

DEMONSTRATION OF CESIUM REMOVAL TECHNOLOGIES USING HIGH-LEVEL WASTE IN SUPPORT OF THE SALT PROCESSING PROJECT AT THE SAVANNAH RIVER SITE

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ABSTRACT

The Department of Energy established the Salt Processing Program (SPP) at the Savannah River Site to develop and implement technologies for the treatment of the stored salt (i.e., soluble) portion of the High Level Waste (HLW) at that site. The SPP encompasses the selection, design, construction and operation of treatment technologies to prepare the salt waste feed material for the site's Saltstone Facility and vitrification facility (Defense Waste Processing Facility {DWPF}). The selected technologies must remove the majority of the actinides, strontium, and cesium from the soluble waste and transfer these constituents to the vitrification facility.

The Program began investigation of three principal technologies for the removal of cesium in 1998. One approach uses an inorganic sorbent, crystalline silicotitanate, to capture the cesium in a non-elutable ion exchange approach. A second process option uses a specially designed crown-ether extractant deployed within an organic solvent extraction system. The third option uses sodium tetraphenylborate to precipitate the cesium with subsequent removal by cross-flow filtration.

A key evolution in the evaluation of the technologies and their relative performance involved demonstrations of the process chemistry using actual high-level waste samples. Demonstrations occurred at the Savannah River Technology Center between February 1999 and May 2001. The work required design, fabrication, and installation of test equipment costing in excess of \$1 million. Testing used more than 125 liters of raw waste samples collected in three major site evolutions, representing the most challenging concurrent test efforts within the past decade for the remote cells at SRTC. Total cost of the completed work exceeded \$2.7 million. This report provides an overview of those research findings and examines the efficiency of the processes.

INTRODUCTION

The Savannah River Site designed and constructed the In-Tank Precipitation process to remove cesium from the stored High-Level Waste. In 1995, batch processing of tetraphenylborate in Tank 48H started.(1) However, extensive production of benzene during processing led to the cessation of operations. The Department of Energy directed the site's operating contractor to begin a systems engineering study to select and demonstrate a replacement process and

eventually transferred management of the technical program for the research program to the Tanks Focus Area.

The selection process identified three technologies for the cesium removal portion of the process. One approach uses sodium tetrphenylborate to precipitate the cesium with subsequent removal by cross-flow filtration. A second process option uses an inorganic sorbent, crystalline silicotitanate, to capture the cesium in a non-elutable ion exchange approach. The third option uses a specially designed crown-ether extractant deployed within an organic solvent extraction system. For a full description of the proposed process, consult the detailed engineering overview by Washburn et al.(2) More extensive reports provide the full details of the experimental demonstrations using actual waste.(3,4,5) This report summarizes the three efforts.

Precipitation Process

The precipitation process uses the tetraphenylborate chemistry (6) originally deployed by the Savannah River Site using compact, continuous engineering equipment to provide a more robust safety envelop. Numerous demonstrations of the technology occurred using simulated wastes to establish the necessary design parameters for the demonstration with actual waste.(7,8,9) A previous demonstration of the operation of the precipitation reaction with material from the Savannah River Site (SRS) tank farm occurred in 1999, but failed to achieve steady-state performance due to extensive foaming.(10) Hence, the Savannah River Technology Center (SRTC) conducted an additional test using actual waste to demonstrate sustained decontamination of waste, balanced hydraulics, and reduced foaming. The experiment also measured the degree of catalytic decomposition of the organic induced by the trace metals present in the waste.

Ion Exchange Process

The ion exchange process uses crystalline silicotitanate (CST) particles in the engineered form (IONSIV[®] IE-911, made by UOP LLC, Molecular Sieves Division, Des Plaines, IL)(11). Previous testing with simulated waste solutions demonstrated the feasibility of the ion exchange process.(12,13) The demonstration with actual waste sought to verify cesium removal performance, establish the reliability of predicted mass transfer zone lengths (e.g., column dimensions), monitor gas formation and collection within the column, and examine the stability of the sorbent after prolonged contact with waste.

