.2	100.1	100.9	95.52

Stream	Mn	Mo	Na	Ni	Pb	Rh	Ru
Aqueous Raffinate	98.09 (99.98)	3.30 (3.35)	94.99 (99.98)	103.4 (99.89)	92.68 (99.50)	173.7 (78.94)	89.43 (91.61)
Strip Product	<0.002 (0)	93.86 (95.27)	0.003 (0.003)	0.033 (0.032)	0.29 (0.31)	46.34 (21.06)	<3.49 (0)
Carbonate Wash	<0.002 (0)	1.28 (1.30)	0 (0)	0.060 (0.058)	0.17 (0.18)	<47.6 (0)	8.19 (8.39)
Acid Rinse	0.023 (0.023)	0.082 (0.083)	0.013 (0.014)	0.024 (0.024)	0.022 (0.023)	<5.34 (0)	<0.44 (0)
Organic Product	<0.01	0.6	0.02	<0.1	0.13	<170	<14
Mass Balance[b]	98.12	98.52	95.01	104	93.16	220	97.6

Stream	Se	Sr	Zr	F	Cl	Pd
Aqueous Raffinate	98.75 (100)	96.84 (99.98)	71.49 (71.43)	143.3 (99.86)	80.20 (90.10)	348.5 (100)
Strip Product	<4.59 (0)	<0.01 (0)	28.46 (28.43)	0.203 (0.142)	<0.94 (0)	<82 (0)
Carbonate Wash	<5.19 (0)	<0.01 (0)	0.133 (0.133)	<0.06 (0)	8.82 (9.90)	<92.3 (0)
Acid Rinse	<0.58 (0)	0.021 (0.022)	0.011 (0.011)	<0.01 (0)	<0.12 (0)	<10.4 (0)
Organic Product	<19	<0.04	0.63	<0.2	<3.7	<330
Mass Balance[b]	98.75	96.86	100.1	144	89.0	349

[a] Normalized percentages for a 100% material balance.

[b] Material balance based on sample analysis, calculated as out/in*100%. Organic product was not included in the material balance since the organic was recycled. Normalized organic percentages are not reported.

too little was added during chemical make-up, or the Pd precipitated from the feed solution. This is also reflected in the extremely poor Pd material balance of 349%.

The strip product contained most of the extracted Mo and Zr, indicating the HEDPA was effective at stripping these components from the TRUEX solvent. It also appears that the extracted Rh was quantitatively removed from the solvent by the HEDPA strip solution. It should be noted that the Rh results are uncertain based on the extremely poor material balance of 220%.

The carbonate wash section was effective at stripping Cl and Ru from the TRUEX solvent. The chloride present in the carbonate wash product is not surprising since Hg is known to extract into the TRUEX product as $HgCl_2$ [9]. Confirmation of this phenomenon is reinforced when observing the measured concentrations of Cl and Hg in the carbonate wash product, based on the composite samples. The concentration of Hg in the wash product was analytically determined to be 314 mg/L or 0.00157 *M* Hg. Likewise, the chloride concentration was measured to be 147 mg/L or 0.00415 *M* Cl. The molar ratio of Cl to Hg in the carbonate wash is therefore 2.65, slightly higher, but consistent with the molar ratio of 2 for the $HgCl_2$ compound. Assuming the mercury was extracted into the solvent as $HgCl_2$ and completely stripped in the carbonate wash, the excess chloride (above the molar ratio of 2) in the wash product may be an indirect measure of the amount of mercury lost to precipitation of HgO. The moles of Hg lost to the precipitate is given by:

$$(4.15\text{E-}03\ M\,\text{Cl} \ * 1\ \text{mole Hg}/\ 2\ \text{mol Cl}) - 1.57\text{E-}03\ M\,\text{Hg}$$
$$= 5.05\text{E-}04\ M\,\text{Hg lost as HgO}$$

For a total final volume of carbonate wash product of 36.6 L:

$$5.05\text{E-}04\ M\,\text{Hg} * 200.59\ \text{g Hg / mol} * 36.6\ \text{L solution}$$
$$= 3.71\ \text{g Hg lost as HgO}$$

Consequently, a total mass of 3.71 g of Hg was estimated to be lost as precipitated HgO in the carbonate wash section over the course of the experiment. This mass corresponds to 22.1% of the mercury in the feed. Including this mass in the overall (non-normalized) material balance leads to a respectable 94.7% overall mass balance for mercury.

The rinse product sample taken at 40 hours was analyzed for all metals. Trace quantities of Al (0.9 mg/L), B (0.3 mg/L), Cr (0.03 mg/L), K (0.04 mg/L), Mo (0.7 mg/L), and Na (17.1 mg/L) were detected in that sample. The sodium detected in this sample was likely carried over with the organic from the sodium carbonate wash and stripped with the dilute nitric acid in the acid rinse section.

Contactor Stage Efficiency

It is of interest to estimate the stage efficiency obtained with the 3.3 cm contactors during the TRUEX test. This can be accomplished using the actual stagewise data in conjunction with the Generic TRUEX Model (GTM). Stage efficiency is a variable parameter in the GTM and iterations at different stage efficiencies can be performed until the actual and modeled concentrations in a given phase coincide. With this analysis it is desirable that the distribution coefficients for the element being evaluated to be fairly high (>1) to observe the impacts of variable stage efficiency in the GTM. The only element with high distribution ratios in the extraction section was europium. The europium concentrations were available in the organic phase for stages 5 through 8 in the extraction section; Eu analyses for the organic phase in the other stages in the extraction section were reported as below detection limits. Consequently, these four points were used in the modeling analysis and the results extrapolated for the remaining stages. A plot of organic Eu concentration versus stage number is given in Figure 10 for the extraction section consisting of stages 1 through 8. The data shown in Figure 10 indicate that the extraction section was operating at approximately 99% stage efficiency with regard to Eu during the TRUEX test. With higher stage efficiencies, the Eu concentration in the aqueous raffinate exiting stage 1 substantially decreases. In reality, contamination occurring in an actual process would likely impact the raffinate activity before such extremely high removal efficiencies would be achieved.

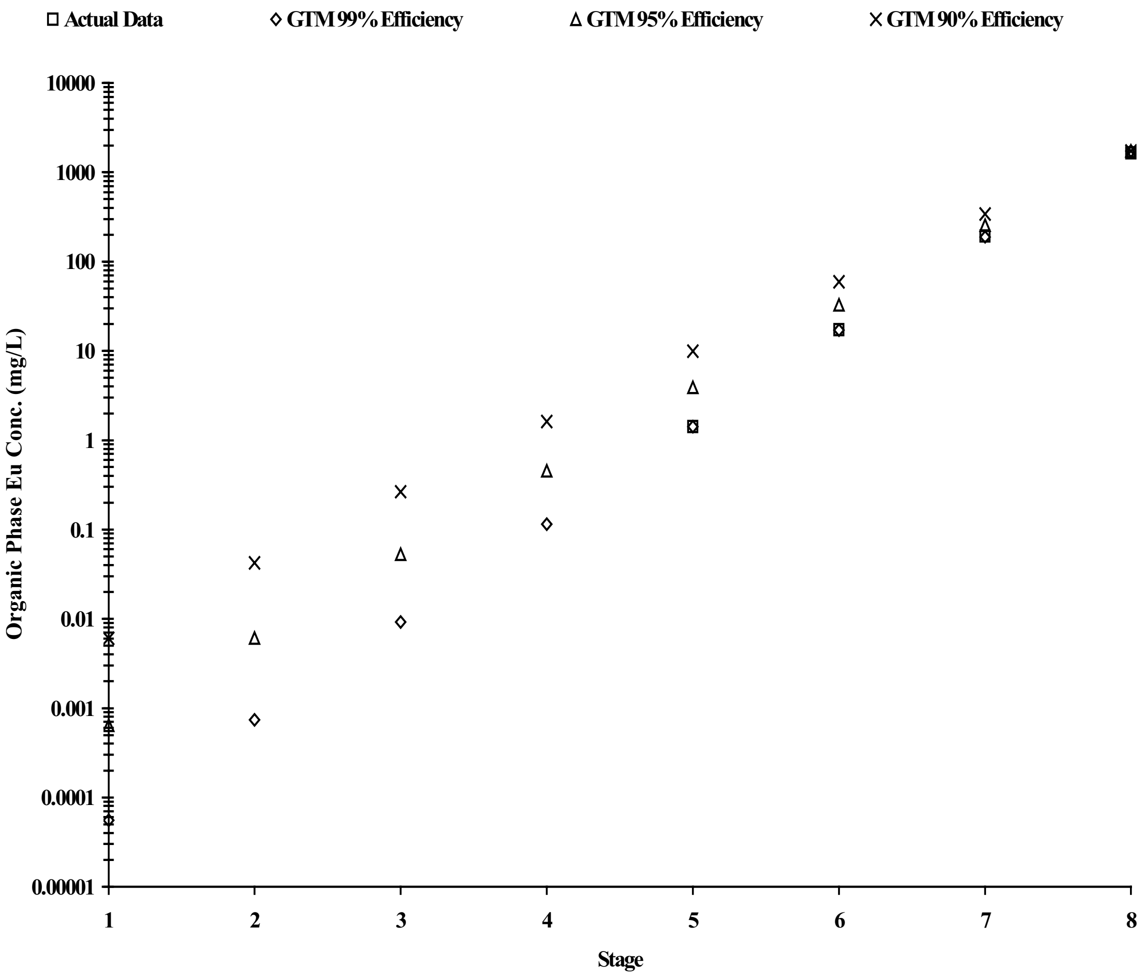

Figure 10. Predicted vs. actual Eu organic phase concentration profiles in the extraction section.

SREX Solvent Extraction

Contactor Operation

The SREX solvent extraction portion of the test was designed to remove predominately the strontium and lead from the TRUEX raffinate. The SREX test was operated a cumulative of 77.9 hours and processed 50.97 L of the TRUEX raffinate used as feed to the SREX flowsheet. Note that the SREX portion of the integrated test was performed approximately 5 weeks after completing the TRUEX test because the centrifugal contactors had to be reconfigured and hydrodynamically tested before the SREX test.

Startup and operation of the equipment proceeded as follows: The centrifugal contactor motors were started at 2700 rpm. Aqueous solution flows, with the exception of the feed (TRUEX raffinate), were established in the contactors. A startup solution consisting of 1.2 M HNO_3, 0.56 M $Al(NO_3)_3$, and 1.2 M $NaNO_3$ was used in place of the simulated tank waste while solution flows were being established in the system. The organic (SREX) solvent flow was started after aqueous flows were established. When organic was observed entering stage 23 in the acid rinse section, the feed (TRUEX raffinate) was started in place of the initial startup solution, marking time zero for the test.

During the course of the run, flooding or precipitation was never observed in any of the effluent streams. The test proceeded for the entire 78 hour duration without stopping the contactors or shutting down solution flows.

Product and feed flowrates during the run were determined by periodic measurement of the effluent streams and by calculation from feed tank depletion and product tank filling rates. The different methods of determining flowrates were used to establish a time-weighted average for each flowrate during the course of the run. The actual and target flowrates, as well as organic-to-aqueous (O/A) phase ratios are listed in Table 12. The actual flowrates shown in Table 12 are in excellent agreement with the target values and were used for all subsequent calculations.

Based on the 1500 mL initial volume of SREX solvent, a run time of 77.9 hours, and an organic flowrate of 14.6 mL/min, the organic was recycled through the centrifugal contactors a calculated 45.5 times.

Table 12. Flowrates and O/A ratios for the SREX flowsheet test.

		Flowrate (mL/min)		O/A Ratio		Total Flow
Section	Phase	Target	Actual	Target	Actual	(mL/min)
All	Org.	14.7	14.6	---	---	---
Extraction	Aq.	14.7	14.5	1.0	1.0	29.2
Scrub	Aq.	3.67	3.60	4.0	4.06	18.2
Sr Strip	Aq.	29.3	30.4	0.5	0.48	45.0
Pb Strip	Aq.	14.7	14.2	1.0	1.03	28.8
Carbonate Wash	Aq.	2.93	2.73	5.0	5.34	17.3
Acid Rinse	Aq.	7.34	6.63	2.0	2.2	21.2

Real Time Product Concentrations

The primary species of interest for evaluating flowsheet characteristics were Sr, Pb, and K. Samples of the aqueous raffinate, Pb strip, and Sr strip products were taken every half-hour for the first 3 hours to determine when steady state conditions were achieved. Beginning at 5 hours into the run, product samples of the aqueous raffinate, Sr strip, Pb strip, carbonate wash, acid rinse, and organic product were taken at 5-hour intervals. Samples were also taken of the product streams just prior to shutdown (~78 hours) and composite samples were obtained from the product tanks after concluding the experiment. Analyses of the composite samples provide an indication of "average" behavior and flowsheet performance for each of the species over the duration of the experiment. The entire array of metal analysis was requested for the final product and composite samples. Additional metals analysis (Ag, Se, Ru, Pd, Al, B, Ca, Cs, Cr, K, Mn, Mo, Na, Ni, Pb, Sr, Zr, Rh, Cl, and F) were requested on the raffinate, strip, wash, rinse, and organic product samples taken at 20, 40, and 60 hours. The entire array of metal analysis was requested for the final product and composite samples.

Aqueous Raffinate. The concentrations of Sr and K in the aqueous raffinate are shown as a function of time in Figure 11. The lead analyses in all aqueous raffinate samples were reported as <0.11 mg/L Pb. It is apparent from the data in Figure 11 that the extraction section was operating at steady state with regard to strontium and potassium within the first five hours of testing. The solid lines represent the composition of the composite aqueous raffinate sample taken at the conclusion of the experiment. There is excellent agreement between the composite and real time raffinate samples.

The analytical results for other species of interest are listed in Table 13. The analysis of the aqueous feed solution (TRUEX raffinate) to the SREX flowsheet is indicated for comparison. Note that the analytical results for the raffinate (Aq. Raff.) and composite (Aq. Raff. Comp.) samples are very consistent and typically lower than those of the feed. This is due to dilution of the feed by ~33 vol. % from the introduction of the scrub solution. The time wise analyses are very consistent for each of the species listed in Table 13, indicating that the extraction section was indeed operating at steady state with regards to each of these species. The results also indicate that Ba, Hg, Re, and perhaps Cl (poor material balance) were the other matrix components extracted from the feed by the SREX process to any significant extent.

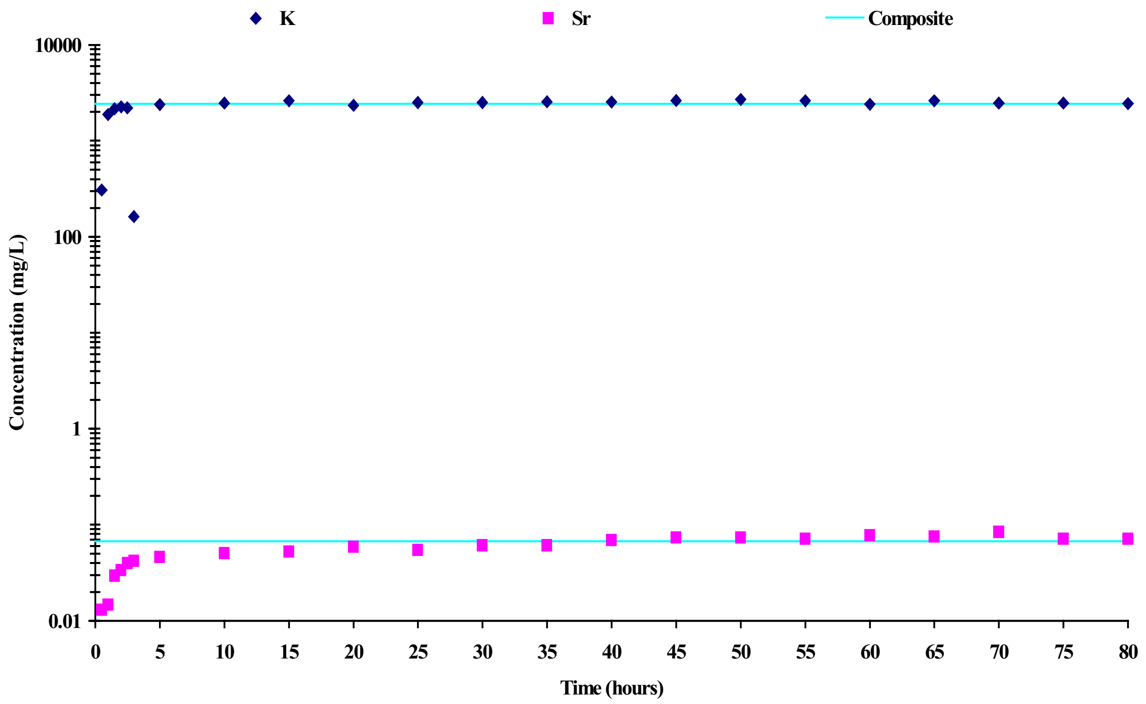

Figure 11. Composition of K and Sr in the aqueous raffinate as a function of time.