Solvent Extraction Process

The solvent extraction process uses a novel solvent consisting of a calix[4]arene-crown-6 extractant dissolved in an inert hydrocarbon matrix.(14) An added modifier, an alkylphenoxy alcohol, enhances the extraction power of the calixarene and limits the formation of a third phase. An additional additive, tri-octyl amine, improves stripping performance and mitigates the effects of any surfactants present in the feed stream.(15) A number of laboratory studies established the stability of the solvent system in the presence of the chemicals in the SRS high-level waste tanks and under radiation fields. A continuous experiment with simulated SRS waste

proved effective.(16) The demonstration with actual waste explored whether trace components in the waste detracted from cesium removal efficiency, determined stage performance efficiency, examined the hydraulics, determined the influence of temperature on operation, and verified that the process provided the necessary volume reduction for the recovered cesium stream.

EXPERIMENTAL DESIGN AND DETAILS

Each process demonstration required design, fabrication, and installation of complex equipment in biologically shielded cells for remote operation. Figure 1 shows the major processing equipment used in each experiment. In addition to these racks containing the main process vessels, the experiments required ancillary tanks, pumps, and instrumentation as described elsewhere.(3,5,4) For the more complex precipitation and solvent extraction experiments, the equipment included an automated data acquisition and control system. The ion exchange test, conducted earliest, relied upon manual control and data collection.



Fig. 1. Main processing equipment for the demonstrations, from left to right: precipitation, ion exchange, and solvent extraction.

The research and management philosophy for each demonstration incorporated preliminary verification of the chemistry with batch tests to verify cesium removal performance. Also, personnel demonstrated the adequacy of the equipment using simulated wastes and altered the designs for lessons learned. This report will not provide the details of those preliminary results.

Precipitation Demonstration

The waste used in this demonstration represented a blended mixture of samples from 13 of the 49 storage tanks at SRS. Personnel selected samples from these tanks to enhance the amount of noble metals in the feed since these elements can catalyze decomposition of the organic. The composite waste includes samples from the same source tanks as used in the previous demonstration (10) and known to foam when treated with tetraphenylborate and mixed under adverse conditions. Use of waste from so many of the available tanks also ensured that the demonstration included all general waste sources for the site. Hence, the waste provided as challenging a composition as available. Table I notes the principal constituents in the waste.

Table I. Composition of waste.

| Component | Concentration | Component | Concentration |
|---|----------------------|--|----------------------|
| Cesium | 0.000814 M | Mercury | $\leq 5E-08$ M |
| Sodium | 10.3 M | gamma scan | 8.15E+09 dpm/mL |
| Potassium | 0.0873 M | Nitrate (NO ₃ ⁻) | 0.0191 M |
| ⁹⁰ Sr | 5.57E+06 dpm/mL | Nitrite (NO ₂ ⁻) | 0.0155 M |
| ²³⁸ Pu + ^{239/240} Pu | 1140 dpm/mL | Sulfate (SO ₄ ²⁻) | ≤ 0.000521 M |
| ²³⁸ U + ²³⁵ U | 3.84 mg/L | Hydroxide (HO ⁻) | 6.19 M |

The demonstration used two continuous stirred tank reactors, both blanketed with flowing nitrogen to maintain an oxygen content in the vapor space below 4.5 vol %, a value previously shown to promote catalytic decomposition of the tetraphenylborate. Each reactor held a working volume of 1 L of slurry. Personnel controlled flow rates of the various streams to achieve a bulk average sodium concentration in the reactors of 4.46 M, or ~5% below the nominal value for the process. Dilution of the waste occurred chiefly through addition of a solution containing sodium tetraphenylborate. Personnel controlled the amount of tetraphenylborate added to achieve a 60% stoichiometric excess of that needed to achieve precipitation of the cesium and potassium in the waste.

Personnel also added monosodium titanate in a semi batch style so as to maintain an average concentration of 0.4 g/L in the first reactor. The monosodium (MST) titanate served to remove strontium and alpha-emitting actinides, another requirement for the treatment of SRS waste. All three demonstrations included treatment of waste with MST. However, only this process includes simultaneous treatment of the waste to remove actinides, strontium, and cesium.

Personnel added a proprietary additive designed to control foaming in these tetraphenylborate slurries. The additive consists of bis-2(ethylhexyl) sulfosuccinate combined with a diluent. Addition occurred continuously into the both reactors to achieve a target concentration of 500 ppm.

The demonstration held the temperature at 25 °C for 60 hours. Personnel then raised the temperature to 45 °C and continued to operate for another 32 hours. Then, personnel stopped flows and held the contents of the reactors at temperature for another week to observe any catalyzed decomposition of the organic.

Ion Exchange Demonstration

Table II shows the composition of the waste selected for the demonstration of the ion exchange process. The waste contained an elevated free hydroxide concentration that requires a relatively short mass transfer zone (i.e., column length) near the 1.6 meters provided in the three sequential columns used in the experiment. The waste also contained a relatively high concentration of radioactive cesium to provide as high rate as practical for radiolytic generation of gases within the column.

Table II. Composition of Tank 44F radioactive waste.

| <u>Component</u> | <u>Concentration</u> | |
|---------------------------------------|----------------------|-----------------|
| | <u>As Received</u> | <u>Diluted*</u> |
| Na ⁺ (molar) | 15.0±.2 | 5.4 |
| K ⁺ (molar) | 0.15 | 0.051 |
| Cs ⁺ (mmolar) | -- | 0.351** |
| OH ⁻ (molar) | 11.8 | 4.3 |
| NO ₃ ⁻ (molar) | 1.19 | 0.37 |
| NO ₂ ⁻ (molar) | 1.22 | 0.35 |
| SO ₄ ²⁻ (molar) | -- | 0.001 |
| AlO ₂ ⁻ (molar) | 0.35 | 0.126 |
| PO ₄ ³⁻ (molar) | 0.007 | 0.004 |
| Cl ⁻ (molar) | -- | 0.009 |
| Formate (molar) | -- | <0.003 |
| Oxalate (molar) | -- | <0.001 |
| Cr (mg/L) | -- | 4.5 |
| Hg (mg/L) | -- | <1 |
| ¹³⁷ Cs (Ci/L) | 1.26 | 0.445 |
| ⁹⁰ Sr (μCi/L) | -- | 28 |
| ⁹⁹ Tc (mCi/L) | 0.14 | 0.054 |
| (mg/L) | 8.3 | 3.2 |
| ²³⁵ U (mg/L) | -- | <0.07 |
| (nCi/mL) | -- | <0.2 |
| U-238 (mg/L) | 1.3 | 0.42±.03 |
| (nCi/mL) | 0.44 | 0.14±.01 |
| ²³⁸ Pu (d/m/mL) | -- | 440±220 |
| (nCi/mL) | -- | 0.2±.1 |
| ^{239/240} Pu (d/m/mL) | -- | 50±50 |
| (nCi/mL) | -- | 0.02±.02 |
| Mass 239 (mg/L) | -- | <0.07 |
| Mass 240 (mg/L) | -- | <0.07 |
| Density (g/mL) | 1.496 | 1.201 |

* After the second dilution and before strontium/alpha removal and filtration.

**CsNO₃ added to adjust total cesium to the target concentration of 0.37 mM.

Prior to performing the ion exchange demonstration, personnel performed a batch treatment of the waste with MST to remove the strontium and actinides. Researchers added MST to the Tank 44F waste, stirred the slurry for 24 hours, and sampled before proceeding with filtration. Filtration used a 0.45 μ nominal pore size, polytetrafluorethylene membrane to separate the waste solution from the MST solids.

The ion exchange demonstration processed 75 L of pretreated waste over 6.5 days. The test maintained a waste flow rate of 9.4 ± 0.2 mL/min for a superficial velocity of 5.3 cm/m. Testing occurred at 31 ± 2 °C. Personnel collected samples from the column at lengths of 10, 85, and 160 cm from the inlet analyzing for cesium content. Installed instrumentation allowed monitoring of pressure drop across the bed and the use of a glass column allowed visual observation for trapped gas.