Table 13. Analytical results (in mg/L) of several metals in the SREX raffinate samples.

Sample	Al	Ag	B	Ba	Ca	Cr
SREX Feed	10200	0.065	136	10.7	1330	170
Aq. Raff. 20 hrs	7480	<0.03	92.0	2.76	857	115
Aq. Raff. 40 hrs	7410	<0.03	93.1	2.95	864	116
Aq. Raff. 60 hrs	7240	<0.03	92.1	3.10	851	115
Aq. Raff. 78 hrs	7320	<0.03	94.0	2.98	866	116
Aq. Raff. Composite	7290	<0.03	93.7	2.94	866	116
Percent Extracted [a]	<1	--- [b]	<1	64	<1	<1
	Fe	Hg	Mn	Na	Ni	Pd
TRUEX Raffinate	867	11.1	297	20000	76.1	0.166
Aq. Raff. 20 hrs	624	0.326	206	19100	49.1	<0.07
Aq. Raff. 40 hrs	632	0.389	202	20000	52.2	<0.07
Aq. Raff. 60 hrs	627	---	202	20100	50.7	<0.07
Aq. Raff. 78 hrs	640	0.460	204	19200	50.6	<0.07
Aq. Raff. Composite	631	0.425	206	19300	52.0	<0.07
Percent Extracted [a]	<1	91 [b]	<1	0 [b]	<1	--- [b]
	Re	Rh	Se	Zr	Cl	F
TRUEX Raffinate	204	0.246	1.62	59.1	875	4560
Aq. Raff. 20 hrs	4.63	0.252	1.31	40.9	689	131
Aq. Raff. 40 hrs	5.36	0.227	1.37	41.0	692	2332
Aq. Raff. 60 hrs	6.19	0.244	1.34	40.7	680	2243
Aq. Raff. 78 hrs	6.24	0.248	1.31	41.5	675	2274
Aq. Raff. Composite	5.49	0.275	1.28	41.0	673	2240
Percent Extracted [a]	96	--- [b]	--- [b]	<1	<9 [b]	<1 [b]

[a] Percentage extracted based on the normalized composite sample, refer to Appendix Table A-18.

[b] Material balance (non-normalized) was poor for this component (>±10%).

Sr Strip Product. The concentrations of K, Sr, and Pb in the Sr strip product are shown as a function of time in Figure 12. From the data in Figure 12, it is apparent that the stripping section was operating at steady state conditions, with regard to Sr and Pb, within the first five hours of the experiment. The K concentrations in the Sr strip product for the 10, 15, 25, and 35 hour samples were considerably lower than the remaining samples, indicating steady state may have not been reached for K until 40 hours. It is not understood why K would take longer to reach steady state, particularly since K reached steady state in the raffinate within 5 hours, and the Pb strip product and carbonate wash product within 5 to 15 hours (*vide infra*). More likely the discrepancy is due to analytical error and steady state was actually reached within 5 hours in the Sr strip product section. The solid lines represent the analytical results for the composite strip product sample taken at the end of the run. There is excellent agreement between the steady state and composite sample analyses.

The analytical results for the species extracted from the feed and entering the Sr strip section are listed in Table 14. Note that analytical results for the Sr strip product samples and composite sample are very consistent for all elements except Hg. The Sr strip product samples averaged 0.22 mg/L Hg and the composite was 0.01 mg/L Hg, near analytical detection limits, so it is likely that analytical error accounts for this discrepancy. These results confirm that the Sr strip section was operating at steady state with regard to these components. Of the metals listed in Table 14, virtually all of the extracted Sr, K, and Ba were completely recovered in the Sr strip section. Pb, Hg, and Cl were only partially recovered in the Sr strip with 15% of the Pb, 0.4% of the Hg, and 0.002% of the Cl exiting in the Sr strip product. The data indicate the strip section was operating very efficiently with regard to Sr, K, and Ba stripping. Note that the percentage of each component extracted from the SREX feed and recovered in the Sr strip product is based on the composite (normalized) sample analysis (refer to Table A-18 in the Appendix).

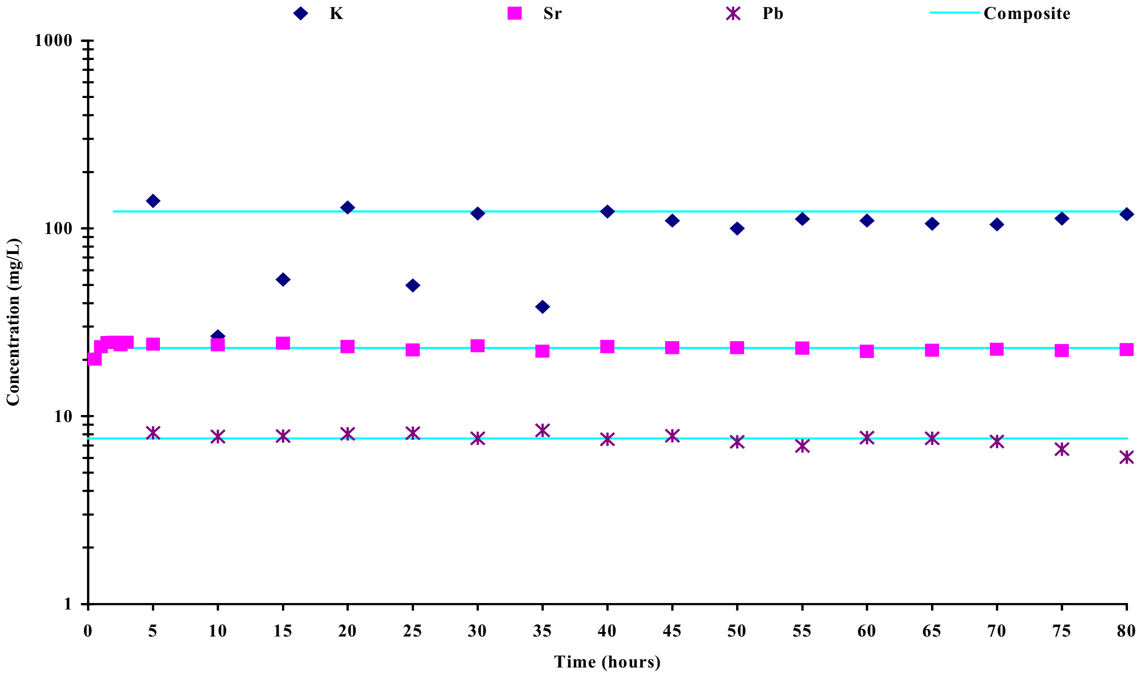

Figure 12. Composition of K, Sr, and Pb in the Sr strip product as a function of time.

Table 14. Analytical results (in mg/L) of several metals in the Sr strip product samples.

Sample	Sr	K	Pb	Ba	Hg	Cl
SREX Feed	63.9	3520	128.5	10.7	11.1	875
Sr Strip Product 20 hrs	23.5	129	8.1	2.6	0.22	<35.3
Sr Strip Product 40 hrs	23.5	123	7.5	2.6	0.23	<35.3
Sr Strip Product 60 hrs	22.1	110	7.7	2.7	0.22	<35.3
Sr Strip Product 78 hrs	22.6	119	6.0	2.4	0.21	<35.3
Sr Strip Prod Composite	23.1	123	7.6	2.5	0.01	<17.7
Percentage Extracted	99.86	9.6	99.9	64.2	90.9	<8.7
Percentage in Sr Strip [a]	99.82	9.55	15.0	64.0	0.4	0.002

[a] Percentage in strip based on analysis of the composite sample and normalized for a 100% material balance.

Pb Strip Product. The concentrations of K and Pb in the Pb strip product are shown as a function of time in Figure 13. Sr concentrations in the Pb strip product samples were below analytical detection limits. From the data in Figure 13, it is apparent that the Pb stripping section was operating at steady state conditions for Pb and K within the first five hours of the experiment. The concentration of K in the 20 hour sample was lower than the other samples by approximately 50%, likely due to analytical error since the low concentrations of K in the samples (~1 mg/L) are near analytical detection limits. The solid lines represent the analytical results for the composite strip product sample taken at the end of the run. There is excellent agreement between the steady state and composite sample analyses.

The analytical results for the species extracted from the feed and entering the Pb strip section are listed in Table 15. Note that analytical results for the Pb strip product samples and composite sample are very consistent. Of the metals listed in Table 15, the remaining Pb (15% of which was recovered in the Sr strip section) was nearly completely recovered in the Pb strip section. Strontium, K, and Ba were recovered in the Sr strip section, resulting in very little recovered in the Pb strip section. Mercury and Cl were only partially recovered in the Pb strip section. Note that the percentage of each component extracted from the SREX feed and recovered in the Pb strip product is based on the composite (normalized) sample analysis (refer to Appendix Table A-18).

Table 15. Analytical results (in mg/L) of several metals in the Pb strip product samples.

Sample	Sr	K	Pb	Ba	Hg	Cl
Feed	63.9	3520	128.5	10.7	11.1	875
Pb Strip Product 20 hrs	<0.013	0.53	91.7	<0.01	---	<35.3
Pb Strip Product 40 hrs	<0.013	1.02	98.5	<0.01	---	<35.3
Pb Strip Product 60 hrs	<0.013	1.03	92.6	0.01	---	<35.3
Pb Strip Product 78 hrs	<0.013	1.00	96.6	<0.01	3.7	<35.3
Pb Strip Prod Composite	<0.013	1.07	91.9	<0.01	3.0	<17.7
Percentage Extracted	99.86	9.6	99.9	64.2	90.9	<8.7
Percentage in Pb Strip [a]	<0.03	0.04	84.7	<0.13	63.7	<2.4

[a] Percentage in strip based on analysis of the composite sample and normalized for a 100% material balance.

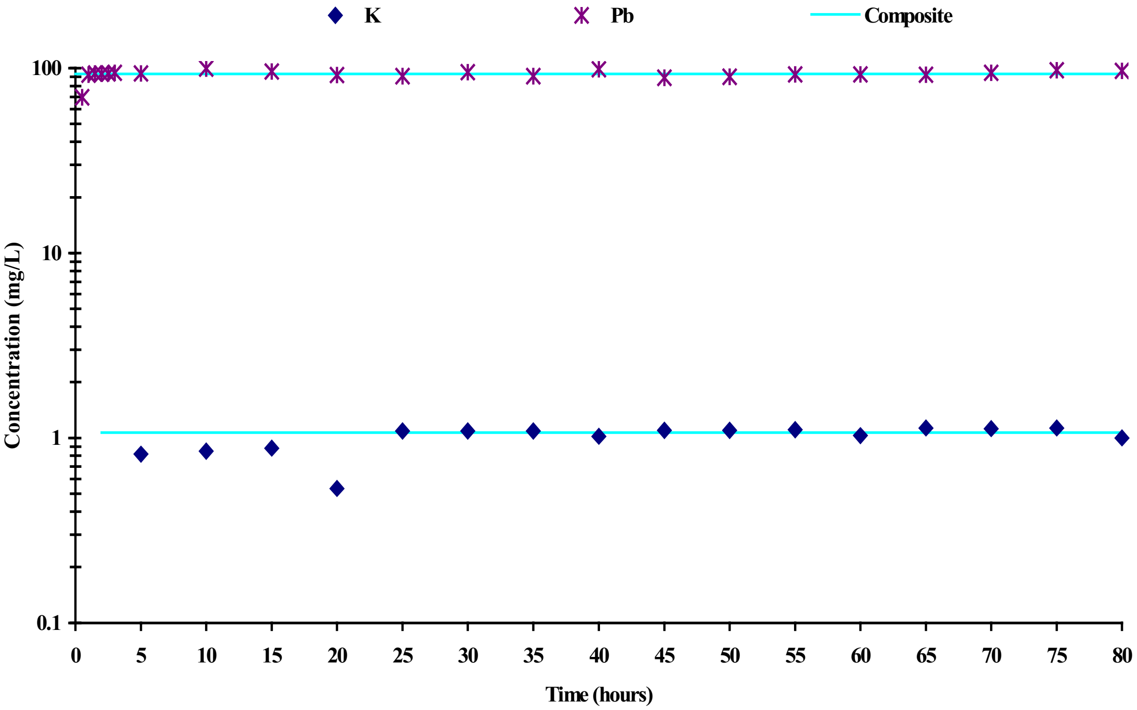

Figure 13. Composition of K and Pb in the lead strip product as a function of time.

Carbonate Wash Product. The concentrations of K and Pb in the carbonate wash product are shown as a function of time in Figure 14. Sr concentrations in the wash product samples were below the analytical detection limits. From the data in Figure 14, it is apparent that the wash section was operating at steady state conditions for Pb and K within the first five to ten hours of the experiment. There is some scatter to the Pb data due primarily to the low concentrations of Pb in the wash (<1 mg/L) which are very near the analytical detection limit for Pb (~0.1 mg/L). The solid lines represent the analytical results for the composite wash product sample taken at the end of the run. There is excellent agreement between the steady state and composite sample analyses for Pb, but the composite concentration for K is slightly higher than concentrations in the wash products. In light of the low concentrations of K in the wash product, this slight difference is not surprising.

Concentrations of Sr, K, Ba, and Pb in the wash product were very small (<<1%). Mercury was partially stripped from the SREX solvent in the carbonate wash section. Approximately 25% of the Hg in the feed solution exited with the wash product, the remaining mercury could potentially build up in the solvent to a steady state level.

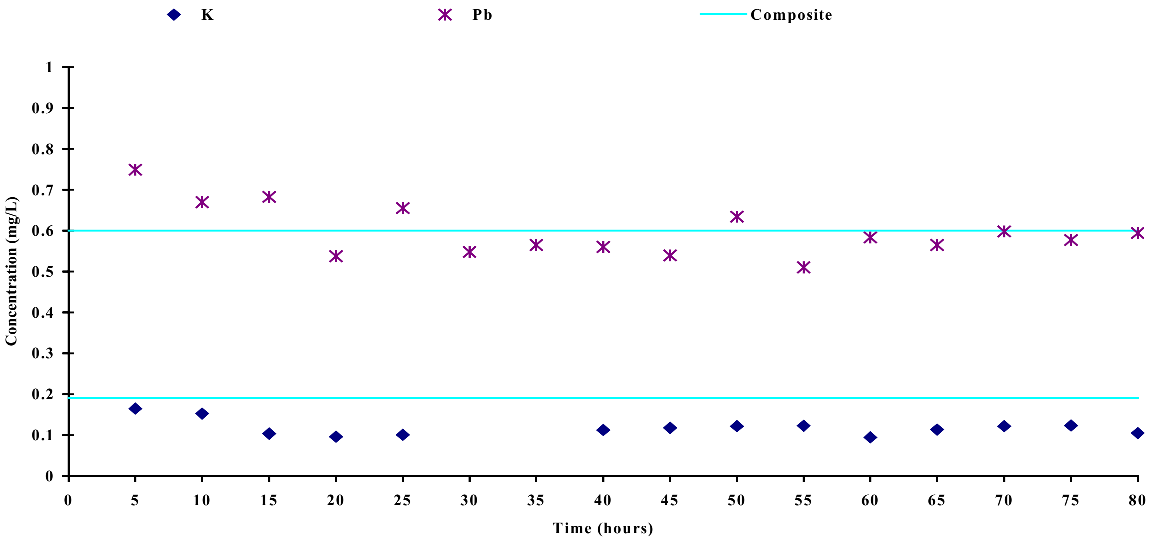

Figure 14. Composition of K and Pb in the carbonate wash product as a function of time.

Acid Rinse Product. The concentration of K in the acid rinse product is shown as a function of time in Figure 15. Sr and Pb concentrations in the rinse product were all below analytical detection limits. The concentrations of K in the rinse product are very low (<0.1 mg/L) making it difficult to evaluate steady state operation in the rinse section. The solid lines represent the analytical results for the composite rinse product sample taken at the end of the run. The concentration of K in the composite sample is slightly higher than in the rinse product samples. This discrepancy is not surprising due to the extremely low concentrations of K being measured and the associated analytical uncertainty.

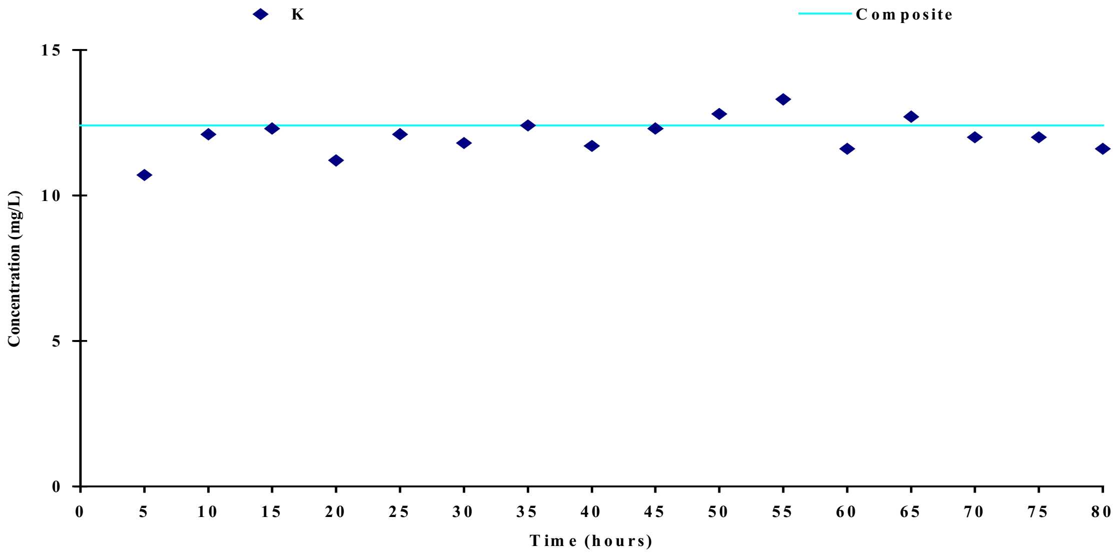

Figure 15. Composition of K in the acid rinse product as a function of time.

Organic Product. The concentrations of K in the organic product stream, as it exited from the acid rinse section, are shown as a function of time in Figure 16. Potassium was the only element consistently present above analytical detection limits. Mercury analysis was not requested on any of the time-wise samples. There is no evidence of potential build up of K based on these analytical results.

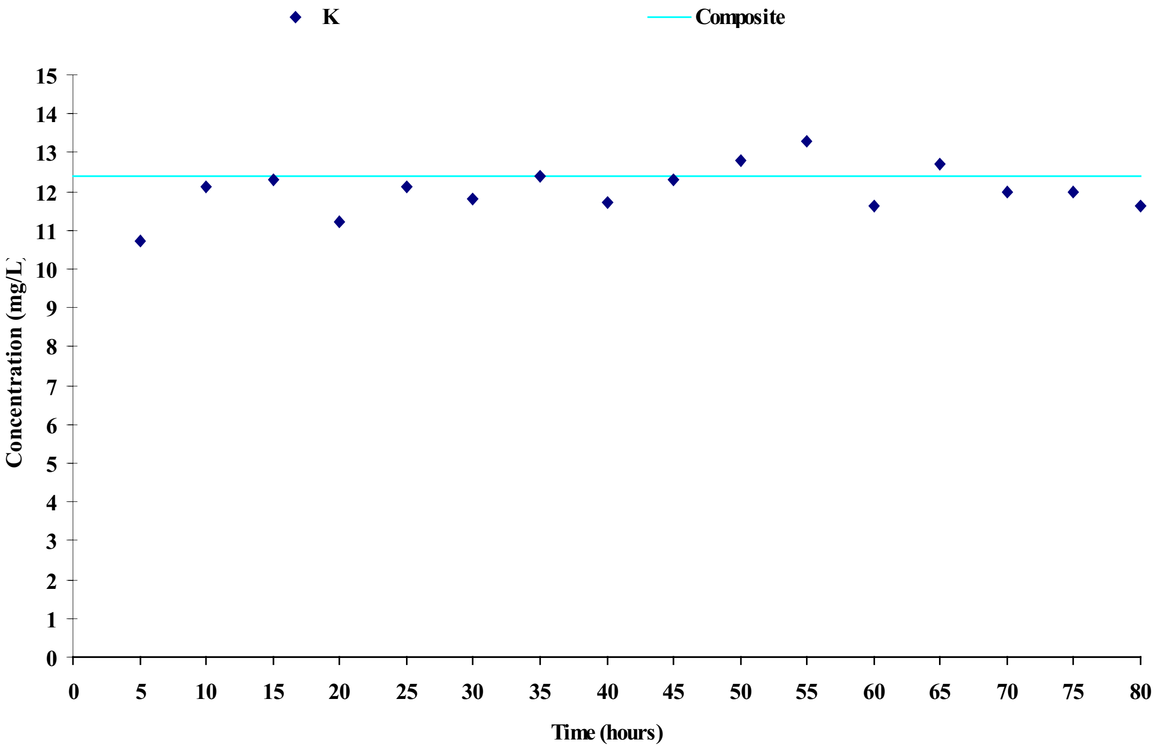

Figure 16. Concentration of K in the SREX organic product as a function of time.

Composite Product Concentrations and Stagewise Distribution Coefficients

An important piece of information from the extended flowsheet test is the composition of all final products from the SREX run. Composite samples of the product solutions were anticipated to be representative of the products that would be expected in full scale, continuous operation. Concentrations in the composite products of the primary metals of concern, e.g., Sr, K, Pb, and Hg, and the percentage of each component in the various streams, are indicated in Table 16. Average flowrates for the duration of the test, final total volumes, and the material balances for these components are also indicated. The stagewise distribution coefficients for these species were also calculated for each stage after shutdown and are presented in Table 17.

Strontium. Based on the data in Table 16, 99.86% of the strontium was removed from the feed. It is estimated that approximately 99.9% Sr removal is required to meet the NRC Class A low level waste requirement of 0.04 Ci/m^3 in the final waste form for the INTEC tank wastes. Thus the Sr removal obtained in this test is marginally acceptable to meet this requirement. Note that due to the analytical limitations for stable Sr in the simulated waste feed used in this experiment, the amount of Sr in the feed to the SREX process (63.9 mg/L) is substantially higher than anticipated in the actual tank wastes (~1.5 mg/L). Most of the stable Sr in actual wastes is “tramp” Sr from chemical additions during processing. Strontium removal is consistent with the extraction distribution coefficients of D_{Sr} = 2.3 to 3.2 measured in the extraction stages for the test (refer to Table 17). The only scrub stage distribution measured (stage 11) was D_{Sr} = 1.56, and is low enough to indicate a fraction of the extracted Sr may have scrubbed and recycled to the extraction section. The scrub section conditions could be improved by altering the $NaNO_3$ concentration in the scrub feed or the scrub feed flowrate to minimize Sr recycle to the extraction section. All of the extracted Sr, 100.8%, was recovered in the Sr strip section, indicating this section was very effective for Sr recovery. This result is consistent with the Sr distribution coefficient of D_{Sr} = 0.125 calculated on the first Sr strip stage (stage 13). Strontium distributions could not be obtained for any of the stages past 13 since the concentrations were below the analytical detection limits. The material balance for Sr, based on the composite samples, was excellent at 101%.

Table 16. Primary components and parameters of the composite product samples.

Component	Aqueous Feed	Aqueous Raffinate	Sr Strip Product	Pb Strip Product	Carb Wash	Acid Rinse	SREX Organic[a]	Material Balance[b]
Sr (mg/L)	63.9	0.067	23.1	<0.013	<0.013	<0.013	<0.126	
% per stream	---	0.14	100.8	<0.026	<0.005	<0.012	<0.26	101%
Pb (mg/L)	128.5	<0.11	7.62	91.9	0.60	<0.11	<1.07	
% per stream	---	<0.11	16.5	93.2	0.12	<0.051	<1.12	110%
K (mg/L)	3520	2430	123	1.07	0.19	0.13	12.4	
% per stream	---	92.2	9.74	0.04	0.001	0.002	0.47	101.9%
Hg (mg/L)	11.1	0.425	0.008	3.04	6.33	0.14	1.92	
% per stream	---	5.11	0.201	35.7	14.3	0.77	23.2	56.1%
Final Volume[c] (L)	---	68.1	142.1	66.5	12.8	31.0	0.42	---
Flow (mL/min)	10.9	14.5	30.4	14.2	2.73	6.63	14.6	---

[a] Organic composition not included in the material balance since solvent was recycled.

[b] Calculated as a percentage of out/in.

[c] Does not account for samples removed during the course of the experiment or the volume remaining in the stages after shutdown.

Table 17. Measured stagewise distribution coefficients from the SREX test.

Section	Stage #	Sr	Pb	K
Extraction	1	3.23	13.3	0.22
	3	2.38	10.5	0.22
	5	2.32	7.10	0.27
	7	2.26	7.72	0.27
	9	2.37	7.63	0.29
	10	2.28	99.6	0.28
Scrub	11	1.56	115	0.23
Sr Strip	13	0.125	35.3	0.093
	16	---	3.73	409
Pb Strip	17	---	0.017	8.70
	20	---	3.53	3.53
Carbonate Wash	21	---	2.73	60.3
	22	---	2.37	23.9
Acid Rinse	23	---	20.2	78.7
	26	---	8.51	104

Lead. Lead is a RCRA regulated metal, present in significant quantities (~320 mg/L) in INTEC tank wastes. The SREX process is capable of removing Pb from the tank waste for subsequent disposal. The data in Table 16 indicate >99.89% of the Pb in the SREX feed was extracted in the process. This is consistent with the Pb distribution coefficients of D_{Pb} = 7.6 to 100 measured in the extraction stages. The measured scrub distribution of D_{Pb} = 115 on stage 11 indicates lead was not scrubbed from the SREX solvent. A fraction of the extracted Pb, 16.5%, was recovered with the Sr strip product and the balance, 93.2%, was recovered with the Pb strip product. The overall material balance for Pb was marginal at 110%, indicating the fraction of Pb reporting to the Sr strip and/or Pb strip product streams may be high, and/or slight errors in the Pb feed analysis or flowrate. The SREX portion of the flowsheet was extremely effective at removing Pb from the simulated tank waste. It is anticipated that optimization of the flowrates and/or compositions for the Sr and Pb strip feed streams could result in the complete recovery of lead in the Pb strip product.

Potassium. It is known that the SREX process has a slight affinity for the extraction of potassium from the tank waste matrix. Potassium is a bulk matrix component present in INTEC tank wastes at ~7.5 g/L. It is desirable to minimize the mass of inert matrix components, including K, that report to the high activity streams, thereby minimizing the final volume of HAW. The data in Table 16 indicate 7.8% of the K in the SREX feed was removed, which is consistent with the distribution coefficients measured for the extraction section of D_K = 0.22 to 0.29. The primary function of the scrub section is to remove extracted K and recycle it back to the extraction section. Much of the K was scrubbed based on the stage 11 distribution coefficient of D_K = 0.23. The Sr strip section effectively recovered virtually all of the extracted potassium. The overall material balance for potassium was excellent at 102%. Optimization of the scrub section may be possible to reduce the amount of K reporting to the Sr strip product stream.

Mercury. One of the major concerns with the SREX process is the extraction of mercury, which currently cannot be stripped very effectively from the solvent. Note that approximately 95% of the residual Hg in the feed (TRUEX raffinate) was removed in the SREX process. Approximately 50% of the removed Hg was recovered in the Pb strip and carbonate wash products. Note that the material balance for Hg (which does not include the solvent due to recycle of that stream) was particularly poor at 56.1%. The poor material balance could be attributed to continuous mercury build up in the solvent and/or analytical error at the low mercury concentrations of the aqueous samples. Note that the organic phase mercury concentration of 1.92 mg/L could be relatively low due to poor Hg stripping by the 0.1 *M* ammonium citrate used for preparation of the organic samples for analysis. Mercury build up in the SREX solvent should not pose problems at the low concentrations remaining in the TRUEX raffinate and enters the SREX extraction section. With some removal in the solvent wash section (~50% in this test), Hg should build up to a steady state value in the SREX solvent. Recent calculations indicate that the mercury could potentially build up in the solvent to ~63% of that in the SREX feed (or ~7 mg/L Hg in the solvent during this test) at steady state [18]. Based on this estimate, the analytical data for the composite organic sample indicate that steady state Hg concentration in the solvent was likely not achieved during this 78 hour test. Additional insight regarding this phenomenon could have been gained had analytical results been obtained regarding the Hg content of the real time SREX solvent samples. Note that placement of the TRUEX process prior to the SREX process will reduce the mercury concentrations in the SREX feed to the point that Hg build up will not have deleterious effects on the SREX process efficiency.

Other Matrix Components. The fractionation of numerous other matrix components as analyzed in the composite samples is summarized in Table 18. Based on the *normalized* percentages in the raffinate samples, less than 1% of the Al, B, Ca, Cr, Fe, Mn, Ni, Zr, and F were removed in the SREX flowsheet. With the exception of Ca and F, the overall material balances were acceptable for these elements, i.e., 100±10%, lending credibility to the results. These results are consistent with previous SREX flowsheet tests, which indicated Al, B, Ca, Cr, Fe, Mn, and Ni were essentially inextractable by the SREX solvent [13]. The behavior of F in the SREX flowsheet has not previously been reported. In prior testing [13], >81.6% of the Zr has been extracted, and was recovered in the Sr strip product, using a 2.0 *M* HNO_3 scrub solution. It is postulated that the 1.0 *M* $NaNO_3$ scrub solution used in this test effectively scrubbed Zr back to the extraction section. Unfortunately, stage wise Zr distributions were not determined to confirm this hypothesis.

Based on the chemical similarity of barium and strontium, Ba is expected to extract in the SREX process. Table 18 indicates that 63.2% of the Ba in the simulated feed was removed in the SREX test and was recovered in the Sr strip product. This result is consistent with those in previous flowsheet tests where 63.6% of the Ba in actual tank waste was removed and recovered in the Sr strip product [13]. Due to the similarity of Ba and Sr, it is unlikely that a substantially better separation between the two could be achieved through flowsheet optimization.

The results in Table 18 indicate that approximately 3% of the Mo in the simulated feed was removed in the SREX process and recovered in the Sr strip product. Analyses for Mo in the SREX process have previously not been performed for INTEC tank wastes. This result will be evaluated future experiments.

Rhenium was added to the simulated feed as perrhenate to function as a surrogate for technetium (as pertechnetate). Rhenium was effectively extracted from the feed, with 96.4% of the Re in the SREX feed being removed. Much of the removed Re, 92.4% of the total, was recovered in the Sr strip product and 3.4% of the total Re reported to the Pb strip product. These results will be evaluated for Tc in actual tank wastes during future tests.

Table 18. Percentage of other matrix components in the composite products.

Stream	Al	B	Ba	Ca	Cr	Fe	Mn
Aqueous Raffinate	95.55 (99.995) [a]	91.92 (99.72)	36.50 (35.79)	87.08 (99.95)	90.58 (99.96)	97.18 (99.96)	92.74 (99.98)
Sr Strip Product	<0.002 (<0.002)	<0.15 (<0.16)	65.23 (63.96)	<0.01 (<0.02)	<0.01 (<0.02)	<0.01 (<0.01)	<0.01 (<0.01)
Pb Strip Product	<0.001 (<0.0010	<0.07 (<0.07)	<0.13 (<0.13)	<0.01 (<0.01)	0.013 (0.014)	0.013 0.013	<0.01 (<0.01)
Carbonate Wash	<0.001 (<0.001)	<0.01 (<0.01)	0.064 (0.063)	0.021 (0.024)	<0.001 (<0.001)	0.004 (0.004)	<0.001 (<0.001)
Acid Rinse	0.001 (0.001)	<0.03 (<0.03)	<0.06 (<0.06)	<0.003 (<0.004)	0.008 (0.008)	0.013 (0.014)	0.003 (0.003)
Organic Product	<0.01 (<0.011)	<0.7 (<0.76)	<1.31 (<1.29)	<0.07 (<0.08)	0.066 (0.073)	0.049 (0.050)	<0.06 (<0.06)
Mass Balance [b]	<95.56	<92.2	<102.0	<87.13	<90.62	<97.22	<92.76

Stream	Mo	Na	Ni	Re	Zr	F	Cl
Aqueous Raffinate	97.73 (95.56) [a]	84.54 (92.0)	91.27 (99.78)	3.59 (3.58)	92.69 (99.16)	65.57 (99.51)	102.7 (91.33)
Sr Strip Product	3.10 (3.03)	0.467 (0.508)	<0.11 (<0.12)	92.37 (92.02)	<0.13 (<0.14)	<0.18 (<0.27)	<5.63 (<5.01)
Pb Strip Product	<0.87 (<0.85)	0.001 (0.001)	<0.05 (<0.06)	3.38 (3.37)	<0.06 (<0.06)	<0.08 (<0.12)	<2.63 (<2.34)
Carbonate Wash	<0.17 (<0.16)	6.86 (7.47)	<0.01 (<0.01)	0.58 (0.58)	0.567 (0.607)	<0.03 (<0.04)	<0.25 (<0.23)
Acid Rinse	<0.41 (<0.40)	0.024 (0.026)	0.037 (0.040)	0.45 (0.45)	<0.03 (<0.03)	<0.04 (<0.06)	<1.23 (<1.09)
Organic Product	<8.94 (<8.74)	0.017 (0.019)	<0.52 (<0.57)	0.18 (0.18)	<0.62 (<0.66)	<0.84 (<1.3)	<27.1 (<24.1)
Mass Balance [b]	<102.3	91.89	<91.47	100.4	<99.47	<65.9	<112.4

[a] Normalized percentages for a 100% material balance.

[b] Material balance based on sample analysis, calculated as out/in*100%. Organic product was not included in the material balance since the organic was recycled. Normalized organic percentages are not reported.

Contactor Stage Efficiency

It is of interest to estimate the stage efficiency achieved in the 3.3 cm contactors for the SREX test. This can be accomplished using the actual stage-wise data in conjunction with the Generic TRUEX Model (GTM). The stage efficiency is variable parameter in the GTM and iterations at different efficiencies are performed until the actual and modeled aqueous phase concentrations coincide. A plot of aqueous Sr concentration versus stage number is shown in Figure 17 for the extraction section consisting of stages 1 through 10. The data shown in Figure 17 indicate that the extraction section was operating at 83% stage efficiency during the SREX test. The low stage efficiency could be attributed to slow chemical kinetics; however, such an effect has not been observed experimentally at contact times of 10 seconds or less. The low efficiency is likely due to limitations in terms of contactor design, where mixing and/or residence times are too short, effectively limiting mass transfer. In an actual, full scale process, the contactor design would be optimized for the system and the anticipated stage efficiency would be in the range of 90% to 95%. It is apparent from Figure 17 that with a higher stage efficiency, the Sr concentration in the aqueous raffinate exiting stage 1 could decrease by as much as an order of magnitude. Operating at 100% stage efficiency, the predicted Sr removal would increase from 99.71% to 99.99%.

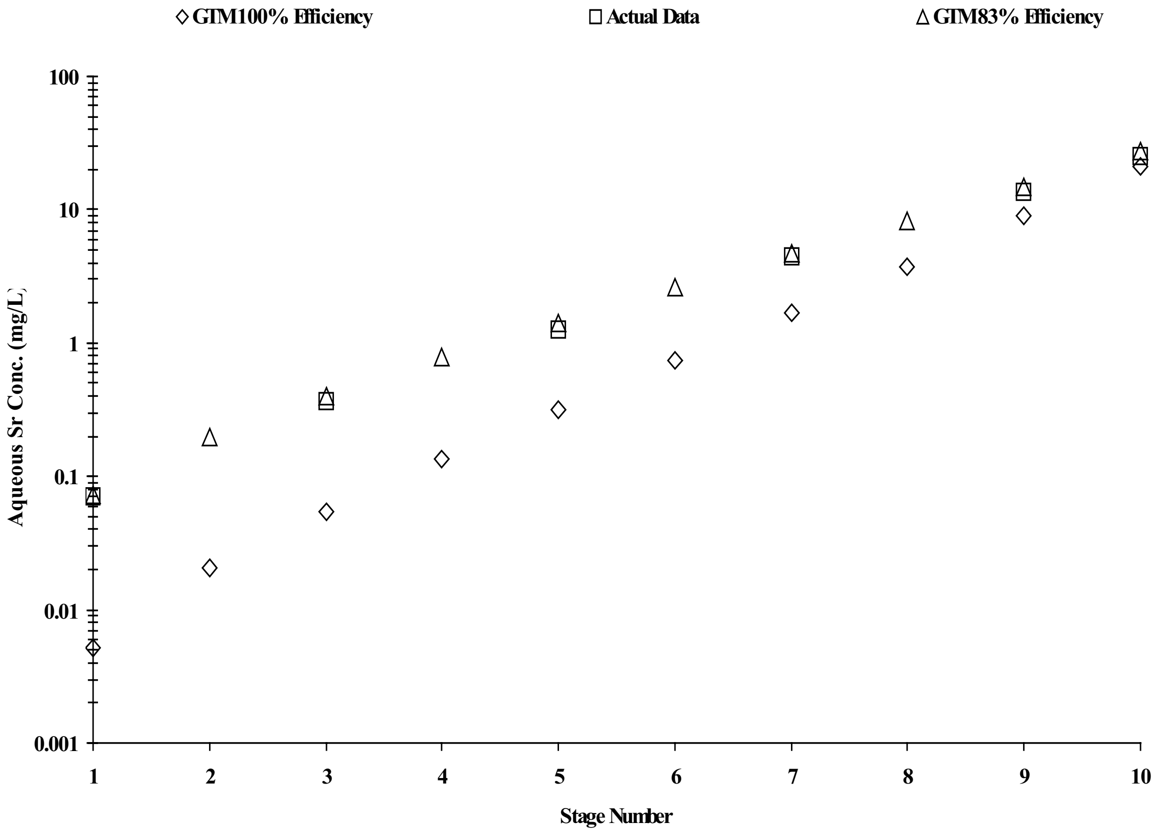

Figure 17. Predicted versus actual Sr concentration profiles in the extraction section.

Compositions and Volumes of the Liquid HAW and LAW Streams

An important consideration from the integrated test is an evaluation of the relative volumes and compositions of the liquid HAW and LAW streams generated from the three separations unit operations. The disposition of the individual streams as a portion of the total liquid HAW or LAW streams was previously indicated in Figure 3 (*vide supra*), and is also indicated in Table 19 with the volumes generated during the integrated test. Table 19 reflects the current anticipated disposal path for the individual streams, and is subject to change based on future analysis. The Hg laden carbonate wash from the TRUEX process and the Pb containing SREX Pb strip streams may require further treatment to meet RCRA disposal standards, pending determination of the necessary requirements. As an example, samples of the TRUEX carbonate wash stream will be used in future sulfide precipitation studies to determine the potential of such a technology for Hg treatment and subsequent disposal. For the purposes of this analysis, these streams are added back into the LAW liquid fraction without subsequent treatment. The disposition of any spent organic solvents from the TRUEX and SREX processes are not indicated in Table 19. It is anticipated that the spent solvents would be disposed of as LAW, and would likely be treated separate from the aqueous phases via incineration or other appropriate means. The volume of spent solvents is insignificant, relative to aqueous volumes, due to recycle and reuse of the organic phases in the solvent extraction operations.

The volumes listed in Table 19 are for the liquid streams and do not reflect any down stream processing, such as denitration, evaporation, or treatments such as Hg removal from TRUEX carbonate wash or Pb removal from the SREX Pb strip product. The liquid volumes do not reflect volumes of the final waste forms, which are dependent on the selected downstream processing options and waste loading in the final waste forms. Much of the volume of the liquid HAW and LAW fractions is associated with water and nitric acid, both of which can be recovered and/or recycled in the process via evaporation. Denitration and solidification prior to final waste form production (glass or grout) can dramatically reduce the liquid waste volumes. Consequently, the liquid volumes of the HAW and LAW streams effect process size and economics, but only the compositions of the liquid streams (and waste loading) effect final HAW and LAW volumes to be dispositioned.

Note that the quantity of cesium laden AMP-PAN (solid IX sorbent) can be considered conservative since excess sorbent was utilized in the polishing column to insure complete Cs removal. The volume of spent sorbent indicated reflects direct disposal, without provisions to dissolve the AMP with caustic and discard PAN as LAW. The contribution of the spent resin is a minute fraction of the liquid HAW stream at ~0.2 % by volume. The bulk of the liquid HAW volume is associated with the SREX Sr strip product, ~ 81 volume %, indicating that adjustments in this part of the flowsheet may be practical should the liquid HAW volume become an issue with regard to downstream process economics, such as denitration/evaporation.

The overall compositions and volumes of the HAW and LAW liquid fractions are indicated in Table 20. Note that the compositions of these liquid streams were estimated (based on volumes and analytical results of the composite product samples), since the respective streams were never physically combined and sampled for analysis at the conclusion of the test. Compositions of the individual streams (based on analysis of the composite samples) are summarized in Table A-20 of the Appendix. The HAW fraction is comprised of the radionuclides (or their surrogates) and low concentrations of several bulk matrix components (K, Mo, Pb, Zr, and Ba). As anticipated, the majority of the bulk matrix components, such as Al, Na, Ca, Fe, Mn, Ni, and Cr remain in the

Table 19. Disposition and volumes of the individual liquid products of the integrated test.

HAW Fractions			LAW Fractions		
Stream	Volume (L)	% of Total	Stream	Volume (L)	% of Total
Solid IX Sorbent	0.34	0.19	SREX Raffinate	68.1	31.0
			TRUEX Wash	37.0	16.9
TRUEX Strip	32.7	18.7	TRUEX Rinse	4.2	1.9
			SREX Pb Strip	66.5	30.3
SREX Sr Strip	142.1	81.1	SREX Wash	12.8	5.8
			SREX Rinse	31.0	14.1
Total	175.1	100	Total	219.6	100

LAW fraction. Note that the components listed in Table 20 reflect only those constituents present in the tank waste. Major components that were added during the process, H^+, NO_3^-, and P_2O_5 (added as HEDPA and assumed decomposed) were not analyzed in many of the product streams. Consequently, the concentrations of these components, as indicated in Table 20, were estimated based on input concentrations and dilution.

Using the current baseline assumptions, the limiting factor in vitrification of the HAW stream is a maximum of 2.5 wt. % phosphate, as P_2O_5, in the final waste form. Phosphate is added (as HEDPA) in the TRUEX portion of the separation process as a stripping reagent. For the composition listed in Table 20, an estimated 0.195 kg of glass is produced per L of SBW treated in the process. By comparison, direct vitrification of the tank waste would produce an estimated 0.32 kg of HAW glass per L of SBW vitrified. Promising development activities under continued study include the reduction or complete elimination of phosphate from the TRUEX flowsheet. An order of magnitude decrease in the HEDPA concentration of the TRUEX strip solution (from 0.04 *M* to 0.004 *M* HEDPA) would result in 0.09 kg of glass produced per L of SBW treated. Under conditions of phosphate elimination, HEDPA would be replaced by an innocuous compound (such as DTPA or oxalic acid) that would be broken down or volatilized under vitrification temperatures and liberated with the melter off-gas, thereby adding little to the mass of glass produced. As an analogy, nitrates are broken down or volatilized in the melter and liberated to the off-gas system. Based on the HAW composition in Table 20, without phosphate, it is estimated that 0.02 kg of HAW glass would be produced per L of SBW processed. The dramatic reduction in glass volumes associated with phosphate reduction/elimination emphasizes the necessity for continued efforts in this area of TRUEX flowsheet development.

The Hg and Pb were successfully removed from the simulated waste and was indicated these components were added back into the liquid LAW fraction. Future efforts regarding the treatment of the components (in their respective stream) will be required once the final disposition route and waste form requirements is defined for these RCRA metals.

The current baseline assumptions for grouting the LAW fraction include solidification of the solids via evaporation and denitration. Based on the composition listed in Table 20, an estimated 1.2 L of grout would be produced per L of SBW treated. As mentioned previously in the introduction (*vide supra*), the grout produced would be a “performance grout”, meeting NRC Class A requirements for near-surface disposal in an appropriate land fill. Future development efforts for the separation flowsheet will focus on the reduction of the LAW volume (such as minimizing the SREX Pb strip volume) to minimize the volume of LAW that must be evaporated and denitrated.

Table 20. Overall HAW and LAW liquid compositions and volumes from the integrated test.

Component	Simulated Waste (M)	Total HAW (M)	Total LAW (M)	Normalized % In HAW	Normalized % in LAW
Al	0.54	<2.88E-06	8.39E-02	<0.003	>99.997
B	1.80E-02	<6.60E-06	2.69E-03	<0.2	>99.81
Ca	4.48E-02	<2.14E-06	6.71E-03	<0.03	>99.97
Cl	4.09E-02	<4.99E-04	6.83E-03	<5.5	>94.51
Cs	9.78E-04	7.59E-08	4.79E-07	99.7	0.27
Cr	4.76E-03	<2.37E-07	6.90E-04	<0.03	>99.97
F	0.26	<2.33E-04	3.67E-02	<0.5	>99.50
Fe	2.30E-02	<9.89E-06	3.51E-03	<0.2	>99.78
Hg	1.97E-03	1.08E-06	2.71E-04	0.3	99.68
K	0.12	2.56E-03	1.93E-02	9.5	90.45
Mn	7.76E-03	<2.29E-07	1.17E-03	<0.02	>99.98
Mo	1.25E-03	2.72E-04	9.94E-06	95.6	4.4
Na	1.24	1.81E-03	0.37	0.4	99.61
Ni	1.83E-03	<5.41E-07	2.76E-04	<0.2	>99.84
Pb	9.25E-04	3.05E-05	1.35E-04	15.3	84.74
Sr	9.91E-04	2.14E-04	<3.76E-07	99.8	<0.2
Zr	1.43E-03	<8.37E-05	1.41E-04	32.1	67.9
Eu	7.02E-03	<1.70E-03	4.95E-06	99.6	0.4
Re	3.58E-03	5.86E-04	1.44E-04	76.4	23.6
Ba	1.09E-04	<1.49E-05	6.71E-06	63.9	36.1
Se	2.87E-05	<1.65E-06	<6.15E-06	<20.7	>79.3
Rh	1.00E-06	<1.02E-06	1.52E-06	<49.5	>50.5
H^+ (a)	1.55	0.17	0.49	---	---
P_2O_5 (a,b)	---	0.015	---	---	---
NO_3 (a)	4.8	0.17	1.14	---	---
Volume (L)	40.1	174.8	219.6	---	---

[a] Calculated, no analytical data available.

[b] HEDPA used as TRUEX strip, assumed decomposed to P_2O_5.

Using the above assumptions it is possible to estimate the volumes of HAW glass and LAW grout produced from the full separation option to treat the existing ~5E+06 L (5,000 m^3) of tank waste. Using a glass density of 2600 kg/m^3, 375 m^3 of HAW glass would be produced, as compared to 615 m^3 of HAW glass from direct vitrification of this liquid waste inventory. An order of magnitude reduction in the HEDPA concentration used in the TRUEX strip solution (from 0.04 *M* to 0.004 *M*) would reduce the volume of HAW glass to 170 m^3; elimination of phosphate from the TRUEX strip would reduce the volume of HAW glass to 38 m^3. These substantial reductions in HAW glass volume indicate the dramatic effect phosphate has on the final HAW volume. In is imperative that future optimization efforts are directed at reduction or elimination of phosphate in the TRUEX strip solution. For the case of LAW grout volume originating from the full separation treatment of this amount of tank waste, 1,135 m^3 of grout would be produced based a grout density of 1,630 kg/m^3.

SUMMARY

Three unit operations for the removal of fission products, actinides, and RCRA metals (mercury and lead) were successfully integrated and tested for extended run times with simulated INEEL acidic tank waste. The integrated unit operations performed effectively without deleterious effects to the individual unit operations from coupling the systems together. Furthermore, the accumulation of components was not observed in any of the unit operations.

Approximately 45 L of simulated INTEC tank waste were processed through three ion exchange columns packed with a composite (AMP-PAN) sorbent for cesium removal. The ion exchange system was operated continuously for ~34 hours at 22 bed volumes per hour. The Cs removal was >99.83% in the ion exchange system with little interference from other species. The first two columns were each sized at a bed volume of 60 cm^3 and were both operated to 100% breakthrough. The experimental breakthrough data were in excellent agreement with modeling predictions based on data obtained with much smaller (1.5 cm^3) columns, lending confidence to column scale-up predictions. The dynamic capacities calculated for the two 60 cm^3 columns (operated to 100% breakthrough) were in excellent agreement with the equilibrium capacity, indicating virtually all of the Cs removal capacity can be realized on the sorbent.

The effluent from the ion exchange (IX) system was processed through a TRUEX solvent extraction flowsheet to remove europium (americium surrogate), mercury and rhenium (technetium surrogate) from the simulated waste. The TRUEX test was operated a cumulative of 71.3 hours and processed ~41 L of the IX effluent using 1.5 L of TRUEX solvent with constant solvent recycle. The TRUEX solvent was recycled through the flowsheet an estimated 17.2 times without detectable degradation of process performance from acid hydrolysis, component build-up, or changes in solvent composition. Greater than 99.999% of the Eu, 96.3% of the Hg, and 56% of the Re were removed from the simulated feed, these components were effectively recovered in the strip and wash streams. Although the composition of the HEDPA strip solution used in this test was very effective at recovering these elements, its use has a major impact on the final HAW glass volume. The HEDPA concentration must be substantially reduced (by a factor of 4 to 10), or it must be completely eliminated in future optimization efforts to reduce the final HAW glass volume. It is estimated that >22.1% of the Hg was lost as an HgO precipitate in the carbonate wash section. The HgO precipitation did not result in operational problems during the course of the experiment. Precipitate formation in the contactors is unacceptable and future efforts must be directed at defining conditions to prevent the mercury precipitation.

The raffinate from the TRUEX test was stored and subsequently processed several weeks later through a SREX solvent extraction flowsheet to remove strontium and lead from the simulated waste. Approximately 51 L of TRUEX raffinate was processed through the system during 77.9 hours of continuous operation with 1.5 L of SREX solvent and continuous solvent recycle. The SREX solvent was recycled through the system an estimated 45.5 times without measurable build-up of matrix components in the solvent. Approximately 99.9% of the Sr, >99.89% of the Pb, and >96.4% of the Re were removed from the aqueous feed to the SREX flowsheet and recovered in the strip and wash sections. Operational problems such as flooding and precipitation were not encountered during the SREX test.

Approximately 40.1 L of simulated tank waste were processed during the test, resulting in 175 L of HAW and 219.6 L of LAW. The HAW fraction would be evaporated, dried and subsequently vitrified for final disposal. Based on current baseline assumptions, including a maximum phosphate loading of 2.5 wt. % in the HAW glass, the flowsheet tested would produce

0.195 kg of glass per L of tank waste processed. The LAW fraction would be solidified (via evaporation and denitration) and subsequently grouted. Based on the current baseline assumptions for grouting the LAW stream, 0.37 kg of grout would be produced per L of tank waste treated. Under these assumptions, treating the current inventory of ~5E+6 L (5,000 m^3) of tank waste would result in 375 m^3 of HAW glass and 1,135 m^3 of LAW Class A performance grout under the flowsheet conditions tested. The HAW glass volume could be markedly reduced if the HEDPA used as the strip reagent in the TRUEX flowsheet were reduced or eliminated.

RECOMMENDATIONS FOR FUTURE WORK

Any simulated tank waste used in future tests should be representative of that anticipated to exist at the time tank waste would be processed. The composition of the simulated tank waste used in this study was based on the average composition of the current inventory of waste stored in the tank farm. Projected tank farm management scenarios include the use of the High Level Liquid Waste Evaporator to concentrate and combine the present waste into fewer tanks and smaller volume. This scenario will continue for the next several years and result in ~4 million liters of liquid waste with slightly higher metals and acid concentration (<10% relative to the average composition indicated in Table 3). Future testing with the full separation flowsheet should be based on the compositions predicted via this waste management scenario. The anticipated concentration changes to the tank waste will have negligible impact on performance of the separation portion of the flowsheet; however, this postulate requires validation.

It is imperative that future work on the full separation flowsheet includes integration of the HAW and LAW immobilization aspects. At a minimum this would include crucible testing with the HAW fraction and evaporation/drying and grout formulation with the LAW fraction to define product consistency and leachability for both final products.

Continued efforts on the IX portion of the flowsheet should include further scale-up of the columns. Efforts should also be directed at caustic dissolution of the Cs laden AMP from the PAN substrate to determine how the caustic fraction would be handled (dried, etc.) and sent to the HAW glass, and if the PAN fraction could be washed and disposed of in the LAW grout. The causes for the perceived bias in the Cs analytical results should be identified and additional information collected regarding potential interferences, such as Ag. Additional work is also recommended to define stability of the AMP-PAN sorbent, not only with regard to radiolysis, but with regard to mechanical stability as well. Solids drying and storage of the spent sorbent should also be investigated.

Regarding future TRUEX development efforts, the two major considerations should revolve around reduction or elimination of phosphate (currently from HEDPA in the strip solution) and mercury precipitation in the carbonate wash section. Work in both of these areas is currently being conducted. Limited evidence is available indicating the phosphate concentration could be reduced by the use of alternative phosphorus containing complexants (namely VDPA) in the TRUEX strip. Alternatively, the HEDPA concentration could potentially be reduced if the scrub solution was altered to remove Zr (using F) from the organic phase exiting the scrub section. If the additional F were problematic in the HAW fraction, an additional scrub with Al could be used to complex F and recycle it to the LAW fraction. There is also the potential to replace HEDPA by compounds such as DTPA, EDTA, or oxalate without degrading performance of the strip section. Mercury precipitation will likely be eliminated via optimization of the flow rates and O/A phase ratio in the carbonate wash section of the flowsheet. Additional information regarding solvent recycle, process scale-up, and extended operating times should also be pursued.

Current (and continued) development efforts for the SREX process include definitive evaluation of mercury behavior in the SREX process solvent. Such work should be geared toward developing suitable stripping methods for Hg and evaluating the effects of Hg on flow sheet performance. Optimization work will also be required to reduce or eliminate the citrate in the Pb strip feed, pending a determination of the RCRA mandated requirements. As with TRUEX, additional information regarding solvent recycle, process scale-up, and extended operating times should also be pursued.

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APPENDIX:

ANALYTICAL DATA

Table A- 1. Cesium IX breakthrough data.

Flowrate = 22 mL/min Bed Volume = 60 mL Feed Cs Concentration = 130 mg/L

Sample*	Cummulative Time (Minutes)	Cummulative Volume (mL)	Cummulative Bed Volumes	Effluent Cs Conc. Column A (mg/L)	Effluent Cs Conc. Column B (mg/L)	Fractional Breakthrough Column A (C/C_o)	Fractional Breakthrough Column B (C/C_o)
IXB-1	66	1452	24.2		0.294		0.002
IXA-1	72	1584	26.4	0.161		0.001	
IXA-2	140	3080	51.3	0.159		0.001	
IXB-2	144	3168	52.8		0.293		0.002
IXA-3	200	4400	73.3	0.155		0.001	
IXB-3	204	4488	74.8		0.286		0.002
IXA-4	260	5720	95.3	0.15		0.001	
IXB-4	262	5764	96.1		0.276		0.002
IXA-5	320	7040	117.3	0.152		0.001	
IXB-5	322	7084	118.1		0.274		0.002
IXA-6	380	8360	139.3	0.159		0.001	
IXB-6	382	8404	140.1		0.27		0.002
IXA-7	440	9680	161.3	0.159		0.001	
IXB-7	442	9724	162.1		0.262		0.002
IXA-8	500	11000	183.3	0.392		0.003	
IXB-8	502	11044	184.1		0.262		0.002
IXA-9	560	12320	205.3	1.07		0.008	
IXB-9	562	12364	206.1		0.198		0.002
IXA-10	620	13640	227.3	7.08		0.054	
IXB-10	622	13684	228.1		0.2		0.002
IXA-11	680	14960	249.3	20.6		0.158	
IXB-11	682	15004	250.1		0.186		0.001
IXA-12	740	16280	271.3	53.8		0.414	
IXB-12	743	16346	272.4		0.268		0.002
IXA-13	800	17600	293.3	96.4		0.742	
IXB-13	802	17644	294.1		0.266		0.002
IXA-14	860	18920	315.3	117		0.900	
IXB-14	862	18964	316.1		0.267		0.002
IXA-15	920	20240	337.3	123		0.946	
IXB-15	922	20284	338.1		0.261		0.002
IXA-16	1099	24178	403.0	128		0.985	
IXB-16	1101	24222	403.7		0.297		0.002
IXA-17	1277	28094	468.2	133		1.023	
IXB-17	1279	28138	469.0		6.58		0.051
IXA-18	1459	32098	535.0	122		0.938	
IXB-18	1461	32142	535.7		91.7		0.705
IXA-19	1759	38698	645.0	132		1.015	
IXB-19	1760	38720	645.3		137		1.054
IXA-20	2000	44000	733.3	128		0.985	
IXB-20	2000	44000	733.3		133		1.023

* IX column A or B. # designates the sample number (order of samples taken).

Table A- 2. Concentrations of other components in IX samples.

	Cs	Hg	Na	K	Al	Eu	Fe	Pb	Mo	Re	Zr
Sample						(mg/L)					
IX Feed	130	395	2.84E+04	4.60E+03	1.41E+04	1.07E+03	1.28E+03	1.92E+02	1.19E+02	6.67E+02	1.31E+02
IXA-2	0.159	370	2.96E+04	4.88E+03	1.38E+04	1.07E+03	1.22E+03	1.88E+02	1.16E+02	6.52E+02	1.15E+02
IXA-7	0.159	295	2.95E+04	4.98E+03	1.37E+04	1.05E+03	1.24E+03	1.85E+02	1.16E+02	6.39E+02	1.11E+02
IXA-12	53.8	369	2.93E+04	5.08E+03	1.35E+04	1.05E+03	1.18E+03	1.85E+02	1.13E+03	6.41E+02	1.11E+02
IX Effluent (TRUEX Feed)	0.219	422	2.97E+04	5.20E+03	1.46E+04	1.07E+03	1.30E+03	1.96E+02	1.21E+02	6.59E+02	1.17E+02

	Cs	Ag	Re	Pd	Ba	Cr	Eu	F	Ni	Zr	Pb	Sr	Rh
Sample							(mg/L)						
IX Feed	130	4.45	667.4	<0.07	15.00	247.3	1067	4930	107.6	130.7	191.7	86.85	<0.10
TRUEX Feed	0.219	0.048	658.5	<0.07	14.69	238.4	1066	4490	103.8	116.6	195.5	93.03	0.1995
% Removed	99.83	98.91	1.34	---	2.13	3.618	0.091	8.93	3.47	10.83	---	---	---

	Fe	Hg	Cl	Se	Ru	Al	B	Ca	K	Mn	Mo	Na
Sample						(mg/L)						
IX Feed	1282	395	1450	2.27	1.97	1.41E+04	194.7	1797	4600	426.2	119.5	28400
TRUEX Feed	1297	422	1539	2.31	2.06	1.46E+04	198.0	1874	5200	427.0	121.1	29700
% Removed	---	---	---	---	---	---	---	---	---	---	---	---

Table A- 3. Approach to steady data for the raffinate and strip product samples in the TRUEX flowsheet.

	Eu	Fe	Re	Hg		Eu	Fe	Re
Sample*		(mg/L)			Sample*		(mg/L)	
TRUEX Feed	1065.6	1297	658.5	422				
Aq. Raff. 0.5	<0.04	291.3	46.18	0.266	St. Prod. 0.5	418.7	1.15	41.20
Aq. Raff. 1.0	<0.04	610.3	128.6	0.574	St. Prod. 1.0	1150	1.64	161.3
Aq. Raff. 1.5	<0.04	733.5	155.3	0.853	St. Prod. 1.5	1279	2.04	209.4
Aq. Raff. 2.0	<0.04	801.6	169.8	1.72	St. Prod. 2.0	1278	2.13	239.2
Aq. Raff. 2.5	<0.04	824.9	178.6	2.91	St. Prod. 2.5	1302	2.24	267.1
Aq. Raff. 3.0	<0.04	841.7	185.7	4.52	St. Prod. 3.0	1351	2.23	287.1

* Aq. Raff. = Aqueous Raffinate, St. Prod. = Strip Product, #.# following sample indicates time sample was taken relative to time zero.

Table A- 4. Analytical data for the aqueous raffinate samples from the TRUEX flowsheet.

	Eu	Fe	Re	Hg
Sample*		(mg/L)		
TRUEX Feed	1065.6	1296.8	658.5	422
Aq. Raff. 5	<0.04	857.9	193.5	7.496
Aq. Raff. 10	<0.04	894.2	214.4	11.05
Aq. Raff. 15	<0.04	871.7	208.0	11.10
Aq. Raff. 20	<0.01	911.7	207.4	10.90
Aq. Raff. 25	<0.04	880.7	199.1	10.42
Aq. Raff. 30	<0.04	892.7	209.9	11.80
Aq. Raff. 35	<0.04	884.9	210.2	12.41
Aq. Raff. 40	<0.01	901.4	210.7	11.50
Aq. Raff. 45	<0.04	905.9	204.5	12.86
Aq. Raff. 50	<0.04	871.1	205.1	12.60
Aq. Raff. 55	<0.04	866.5	207.2	12.85
Aq. Raff. 60	<0.01	884.1	211.8	12.20
Aq. Raff. 65	<0.04	901.5	203.9	12.74
Aq. Raff. 70	<0.04	897.1	201.0	12.56
Aq. Raff. 72	<0.04	896.9	203.4	12.71
Aq. Composite	<0.01	867.1	203.8	11.1

	Al	Ag	B	Ba	Ca	Cl	Cr	F	K	Mn
Sample*					(mg/L)					
Aq. Raff. 20	1.04E+04	0.046	139.8	10.737	1348	958	168.7	3070	3640	305.4
Aq. Raff. 40	1.03E+04	0.057	139.1		1331	910	171.6	2750	3770	301.7
Aq. Raff. 60	1.02E+04	0.067	138.3		1325	908	166.8	3030	3870	299.9
Aq. Composite	1.02E+04	0.065	136.1		1328	875	170.4	4560	3520	296.9

	Mo	Na	Ni	Pb	Pd	Rh	Ru	Se	Sr	Zr
Sample*					(mg/L)					
Aq. Raff. 20	2.92	2.09E+04	77.51	134.5	<0.07	0.237	1.329	1.697	65.82	60.09
Aq. Raff. 40	3.06	2.03E+04	75.48	141.6	<0.07	0.227	1.380	1.472	65.32	59.62
Aq. Raff. 60	2.90	1.95E+04	75.65	131.8	0.092	0.202	1.212	1.560	64.62	58.86
Aq. Composite	2.83	2.00E+04	76.11	128.5	0.166	0.246	1.306	1.617	63.86	59.07

* Aq. Raff. = Aqueous Raffinate. ## after sample indicates time (hrs) at which sample was taken.

Table A- 5. Analytical data for the strip product samples from the TRUEX flowsheet.

Sample[*]	Eu	Fe	Re	Hg
		(mg/L)		
St. Prod. 5	1354.7	2.894	303.2	0.166
St. Prod. 10	1399.4	2.125	309.8	0.189
St. Prod. 15	1391.0	2.741	311.4	0.339
St. Prod. 20	1364.0	4.001	295.1	0.738
St. Prod. 25	1391.0	3.402	301.1	0.880
St. Prod. 30	1410.6	3.068	299.7	0.675
St. Prod. 35	1420.9	2.888	296.7	0.277
St. Prod. 40	1383.7	2.600	299.7	0.878
St. Prod. 45	1401.5	2.898	292.5	0.682
St. Prod. 50	1433.5	2.869	317.9	0.749
St. Prod. 55	1427.0	2.940	303.0	1.037
St. Prod. 60	1340.9	2.405	308.7	0.865
St. Prod. 65	1442.5	3.616	307.2	0.875
St. Prod. 70	1377.2	3.253	305.1	1.052
St. Prod. 72	1397.1	3.028	307.9	0.207
St. Prod. Composite	1382.4	2.835	289.4	1.12

Sample[*]	Al	Ag	B	Ba	Ca	Cl	Cr	F	K	Mn
					(mg/L)					
St. Prod. 20	<0.08	<0.03	<0.07	<0.01	0.202	<35.3	0.233	6.4	0.099	0.017
St. Prod. 40	<0.08	<0.03	<0.07	<0.01	0.107	<17.7	0.023	10	0.097	<0.01
St. Prod. 60	<0.08	<0.03	<0.07	<0.01	0.134	<17.7	0.036	9.96	0.138	<0.01
St. Prod. Composite	<0.08	<0.03	<0.07	<0.01	0.149	<17.7	0.029	11.2	0.094	<0.01

Sample[*]	Mo	Na	Ni	Pb	Pd	Rh	Ru	Se	Sr	Zr
					(mg/L)					
St. Prod. 20	151.4	0.336	0.120	0.502	<0.07	<0.10	<0.09	<0.13	<0.01	43.20
St. Prod. 40	141.0	0.306	<0.03	0.622	<0.07	0.107	<0.09	<0.13	<0.01	43.93
St. Prod. 60	127.9	0.220	<0.03	0.750	<0.07	<0.10	<0.09	<0.13	<0.01	41.10
St. Prod. Composite	139.4	0.987	0.042	0.695	<0.07	0.113	<0.09	<0.13	<0.01	40.68

[*] St. Prod. = Strip Product. ## after sample indicates time (hrs) at which sample was taken.

Table A- 6. Analytical data for the carbonate wash samples from the TRUEX flowsheet.

	Eu	Fe	Re	Hg
Sample[*]	(mg/L)			
Car. Wash 5	<0.04	0.048	138.2	335.1
Car. Wash 10	<0.04	<0.04	128.5	399.0
Car. Wash 15	<0.04	<0.04	146.6	307.1
Car. Wash 20	0.011	0.034	161.3	286.0
Car. Wash 25	<0.04	<0.04	152.0	247.5
Car. Wash 30	<0.04	<0.04	149.8	341.0
Car. Wash 35	<0.04	<0.04	143.0	310.4
Car. Wash 40	0.036	0.046	128.3	290.0
Car. Wash 45	<0.04	<0.04	139.3	270.0
Car. Wash 50	<0.04	<0.04	131.8	257.7
Car. Wash 55	<0.04	<0.04	141.6	304.8
Car. Wash 60	0.097	0.034	122.4	277.0
Car. Wash 65	<0.04	<0.04	133.2	287.9
Car. Wash 70	<0.04	<0.04	141.2	272.2
Car. Wash 72	<0.04	<0.04	144.1	239.2
Car Wash Composite	4.408	0.1575	136.7	314.0

	Al	Ag	B	Ba	Ca	Cl	Cr	F	K	Mn
Sample[*]	(mg/L)									
Car. Wash 20	<0.08	<0.03	<0.07	0.027	1.012	170	<0.01	<1.43	0.102	<0.01
Car. Wash 40	<0.08	<0.03	<0.07	0.027	0.897	148	<0.01	<2.87	0.105	<0.01
Car. Wash 60	<0.08	<0.03	<0.07	0.032	0.977	141	<0.01	<2.87	0.105	<0.01
Car Wash Composite	0.109	<0.03	<0.07	0.027	1.014	147	0.029	<2.87	0.131	<0.01

	Mo	Na	Ni	Pb	Pd	Se	Sr	Rh	Ru	Zr
Sample[*]	(mg/L)									
Car. Wash 20	0.945	11500	<0.03	0.412	<0.07	<0.13	<0.01	<0.10	0.113	0.227
Car. Wash 40	0.811	11700	<0.03	0.151	<0.07	<0.13	<0.01	<0.10	0.160	0.151
Car. Wash 60	0.764	11000	<0.03	0.218	<0.07	<0.13	<0.01	<0.10	0.153	0.130
Car Wash Composite	1.68	11400	0.067	0.349	<0.07	<0.13	<0.01	<0.10	0.183	0.168

[*] Car. Wash = Carbonate Wash Product. ## after sample indicates time (hrs) at which sample was taken.

Table A- 7. Analytical results for the acid rinse samples from the TRUEX flowsheet.

Sample[*]	Eu	Fe	Re	Hg
	(mg/L)			
Rinse Prod. 5	***** No Sample ****			
Rinse Prod. 10	<0.04	0.2667	<0.06	0.103
Rinse Prod. 15	<0.04	0.3276	<0.06	0.072
Rinse Prod. 20	<0.04	0.21	0.0609	0.084
Rinse Prod. 25	<0.04	0.2415	0.0861	0.077
Rinse Prod. 30	<0.04	0.2289	0.0609	0.116
Rinse Prod. 35	<0.04	0.1995	<0.06	0.070
Rinse Prod. 40	0.0084	0.256	0.0294	0.099
Rinse Prod. 45	<0.04	0.2016	<0.06	0.097
Rinse Prod. 50	<0.04	0.2373	<0.06	0.114
Rinse Prod. 55	<0.04	0.1785	<0.06	0.117
Rinse Prod. 60	<0.04	0.1785	<0.06	0.097
Rinse Prod. 65	<0.04	0.1848	<0.06	0.095
Rinse Prod. 70	<0.04	0.1701	<0.06	0.098
Rinse Prod. 72	<0.04	0.1911	0.0651	0.084
Rinse Prod. Composite	0.0336	3.207	0.7182	0.281

Sample[*]	Al	Ag	B	Ba	Ca	Cl	Cr	F	K	Mn
	(mg/L)									
Rinse Prod. 40	0.0861	<0.03	0.3423	<0.01	<0.03	<17.7	0.0357	<2.87	0.0441	<0.01
Rinse Prod. Composite	31.563	<0.03	0.691	0.0378	3.872	<17.7	0.548	<2.87	10.6	0.9513

Sample[*]	Mo	Na	Ni	Pb	Pd	Se	Sr	Rh	Ru	Zr
	(mg/L)									
Rinse Prod. 40	0.729	17.1	<0.03	<0.08	<0.07	<0.13	<0.01	<0.10	<0.09	<0.03
Rinse Prod. Composite	0.9576	37.3	0.2457	0.4074	<0.07	<0.13	0.1932	<0.10	<0.09	0.1197

[*] Rinse Prod. = Rinse Product. ## after sample indicates time (hrs) at which sample was taken.

Table A- 8. Analytical results for the organic product samples from the TRUEX flowsheet*.

Sample**	Eu	Fe	Re	Hg
		(mg/L)		
Org. Prod. 5	<0.20	1.733	<0.29	0.588
Org. Prod. 10	<0.20	1.712	<0.29	0.556
Org. Prod. 15	<0.20	1.712	<0.29	0.519
Org. Prod. 20	0.14	1.880	0.294	<0.05
Org. Prod. 25	<0.20	1.733	<0.29	0.442
Org. Prod. 30	<0.20	1.764	<0.29	0.480
Org. Prod. 35	<0.20	1.754	<0.29	0.397
Org. Prod. 40	0.13	1.922	0.168	0.162
Org. Prod. 45	<0.20	1.691	<0.29	0.432
Org. Prod. 50	<0.20	1.691	<0.29	0.470
Org. Prod. 55	<0.20	1.796	<0.29	0.481
Org. Prod. 60	0.07	1.943	0.105	<0.05
Org. Prod. 65	<0.20	1.701	<0.29	0.638
Org. Prod. 70	<0.20	1.670	<0.29	0.460
Org. Prod. 72	<0.20	1.628	<0.29	0.498
Org. Composite	0.002	0.102	<0.01	<0.008

Sample	Al	Ag	B	Ba	Ca	Cl	Cr	F	K	Mn
					(mg/L)					
Org. Prod. 20	1.86	0.200	3.76	<0.05	7.56	<177	0.053	<14.4	0.585	<0.06
Org. Prod. 40	1.52	0.179	3.55	<0.05	7.56	<177	0.063	<14.4	0.690	<0.06
Org. Prod. 60	1.64	<0.15	4.18	<0.05	8.06	<89	0.074	<14.4	0.452	<0.06
Org. Composite	2.78	<0.15	4.95	<0.05	7.66	<89	0.053	<14.4	0.130	<0.06

Sample	Mo	Na	Ni	Pb	Pd	Rh	Ru	Se	Sr	Zr
					(mg/L)					
Org. Prod. 20	1.05	8.15	<0.15	<0.39	<0.34	<0.51	<0.44	<0.65	<0.06	3.76
Org. Prod. 40	1.56	7.1	<0.15	0.998	<0.34	<0.51	<0.44	<0.65	<0.06	2.27
Org. Prod. 60	1.97	7.05	<0.15	<0.39	<0.34	<0.51	<0.44	<0.65	<0.06	1.74
Org. Composite	1.09	8.35	<0.15	<0.39	<0.34	<0.51	<0.44	<0.65	<0.06	1.13

[*] Sample results reflect dilution from back extraction of organic phase components with aqueous strip solutions.

[**] Org. Prod. = Organic Product, ## after sample indicates the time (hrs) when the sample was taken.

Table A- 9. Percentages of components in each stream and material balance data calculated from composite TRUEX samples.

Sample	Eu	Fe	Re	Hg	Al	Ag	B	Ba	Ca	Cl	Cr	F
Aq. Raf. Comp.	<0.001	94.32	43.67	3.71	98.56	190.1	96.949	103.1	100.0	80.2	100.8	143.3
St. Prod. Comp.	105.8	0.18	35.83	0.22	<0.00	<49.6	<0.03	<0.06	0.006	<0.94	0.010	0.203
Car. Wash Comp.	0.382	0.01	19.16	68.68	0.001	<56	<0.03	<0.17	0.050	8.8	0.011	<0.06
Rinse Prod. Comp.	0.000	0.03	0.01	0.007	0.022	<6.3	<0.04	0.027	0.021	<0.12	0.024	<0.01
Org. Prod. Comp.	0.002	0.10	<0.01	<0.008	0.012	<196	<1.61	<0.23	0.263	<3.70	0.014	<0.21
Solid Hg				22.07								
Material Balance	106.2	94.54	98.67	94.68	98.59	190.1	96.95	103.2	100.1	89.0	100.9	143.5
Normalized												
Aq. Raf. Comp.	0.000	99.773	44.254	3.919	99.977	100.0	100.0	99.974	99.922	90.096	99.96	99.86
St. Prod. Comp.	99.640	0.189	36.313	0.229	0.000	0.000	0.000	0.000	0.006	<0.00	0.010	0.142
Car. Wash Comp.	0.360	0.012	19.422	72.536	0.001	0.000	0.000	0.000	0.050	9.904	0.011	0.000
Rinse Prod. Comp.	0.000	0.027	0.011	0.007	0.023	0.000	0.000	0.026	0.021	<0.00	0.024	0.000
Org. Prod. Comp.	0.002	0.108	0.008	0.008	0.012	0.000	0.000	0.000	0.263	<0.00	0.014	0.000
Solid Hg				23.31								
Material Balance	100	100	100	100	100	100	100	100	100	100	100	100
	K	Mn	Mo	Na	Ni	Pb	Pd	Rh	Ru	Se	Sr	Zr
Aq. Raf. 80	95.49	98.09	3.30	94.99	103.4	92.68	348.5	174	89.43	98.75	96.84	71.49
St. Prod. 80	0.001	<0.00	93.86	0.003	0.03	0.29	<81.5	46.3	<3.49	<4.6	<0.01	28.46
Car. Wash 80	0.002	<0.00	1.28	0.000	0.06	0.17	<92.3	<48	8.19	<5.2	<0.01	0.13
Rinse Prod. 80	0.021	0.023049	0.08	0.013	0.02	0.02	<10.3	<5.3	<0.44	<0.6	0.02	0.01
Org. Prod. 80	0.002	<0.01	0.58	0.018	<0.09	0.13	<322	<166	<13.8	<18.2	<0.04	0.63
Material Balance	95.52	98.12	98.52	95.01	103.5	93.16	349	220	97.62	98.75	96.86	100.1
Normalized												
Aq. Raf. 80	99.97	99.98	3.35	99.98	99.89	99.49	100.0	78.94	91.61	100.0	99.98	71.4
St. Prod. 80	0.002	0.00	95.27	0.00	0.03	0.31	0.000	21.06	0.000	0.000	0.000	28.4
Car. Wash 80	0.002	0.00	1.30	0.00	0.06	0.18	0.000	0.000	8.39	0.000	0.000	0.13
Rinse Prod. 80	0.022	0.02	0.08	0.01	0.02	0.02	0.000	0.000	0.000	0.000	0.02	0.01
Org. Prod. 80	0.002	0.00	0.59	0.01	0.00	0.14	0.000	0.000	0.000	0.000	0.000	0.63
Material Balance	100	100	100	100	100	100	100	100	100	100	100	100

Table A- 10. Stagewise concentration data for distribution coefficient calculation in the TRUEX flowsheet.

	Eu	Fe	Re	Hg	H+		Eu	Fe	Re	Hg	H+
Sample		(mg/L)			(*M*)	Sample		(mg/L)			(*M*)
Stage1 –O	<0.20	627.6	145.8	7.5	0.78	Stage13-O	29.86	1.45	542.0	310.5	0.028
Stage1-A	<0.04	829.5	186.1	12.8	1.13	Stage13-A	1668	4.25	273.6	0.594	0.176
Dist. Ratio	---	0.757	0.784	0.586	0.689	Dist. Ratio	0.018	0.341	1.98	523	0.159
Stage2-O	<0.20	822.3	175.1	10.3	0.852	Stage14-O	<0.20	1.42	530.8	31.4	
Stage2-A	<0.04	890.8	262.5	20	1.416	Stage14-A	26.61	1.56	457.4	0.236	
Dist. Ratio	---	0.923	0.667	0.513	0.602	Dist. Ratio	<0.01	0.911	1.16	133	
Stage3-O	<0.20	952.6	200.1	11.5	0.873	Stage15-O	<0.20	1.45	473.6	247	0.014
Stage3-A	<0.04	1009	293.2	24.3	1.512	Stage15-A	0.57	1.05	430.1	0.248	0.107
Dist. Ratio	---	0.944	0.683	0.472	0.577	Dist. Ratio	<0.35	1.38	1.10	996	0.131
Stage4-O	<0.20	1095	210.5	13.25	0.858	Stage16-O	<0.20	1.449	366.1	176	
Stage4-A	<0.04	1143	323	26.6	1.455	Stage16-A	<0.04	0.384	354.9	0.141	
Dist. Ratio	---	0.958	0.652	0.498	0.590	Dist. Ratio	---	3.77	1.03	1249	
Stage5-O	1.418	1068	258.1	25.6	0.868	Stage17-O	<0.20	1.42	260.1	328.5	
Stage-5A	<0.04	1151	355.5	35.8	1.442	Stage17-A	<0.04	0.725	254.9	0.242	
Dist. Ratio	>35.53	0.928	0.726	0.715	0.602	Dist. Ratio	---	1.96	1.02	1358	
Stage6-O	17.34	1163	295.4	43.5	0.859	Stage18-O	<0.20	1.43	141.0	315	0.012
Stage6-A	<0.50	1258	386.8	53.2	1.433	Stage18-A	<0.04	0.340	135.5	0.274	0.148
Dist. Ratio	>34.54	0.925	0.764	0.818	0.599	Dist. Ratio	---	4.18	1.04	1150	0.081
Stage7-O	193.2	1115	334.7	114	0.858	Stage19-O	<0.20	1.46	9.104	0.7	
Stage7-A	<6.28	1212	418	86.4	1.417	Stage19-A	<0.04	0.149	154.8	176	
Dist. Ratio	>30.75	0.919	0.800	1.314	0.606	Dist. Ratio	---	9.79	0.059	0.004	
Stage8-O	1653	908	361	163	0.847	Stage20-O	<0.20	1.40	0.777	0.63	
Stage8-A	67.28	1316	443.1	129	1.392	Stage20-A	<0.04	<0.04	12.5	0.067	
Dist. Ratio	24.56	0.690	0.815	1.260	0.608	Dist. Ratio	---	>31.7	0.062	9.389	
Stage9-O	2091	176	352.9	285	0.513	Stage21-O	<0.20	1.481	<0.29	0.475	
Stage9-A	145.9	1512	146.3	36.9	1.202	Stage21-A	<0.04	<0.04	0.760	0.021	
Dist. Ratio	14.33	0.116	2.412	7.72	0.427	Dist. Ratio	---	>33.6	<0.39	22.60	
Stage10-O	2254	37.55	357.8	289		Stage22-O	<0.20	1.62	<0.29	0.665	
Stage10-A	187.5	650.8	67.77	18.6		Stage22-A	<0.04	1.36	<0.06	0.75	
Dist. Ratio	12.02	0.058	5.28	15.5		Dist. Ratio	---	1.19	---	0.887	
Stage11-O	2411	7.445	385.1	404		Stage23-O	<0.20	1.44	<0.29	0.4375	0.01
Stage11-A	318.8	213.4	31.29	13.1		Stage23-A	<0.04	0.470	<0.06	0.771	0.061
Dist. Ratio	7.56	0.035	12.31	30.8		Dist. Ratio	---	3.06	---	0.567	
Stage12-O	2050	2.32	393.0	433	0.072						
Stage12-A	544.3	52.5	20.30	12.7	0.21						
Dist. Ratio	3.77	0.044	19.36	34.1	0.343						

Table A- 11. Approach to steady data for the raffinate and strip product samples in the SREX flowsheet.

Sample[*]	Sr	K (mg/L)	Pb	H^+ (*M*)
SREX Feed	63.862	3.52E+03	128.47	---
Aqueous Raffinate				
Aqueous Raffinate – 0.5	<0.013	308	---	1.38
Aqueous Raffinate – 1.0	0.0147	1890	---	1.41
Aqueous Raffinate – 1.5	0.0294	2160	---	1.39
Aqueous Raffinate – 2.0	0.0336	2260	---	1.40
Aqueous Raffinate – 2.5	0.0399	2210	---	1.40
Aqueous Raffinate – 3.0	0.0420	162.3	---	1.39
Sr Strip Product				
Strip 1 Product – 0.5	20.1	---	---	0.22
Strip 1 Product – 1.0	23.4	---	---	0.23
Strip 1 Product – 1.5	24.6	---	---	0.23
Strip 1 Product – 2.0	24.8	---	---	0.22
Strip 1 Product – 2.5	23.9	---	---	0.23
Strip 1 Product – 3.0	24.7	---	---	0.23
Pb Strip Product				
Strip 2 Product – 0.5	---	---	69.5	---
Strip 2 Product – 1.0	---	---	91.9	---
Strip 2 Product – 1.5	---	---	93.8	---
Strip 2 Product – 2.0	---	---	93.1	---
Strip 2 Product – 2.5	---	---	93.7	---
Strip 2 Product – 3.0	---	---	94.3	---

[*] #.# following sample indicates time sample was taken relative to time zero.

Table A- 12. Analytical data for the aqueous raffinate samples from the SREX flowsheet.

Sample[*]	Sr	K	Pb	Zr
		(mg/L)		
SREX Feed	63.9	3.52E+03	129	59.07
Aq. Raff – 5	0.046	2.39E+03	<0.11	---
Aq. Raff-10	0.050	2.48E+03	<0.11	---
Aq. Raff-15	0.053	2.62E+03	<0.11	---
Aq. Raff-20	0.059	2.35E+03	<0.11	40.93
Aq. Raff-25	0.055	2.51E+03	<0.11	44.12
Aq. Raff-30	0.061	2.50E+03	<0.11	41.66
Aq. Raff-35	0.061	2.55E+03	<0.11	43.55
Aq. Raff-40	0.069	2.54E+03	<0.11	40.97
Aq. Raff-45	0.074	2.63E+03	<0.11	43.20
Aq. Raff-50	0.074	2.69E+03	<0.11	43.18
Aq. Raff-55	0.071	2.61E+03	<0.11	42.99
Aq. Raff-60	0.078	2.40E+03	<0.11	40.66
Aq. Raff-65	0.076	2.61E+03	<0.11	43.49
Aq. Raff-70	0.084	2.47E+03	<0.11	43.28
Aq. Raff-75	0.071	2.48E+03	<0.11	41.81
Aq. Raff-78	0.071	2.45E+03	<0.11	866.0
Aq. Composite	0.067	2.43E+03	<0.11	41.01

Sample[*]	Al	Ag	B	Ba	Ca	Cl	Cr	F	Fe
					(mg/L)				
SREX Feed	1.02E+04	0.065	136.1	10.74	1328	875	170.4	4560	867
Aq. Raff-20	7.48E+03	<0.03	91.98	2.76	857	689	115.2	131.1	624
Aq. Raff-40	7.41E+03	<0.03	93.14	2.95	864	692	115.8	2332	632
Aq. Raff-60	7.24E+03	<0.03	92.14	3.10	851	680	114.6	2243	627
Aq. Raff-78	7.32E+03	<0.03	1.39	0.46	41.5	675	116.3	2274	640
Aq. Composite	7.29E+03	<0.03	93.68	2.94	866	673	115.6	2240	631

Sample[*]	H+	Hg	Na	Mo	Mn	Ni	Se	Re	Rh
	(M)				(mg/L)				
SREX Feed	---	11.1	2.00E+04	2.83	297	76.1	1.62	204	0.246
Aq. Raff-20	1.38	0.33	1.91E+04	2.00	206	49.1	1.31	4.63	0.252
Aq. Raff-40	1.38	0.39	2.00E+04	2.15	202	52.3	1.37	5.36	0.227
Aq. Raff-60	1.37	---	2.01E+04	2.08	202	50.7	1.34	6.19	0.244
Aq. Raff-78	---	---	1.92E+04	2.11	204	50.6	1.31	6.24	0.248
Aq. Composite	---	0.43	1.93E+04	2.07	206	52.0	1.28	5.49	0.275

[*] Aq. Raff. = Aqueous Raffinate, #.# following sample indicates time sample was taken relative to time zero.

Table A- 13. Analytical data for the Sr strip #1 product samples from the SREX flowsheet.

	Sr	K	Pb	Zr
Sample[*]	(mg/L)			
St1 Prod – 5	24.15	140	8.16	<0.03
St1 Prod-10	23.92	26.7	7.78	<0.03
St1 Prod-15	24.38	53.4	7.84	<0.03
St1 Prod-20	23.46	129	8.06	0.0357
St1 Prod-25	22.45	49.8	8.14	<0.03
St1 Prod-30	23.71	120	7.62	<0.03
St1 Prod-35	22.22	38.3	8.41	<0.03
St1 Prod-40	23.48	123	7.54	<0.03
St1 Prod-45	23.14	110	7.86	<0.03
St1 Prod-50	23.14	100	7.29	<0.03
St1 Prod-55	23.00	112	6.96	<0.03
St1 Prod-60	22.11	110	7.68	<0.03
St1 Prod-65	22.39	106	7.61	<0.03
St1 Prod-70	22.74	105	7.35	<0.03
St1 Prod-75	22.30	113	6.70	<0.03
St1 Prod-78	22.64	119	6.05	<0.03
St1 Composite	23.10	123	7.62	<0.03

	Al	Ag	B	Ba	Ca	Cl	Cr	F	Fe
Sample[*]	(mg/L)								
St1 Prod-20	0.321	<0.03	<0.07	2.64	0.622	<35.3	0.0126	<2.87	0.048
St1 Prod-40	0.218	<0.03	<0.07	2.55	0.592	<35.3	0.0105	<2.87	0.048
St1 Prod-60	0.223	<0.03	<0.07	2.32	0.573	<35.3	0.0084	<2.87	0.053
St1 Prod-78	0.200	<0.03	<0.07	2.41	0.611	<35.3	<0.015	<2.87	0.034
St1 Composite	<0.08	<0.03	<0.07	2.51	<0.07	<17.7	<0.01	<2.87	<0.03

	H+	Hg	Na	Mo	Mn	Ni	Se	Re	Rh
Sample[*]	*(M)*	(mg/L)							
St1 Prod-20	0.22		77.0	0.090	<0.01	<0.03	<0.13	68.86	<0.10
St1 Prod-40	0.23		73.2	0.040	<0.01	<0.03	<0.13	68.25	<0.10
St1 Prod-60	0.22		69.3	0.023	<0.01	<0.03	<0.13	64.16	<0.10
St1 Prod-78	0.21	0.017	72.8	0.027	<0.01	<0.03	<0.13	66.74	<0.10
St1 Composite	---	0.008	51.1	0.032	<0.01	<0.03	<0.13	67.58	<0.10

[*] St1 Prod. = Sr Strip 1 Product, #.# following sample indicates time sample was taken relative to time zero.

Table A- 14. Analytical data for the Pb strip 2 product samples from the SREX flowsheet.

	Sr	K	Pb	Zr
Sample [*]		(mg/L)		
St2 Prod – 5	<0.013	0.82	93.6	<0.03
St2 Prod-10	<0.013	0.85	99.4	<0.03
St2 Prod-15	<0.013	0.88	96.2	<0.03
St2 Prod-20	<0.013	0.53	91.8	<0.03
St2 Prod-25	<0.013	1.09	90.6	<0.03
St2 Prod-30	<0.013	1.09	95.3	<0.03
St2 Prod-35	<0.013	1.09	90.6	<0.03
St2 Prod-40	<0.013	1.02	98.6	<0.03
St2 Prod-45	<0.013	1.10	88.6	<0.03
St2 Prod-50	<0.013	1.10	90.0	<0.03
St2 Prod-55	<0.013	1.11	92.4	<0.03
St2 Prod-60	<0.013	1.03	92.6	<0.03
St2 Prod-65	<0.013	1.13	92.2	<0.03
St2 Prod-70	<0.013	1.12	94.4	<0.03
St2 Prod-75	<0.013	1.13	97.5	<0.03
St2 Prod-78	<0.013	0.998	96.6	<0.03
St2 Composite	<0.013	1.07	91.9	<0.03

	Al	Ag	B	Ba	Ca	Cl	Cr	F	Fe
Sample [*]					(mg/L)				
St2 Prod-20	0.174	<0.03	<0.07	<0.01	0.603	<35.3	<0.01	<2.87	0.057
St2 Prod-40	0.281	<0.03	<0.07	<0.01	0.626	<35.3	0.011	<2.87	0.107
St2 Prod-60	0.252	<0.03	<0.07	0.011	0.613	<35.3	<0.01	<2.87	0.053
St2 Prod-78	0.231	<0.03	<0.07	<0.01	0.590	<35.3	<0.01	<2.87	0.042
St2 Composite	<0.08	<0.03	<0.07	<0.01	<0.07	<17.7	0.017	<2.87	0.084

	Hg	Na	Mo	Mn	Ni	Se	Re	Rh
Sample [*]				(mg/L)				
St2 Prod-20	---	0.148	<0.02	<0.01	<0.03	<0.13	6.05	<0.10
St2 Prod-40	---	0.136	<0.02	<0.01	<0.03	<0.13	5.62	<0.10
St2 Prod-60	---	0.121	<0.02	<0.01	<0.03	<0.13	4.06	<0.10
St2 Prod-78	3.75	0.128	<0.02	<0.01	<0.03	<0.13	4.96	<0.10
St2 Composite	3.04	0.119	<0.02	<0.01	<0.03	<0.13	5.29	<0.10

[*] St2 Prod = Pb Strip 2 Product, #.# following sample indicates time sample was taken relative to time zero.

Table A- 15. Analytical data for the carbonate wash product samples from the SREX flowsheet.

Sample[*]	Sr	K	Pb	Zr
		(mg/L)		
Car. Wash – 5	<0.013	0.165	0.750	<0.03
Car. Wash-10	<0.013	0.153	0.670	<0.03
Car. Wash-15	<0.013	0.104	0.683	<0.03
Car. Wash-20	<0.013	0.096	0.538	<0.03
Car. Wash-25	<0.013	0.101	0.655	<0.03
Car. Wash-30	<0.013	---	0.548	<0.03
Car. Wash-35	<0.013	---	0.565	<0.03
Car. Wash-40	<0.013	0.113	0.561	<0.03
Car. Wash-45	<0.013	0.118	0.540	<0.03
Car. Wash-50	<0.013	0.122	0.634	<0.03
Car. Wash-55	<0.013	0.123	0.510	<0.03
Car. Wash-60	<0.013	0.095	0.584	<0.03
Car. Wash-65	<0.013	0.114	0.565	<0.03
Car. Wash-70	<0.013	0.122	0.599	<0.03
Car. Wash-75	<0.013	0.124	0.578	<0.03
Car. Wash-78	<0.013	0.105	0.594	<0.03
Wash Composite	<0.013	0.191	0.601	1.34

Sample[*]	Al	Ag	B	Ba	Ca	Cl	Cr	F	Fe
					(mg/L)				
Car. Wash-20	<0.08	<0.03	<0.07	0.032	1.18	<35.3	<0.01	<2.87	0.044
Car. Wash-40	<0.08	<0.03	<0.07	0.029	1.19	<35.3	<0.01	<2.87	0.038
Car. Wash-60	<0.08	<0.03	<0.07	0.029	1.15	<35.3	<0.01	<2.87	0.029
Car. Wash-78	<0.08	<0.03	<0.07	0.029	1.17	<35.3	<0.01	<2.87	0.034
Wash Composite	<0.08	<0.03	<0.07	0.027	1.11	<8.84	<0.01	<4.67	0.126

Sample[*]	Hg	Na	Mo	Mn	Ni	Se	Re	Rh
				(mg/L)				
Car. Wash-20	---	1.21E+04	<0.02	<0.01	<0.03	<0.13	6.19	<0.10
Car. Wash-40	---	1.23E+04	<0.02	<0.01	<0.03	<0.13	4.56	<0.10
Car. Wash-60	---	1.23E+04	<0.02	<0.01	<0.03	<0.13	3.12	<0.10
Car. Wash-78	8.48	1.27E+04	<0.02	<0.01	<0.03	<0.13	3.71	<0.10
Wash Composite	6.30	8.34E+03	<0.02	<0.01	<0.03	<0.13	4.73	<0.10

[*] Car Wash = Carbonate Wash Product, #.# following sample indicates time sample was taken relative to time zero.

Table A- 16. Analytical data for the acid rinse product samples from the SREX flowsheet.

Sample[*]	Sr	K	Pb	Zr
	(mg/L)			
Rinse Prod – 5	---	0.054	<0.11	<0.03
Rinse Prod-10	---	0.040	<0.11	<0.03
Rinse Prod-15	---	---	<0.11	<0.03
Rinse Prod-20	<0.013	0.012	<0.11	<0.03
Rinse Prod-25	---	0.043	<0.11	<0.03
Rinse Prod-30	---	0.067	<0.11	<0.03
Rinse Prod-35	---	0.038	<0.11	<0.03
Rinse Prod-40	<0.013	0.016	<0.11	<0.03
Rinse Prod-45	---	0.009	<0.11	<0.03
Rinse Prod-50	---	0.011	<0.11	<0.03
Rinse Prod-55	---	0.009	<0.11	<0.03
Rinse Prod-60	<0.013	0.023	<0.11	<0.03
Rinse Prod-65	---	0.011	<0.11	<0.03
Rinse Prod-70	---	0.020	<0.11	<0.03
Rinse Prod-75	---	0.014	<0.11	<0.03
Rinse Prod-78	<0.013	0.018	<0.11	<0.03
Rinse Composite	<0.013	0.126	<0.11	<0.03

Sample[*]	Al	Ag	B	Ba	Ca	Cl	Cr	F	Fe
					(mg/L)				
Rinse Prod-20	<0.08	<0.03	<0.07	<0.01	<0.07	<35.3	0.013	<11.5	0.088
Rinse Prod-40	<0.08	<0.03	<0.07	<0.01	<0.07	<35.3	0.011	<2.87	0.088
Rinse Prod-60	<0.08	<0.03	<0.07	<0.01	0.130	<35.3	0.011	<2.87	0.101
Rinse Prod-78	<0.08	<0.03	<0.07	<0.01	<0.07	<35.3	0.011	<2.87	0.078
Rinse Composite	0.20	<0.03	<0.07	<0.01	<0.07	<17.7	0.021	<2.87	0.189

Sample[*]	H+	Hg	Na	Mo	Mn	Ni	Re	Rh	Se
	(M)				(mg/L)				
Rinse Prod-20	1.14	---	8.00	<0.02	<0.01	<0.03	1.92	<0.10	<0.13
Rinse Prod-40	---	---	7.78	<0.02	<0.01	<0.03	1.43	<0.10	<0.13
Rinse Prod-60	---	---	7.79	<0.02	<0.01	<0.03	0.956	<0.10	<0.13
Rinse Prod-78	1.19	0.29	8.66	<0.02	<0.01	<0.03	1.14	<0.10	<0.13
Rinse Composite	---	0.14	12	<0.02	0.013	0.046	1.51	<0.10	<0.13

[*] Rinse Prod. = Rinse Product, #.# following sample indicates time sample was taken relative to time zero.

Table A- 17. Analytical data for the organic product samples from the SREX flowsheet.

Sample[*]	Sr	K	Pb	Zr
		(mg/L)		
Org. Prod - 5	<0.126	10.7	<1.07	<0.27
Org. Prod –10	<0.126	12.1	<1.07	<0.27
Org. Prod – 15	<0.126	12.3	<1.07	<0.27
Org. Prod – 20	<0.126	11.2	<1.07	<0.27
Org. Prod – 25	<0.126	12.1	<1.07	<0.27
Org. Prod – 30	<0.126	11.8	<1.07	<0.27
Org. Prod – 35	<0.126	12.4	<1.07	<0.27
Org. Prod – 40	<0.126	11.7	<1.07	<0.27
Org. Prod – 45	<0.126	12.3	<1.07	<0.27
Org. Prod – 50	<0.126	12.8	<1.07	<0.27
Org. Prod – 55	<0.126	13.3	<1.07	<0.27
Org. Prod – 60	<0.126	11.6	1.62	<0.27
Org. Prod – 65	<0.126	12.7	<1.07	<0.27
Org. Prod – 70	<0.126	12.0	<1.07	<0.27
Org. Prod – 75	<0.126	12.0	<1.07	<0.27
Org. Prod – 78	<0.126	11.6	<1.07	<0.27
Org. Composite	<0.126	12.4	<1.07	<0.27

Sample[*]	Al	Ag	B	Ba	Ca	Cl	Cr	F	Fe
					(mg/L)				
Org. Prod – 20	<0.78	<0.29	<0.71	<0.11	<0.71	<353	<0.08	<28.7	<0.27
Org. Prod – 40	<0.78	<0.29	<0.71	<0.11	<0.71	<177	<0.08	<28.7	1.13
Org. Prod – 60	<0.78	<0.29	<0.71	<0.11	<0.71	<177	0.29	<28.7	0.40
Org. Prod – 78	<0.78	<0.29	<0.71	<0.11	<0.71	<177	0.13	<28.7	<0.27
Org. Composite	<0.78	<0.29	<0.71	<0.11	<0.71	<177	0.084	<28.7	0.32

Sample[*]	H+	Hg	Na	Mo	Mn	Ni	Re	Rh	Se
	(*M*)				(mg/L)				
Org. Prod – 20	0.95	---	4.31	<0.19	<0.13	<0.29	0.46	<1.03	<1.30
Org. Prod – 40	0.95	---	4.29	<0.19	<0.13	<0.29	0.27	<1.03	<1.30
Org. Prod – 60	0.94	---	4.36	<0.19	<0.13	0.32	0.48	<1.03	<1.30
Org. Prod – 78	0.95	2	4.63	<0.19	<0.13	<0.29	<0.17	<1.03	<1.30
Org. Composite	---	1.92	3.98	<0.19	<0.13	<0.29	0.27	<1.03	<1.30

[*] Org Prod. = Organic Product, #.# following sample indicates time sample was taken relative to time zero.

Table A- 18. Percentages of components in each stream and material balance data calculated from composite SREX samples.

Sample	Sr	K	Pb	Zr	Al	Ag	B	Ba	Ca	Cl	Cr
Aq. Raf. Comp.	0.14	92.15	<0.11	92.69	95.55	<60.28	91.92	36.50	87.08	102.7	90.58
St.1 Comp.	100.8	9.74	16.53	<0.13	<0.002	<126	<0.15	65.22	<0.01	<5.63	<0.01
St. 2 Comp.	<0.03	0.04	93.23	<0.06	<0.001	<58.8	<0.07	<0.13	<0.01	<2.63	0.013
Wash Comp.	<0.005	0.001	0.12	0.57	<0.000	<11.32	<0.01	0.06	0.021	<0.25	<0.001
Rinse Comp	<0.01	0.002	<0.05	<0.03	0.001	<27.48	<0.03	<0.06	<0.003	<1.23	0.008
Org. Comp.	<0.26	0.47	<1.12	<0.62	<0.010	<605	<0.70	<1.31	<0.07	<27.06	0.066
Material Balance	101.0	101.9	110.0	<93.5	<95.56	283.7	<92.18	<102.0	<87.13	<112.4	<90.62
					Normalized						
Aq. Raf. Comp.	0.139	90.41	0.101	99.16	99.995	<21.25	99.72	35.79	99.95	91.33	99.96
St.1 Comp.	99.82	9.551	15.02	<0.138	<0.002	<44.34	<0.16	63.96	<0.017	<5.01	<0.02
St. 2 Comp.	<0.025	0.039	84.72	<0.064	<0.001	<20.73	<0.07	<0.125	<0.008	<2.34	0.014
Wash Comp.	<0.005	0.001	0.107	0.607	<0.000	<3.99	<0.01	0.063	0.024	<0.23	<0.001
Rinse Comp	<0.012	0.002	0.046	<0.030	0.001	<9.69	<0.03	<0.058	<0.004	<1.09	0.008
Org. Comp.	<0.262	0.463	1.015	<0.662	<0.011	<214	<0.76	<1.29	<0.083	<24.07	0.073
Material Balance	100	100	100	100	100	100	100	100	100	100	100

Sample	F	Fe	Hg	Mn	Mo	Na	Ni	Re	Rh	Se
Aq. Raf. Comp.	65.57	97.18	5.11	92.74	97.73	84.54	91.27	3.59	149.5	105.4
St.1 Comp.	<0.18	<0.01	0.201	<0.01	3.10	0.467	<0.11	92.37	<117	<22.4
St. 2 Comp.	<0.08	0.013	35.73	<0.01	<0.870	0.001	<0.05	3.38	<54.6	<10.5
Wash Comp.	<0.03	0.004	14.30	<0.001	<0.167	6.86	<0.01	0.581	<10.5	<2.02
Rinse Comp	<0.04	0.013	0.773	0.003	<0.406	0.024	0.037	0.451	<25.5	<4.90
Org. Comp.	<0.84	0.049	23.17	<0.06	<8.94	0.017	<0.52	0.179	<561	<108
Material Balance	<65.89	<97.22	56.11	<92.76	<102.3	91.89	<91.47	100.4	<357	<145
					Normalized					
Aq. Raf. Comp.	99.51	99.96	9.11	99.98	95.56	92.00	99.78	3.58	41.90	72.57
St.1 Comp.	0.27	<0.01	0.36	<0.01	3.03	0.508	<0.12	92.02	32.7	15.45
St. 2 Comp.	0.12	0.013	63.67	<0.01	<0.85	0.001	<0.06	3.37	15.3	7.22
Wash Comp.	0.039	0.004	25.49	<0.001	<0.16	7.47	<0.01	0.579	2.94	1.39
Rinse Comp	0.058	0.014	1.38	0.003	<0.40	0.026	0.040	0.449	7.15	3.37
Org. Comp.	1.28	0.050	41.29	<0.06	<8.74	0.019	<0.57	0.179	157	74.26
Material Balance	100	100	100	100	100	100	100	100	100	100

Table A- 19. Stagewise concentration data for distribution coefficient calculation in the SREX flowsheet.

Stage	Sr	K (mg/L)	Pb	H^+ (M)
Extraction Section				
Stage 1-O	0.231	778	1.43	0.76
Stage 1-A	0.071	3460	<0.11	1.37
Dist. Ratio	3.24	0.225	13.4	0.556
Stage 3-O	0.882	766	2.27	0.67
Stage 3-A	0.37	3480	0.22	1.15
Dist. Ratio	2.38	0.22	10.5	0.582
Stage 5-O	2.90	876	1.49	0.666
Stage 5-A	1.25	3210	0.21	1.14
Dist. Ratio	2.32	0.27	7.1	0.586
Stage 7-O	10.04	881	1.28	0.667
Stage 7-A	4.44	3300	0.166	1.132
Dist. Ratio	2.26	0.27	7.72	0.59
Stage 9-O	32.2	932	2.50	0.662
Stage 9-A	13.6	3190	0.328	1.131
Dist. Ratio	2.37	0.292	7.63	0.586
Stage 10-O	57.2	922	80.98	0.653
Stage 10-A	25.1	3240	0.8130	1.128
Dist. Ratio	2.28	0.285	99.6	0.579
Scrub Section				
Stage 11-O	57.0	612	107	0.544
Stage 11-A	36.6	2610	0.93	1.169
Dist. Ratio	1.56	0.234	115	0.465
Sr Strip 1 Section				
Stage 13-O	4.26	17.3	148	0.053
Stage 13-A	34.2	186	4.2	1.274
Dist. Ratio	0.125	0.093	35.3	0.042
Stage 16-O	<0.13	11.5	100.3	0.004
Stage 16-A	<0.01	0.028	26.9	0.049
Dist. Ratio	---	409	3.73	0.082
Pb Strip 2 Section				
Stage 17-O	<0.13	11.4	1.93	
Stage 17-A	<0.01	1.31	112	
Dist. Ratio	---	8.70	0.017	
Stage 20-O	<0.13	11.3	1.91	
Stage 20-A	<0.01	1.17	0.54	
Dist. Ratio	---	9.66	3.53	
Carbonate Wash Section				
Stage 21-O	<0.13	11.7	2.14	
Stage 21-A	<0.01	0.19	0.79	
Dist. Ratio	---	60.3	2.73	
Stage 22-O	<0.13	11.7	1.49	
Stage 22-A	0.015	0.49	0.63	
Dist. Ratio	<8.8	23.9	2.36	
Acid Rinse Section				
Stage 23-O	<0.13	11.1	2.16	0.39
Stage 23-A	<0.01	0.14	0.11	1.20
Dist. Ratio	---	78.7	20.2	0.33
Stage 26-O	<0.13	11.6	1.20	0.92
Stage 26-A	<0.01	0.11	0.14	2.85
Dist. Ratio	---	103	8.51	0.32

Table A- 20. Concentrations of feed components in the product streams.

	Cs Ion Exchange		TRUEX Process				SREX Process				
Component	IX Feed	IX Product (TRUEX Feed)	Car. Wash (LAW)	Acid Rinse (LAW)	Strip Prod (HAW)	TRUEX Raff. (SREX Feed)	Pb Strip (LAW)	Car Wash (LAW)	Acid Rinse (LAW)	Sr Strip (HAW)	SREX Raff. (LAW)
Acid (*M*)	1.55	1.55	---	---	---	---	---	---	---	---	---
Al (*M*)	0.54	0.54	4.05E-06	1.17E-03	<2.88E-06	0.38	<2.88E-06	<2.88E-06	7.39E-06	<2.88E-06	0.27
B (*M*)	1.80E-02	1.83E-02	<6.60E-06	6.39E-05	<6.60E-06	1.26E-02	<6.60E-06	<6.60E-06	<6.60E-06	<6.60E-06	8.67E-03
Ca (*M*)	4.48E-02	4.67E-02	2.53E-05	9.66E-05	3.72E-06	3.31E-02	<1.78E-06	2.76E-05	<1.78E-06	<1.78E-06	2.16E-02
Cl (*M*)	4.09E-02	4.34E-02	4.15E-03	<4.99E-04	<4.99E-04	2.47E-02	<4.99E-04	<2.49E-04	<4.99E-04	<4.99E-04	1.90E-02
Cs (*M*)	9.78E-04	1.65E-06	1.79E-07	1.02E-07	1.05E-07	8.88E-07	1.38E-07	4.97E-07	1.75E-08	6.91E-08	1.20E-06
Cr (*M*)	4.76E-03	4.58E-03	5.65E-07	1.05E-05	5.65E-07	3.28E-03	3.23E-07	<1.62E-07	4.04E-07	<1.62E-07	2.22E-03
F (*M*)	0.26	0.24	<1.51E-04	<1.51E-04	<5.90E-04	0.24	<1.51E-04	<2.46E-04	<1.51E-04	<1.51E-04	1.18E-01
Fe (*M*)	2.30E-02	2.32E-02	2.82E-06	5.74E-05	5.08E-05	1.55E-02	1.50E-06	2.26E-06	3.38E-06	<4.89E-07	1.13E-02
Hg (*M*)	1.97E-03	2.10E-03	1.57E-03	1.40E-06	5.58E-06	5.53E-05	1.52E-05	3.16E-05	7.03E-07	3.99E-08	2.12E-06
K (*M*)	0.12	0.13	3.35E-06	2.71E-04	2.40E-06	9.00E-02	2.74E-05	4.89E-06	3.22E-06	3.15E-03	6.22E-02
Mn (*M*)	7.76E-03	7.77E-03	<2.29E-07	1.73E-05	<2.29E-07	5.41E-03	<2.29E-07	<2.29E-07	2.29E-07	<2.29E-07	3.76E-03
Mo (*M*)	1.25E-03	1.26E-03	1.75E-05	9.98E-06	1.45E-03	2.95E-05	<1.97E-07	<1.97E-07	<1.97E-07	3.28E-07	2.16E-05
Na (*M*)	1.24	1.29	0.50	1.62E-03	4.29E-05	0.87	5.18E-06	0.36	5.22E-04	2.22E-03	8.40E-01
NO_3 (*M*)	4.46	4.46	---	---	---	---	---	---	---	---	---
Ni (*M*)	1.83E-03	1.77E-03	1.14E-06	4.19E-06	7.16E-07	1.30E-03	<5.01E-07	<5.01E-07	7.87E-07	<5.01E-07	8.87E-04
Pb (*M*)	9.25E-04	9.44E-04	1.68E-06	1.97E-06	3.35E-06	6.20E-04	4.44E-04	2.90E-06	<5.17E-07	3.68E-05	5.17E-07
PO_4 (*M*)	1.00E-02	1.00E-02	---	---	---	---	---	---	---	---	---
Sr (*M*)	9.91E-04	1.06E-03	<1.44E-07	2.20E-06	<1.44E-07	7.29E-04	<1.44E-07	<1.44E-07	<1.44E-07	2.64E-04	7.67E-07
SO_4 (*M*)	0.0386	0.0386	---	---	---	---	---	---	---	---	---
Zr (*M*)	1.43E-03	1.28E-03	1.84E-06	1.31E-06	4.46E-04	6.47E-04	<2.99E-07	1.46E-05	<2.99E-07	<2.99E-07	4.50E-04
Eu (*M*)	7.02E-03	7.01E-03	2.90E-05	2.21E-07	9.10E-03	<4.15E-08	9.67E-08	<4.15E-08	<4.15E-08	<4.15E-08	<4.15E-08
Re (*M*)	3.58E-03	3.54E-03	7.34E-04	3.86E-06	1.55E-03	1.09E-03	2.84E-05	2.54E-05	8.11E-06	3.63E-04	2.95E-05
Ba (*M*)	1.09E-04	1.07E-04	<1.99E-07	2.75E-07	<7.65E-08	7.82E-05	<7.65E-08	1.99E-07	<7.65E-08	1.83E-05	2.14E-05
Ag (*M*)	4.12E-05	4.48E-07	<2.73E-07	<2.73E-07	<2.73E-07	6.04E-07	<2.73E-07	<2.73E-07	<2.73E-07	<2.73E-07	<2.73E-07
Se (*M*)	2.87E-05	2.93E-05	<1.65E-06	<1.65E-06	<1.65E-06	2.05E-05	<1.65E-06	<1.65E-06	<1.65E-06	<1.65E-06	1.62E-05
Ru (*M*)	1.94E-05	2.04E-05	1.81E-06	<8.73E-07	<8.73E-07	1.29E-05	---	---	---	---	---
Rh (*M*)	1.00E-06	1.94E-06	<1.00E-06	<1.00E-06	<1.10E-06	2.39E-06	<1.00E-06	<1.00E-06	<1.00E-06	<1.00E-06	2.67E-06
Pd (*M*)	<6.31E-07	<6.31E-07	<6.31E-07	<6.31E-07	<6.31E-07	1.56E-06	<6.31E-07	<6.31E-07	<6.31E-07	<6.31E-07	<6.31E-07
Volume (L)	40.1	40.1	37.0	4.2	32.7	56.6	66.5	12.8	31.0	142.1	68.1

Table A- 21. Concentrations of feed components in the combined HAW and LAW streams.

					Normalized	
Component	Total HAW	Total LAW	% in HAW	% in LAW	% in HAW	% in LAW
Al (*M*)	<2.88E-06	8.39E-02	<0.002	85.1	<0.003	>99.997
B (*M*)	<6.60E-06	2.69E-03	<0.2	81.9	<0.2	>99.81
Ca (*M*)	<2.14E-06	6.71E-03	<0.02	82.0	<0.03	>99.97
Cl (*M*)	<4.99E-04	6.83E-03	<5.3	91.5	<5.5	>94.51
Cs (*M*)	7.59E-08	4.79E-07	99.73	0.3	99.7	0.27
Cr (*M*)	<2.37E-07	6.90E-04	<0.02	79.4	<0.03	>99.97
F (*M*)	<2.33E-04	3.67E-02	<0.4	77.4	<0.5	>99.50
Fe (*M*)	<9.89E-06	3.51E-03	<0.2	83.7	<0.2	>99.78
Hg (*M*)	1.08E-06	2.71E-04	0.2	75.4	0.3	99.68
K (*M*)	2.56E-03	1.93E-02	9.5	89.8	9.5	90.45
Mn (*M*)	<2.29E-07	1.17E-03	<0.01	82.2	<0.02	>99.98
Mo (*M*)	2.72E-04	9.94E-06	95.3	4.4	95.6	4.4
Na (*M*)	1.81E-03	0.37	0.4	93.3	0.4	99.61
NO_3 (*M*)	---	---	---	---	---	---
Ni (*M*)	<5.41E-07	2.76E-04	<0.1	82.3	<0.2	>99.84
Pb (*M*)	3.05E-05	1.35E-04	14.4	79.9	15.3	84.74
PO_4 (*M*)	---	---	---	---	---	---
Sr (*M*)	2.14E-04	<3.76E-07	94.3	<0.2	99.8	<0.2
SO_4 (*M*)	---	---	---	---	---	---
Zr (*M*)	<8.37E-05	1.41E-04	25.5	53.8	32.1	67.9
Eu (*M*)	<1.70E-03	4.95E-06	105.7	0.4	99.6	0.4
Re (*M*)	5.86E-04	1.44E-04	71.2	22.0	76.4	23.6
Ba (*M*)	<1.49E-05	6.71E-06	59.4	33.6	63.9	36.1
Ag (*M*)	<2.73E-07	<2.73E-07	<2.9	<3.6	---	---
Se (*M*)	<1.65E-06	<6.15E-06	<25.0	95.7	<20.7	>79.3
Ru (*M*)	---	---	---	---	---	---
Rh (*M*)	<1.02E-06	1.52E-06	<444.3	454.0	<49.5	>50.5
Pd (*M*)	<6.31E-07	<6.31E-07	<436.0	<547.5	---	---
Volume (L)	174.8	219.6	---	---	---	---