Solvent Extraction Demonstration

Personnel prepared a composite waste from nearly equal volumes of material from Tanks 37H and 44F to demonstrate the solvent extraction process. The waste composition proves typical of tank supernatants as shown in Table III. Crystallization of the less soluble inorganic salts leaves the waste highly concentrated in sodium hydroxide. In these solutions the free hydroxide accounts for half or more of the total sodium concentration. Dilution with 2 M NaOH also contributes to the high hydroxide concentration in the final waste solution. After dilution, the Tank 37H/44F composite contained 3.9 M free hydroxide compared to 2.0 M in average waste. Nitrate (0.65 M) concentration remained correspondingly low compared to the expected average concentration (1.7 M). Cesium also tends to concentrate in such waste as reflected in the high ^{137}Cs activity of the undiluted sample (5.5×10^9 d/m/mL or 0.7 Ci/L) compared to that of average waste (0.37 Ci/L). Potassium ion (0.037 M), which competes with cesium during extraction, also proved higher than average (0.014 M). The high concentration of these two components makes decontamination of the Tank 37H/44F composite more challenging than average waste.

Personnel pretreated the waste in the same batch contacting process as used for the ion exchange demonstration. The solvent extraction demonstration followed using 33 stages of 316 stainless steel, 2-cm annular centrifugal contactors. The equipment configuration included one solvent wash stage using 0.01 M NaOH, 15 extraction stages, two scrub stages using 0.05 M HNO₃, and 15 strip stages using 0.001 M HNO₃. Control of relative flows resulted in an organic-to-aqueous ratio of 0.33 in the extraction stages and of 5 in the strip stages providing an overall volume concentration factor for the product cesium stream near 15 for the process. Instrumentation also allowed controlling the temperature within the extraction section below 25 °C with the first strip stages held above 28 °C.

The demonstration processed 106 L of waste during 48 hours of contractor operation. Personnel collected samples to demonstrate stage efficiency and monitor cesium removal performance. Visual observations allowed determination of the degree of organic entrainment that occurred in the extraction and stripping sections of the process. Installed instruments allowed researchers to monitor flow rates and perform mass balances to ensure the organic-to-aqueous ratios remained near the target values. Three hydraulic disruptions occurred during testing as personnel tried to meet the volume concentration factor of 15 for the demonstration. In each case, personnel quickly restored the system to steady state operation with limited delays operating 74% of the available time.

Table III. Composition of the Tank 37H/44F composite sample.

| Concentration | | Concentration | |
|--|-----------------------|-------------------------|------------------------|
| <u>Component</u> | <u>(molar)</u> | <u>Component</u> | <u>(mg/L)</u> |
| Na ⁺ (M) | 5.61 | Ag | <6 |
| K ⁺ (M) | 0.037 | Ba | <3 |
| Rb ⁺ (mM) | 0.092 | Ca | <20 |
| Cs ⁺ (mM) | 0.17 | Cd | <2 |
| | | Cr | 31 |
| Free OH ⁻ | 3.9 | Mg | <1 |
| NO ₃ ⁻ | 0.65 | Pb | <30 |
| NO ₂ ⁻ | 0.51 | Sr | 0.048 |
| AlO ₂ ⁻ | 0.17 | | |
| SO ₄ ²⁻ | 0.002 | | |
| | | Concentration | |
| | | <u>Component</u> | <u>(d/m/mL)</u> |
| CO ₃ ²⁻ | 0.072 | ¹³⁷ Cs | 1.6E+09 |
| PO ₄ ³⁻ | 0.003 | ⁹⁰ Sr | 2.2E+05 |
| F ⁻ | <0.01 | ⁹⁹ Tc | 2.5E+05 |
| Cl ⁻ | 0.012 | ²³⁸ Pu | 1.7E+03 |
| Oxalate | <0.01 | ^{239/240} Pu | <1.2E2 |
| Formate | <0.02 | | |
| | | Concentration | |
| | | <u>Component</u> | <u>(mg/L)</u> |
| Table shows composition after dilution with 2M NaOH to achieve 5.6 M Na ⁺ . Analyses performed on unfiltered samples. | | M-235 | 0.074 |
| | | M-236 | <0.003 |
| | | M-237 | <0.003 |
| | | M-238 | 3.9 |
| | | M-239 | <0.003 |
| | | | |
| | | Density (g/mL) | 1.217 |
| | | PH | >14 |

RESULTS AND DISCUSSION

Precipitation Demonstration

The demonstration of the precipitation process met all the design objectives.

The experiment demonstrated successful removal of cesium from the waste. This waste required a decontamination factor (DF) of 10,000 to meet process requirements. Figure 2 shows the cesium removal performance. During the entire experiment, the decontamination factor averaged 1.4 million after the equipment reached steady-state operation.

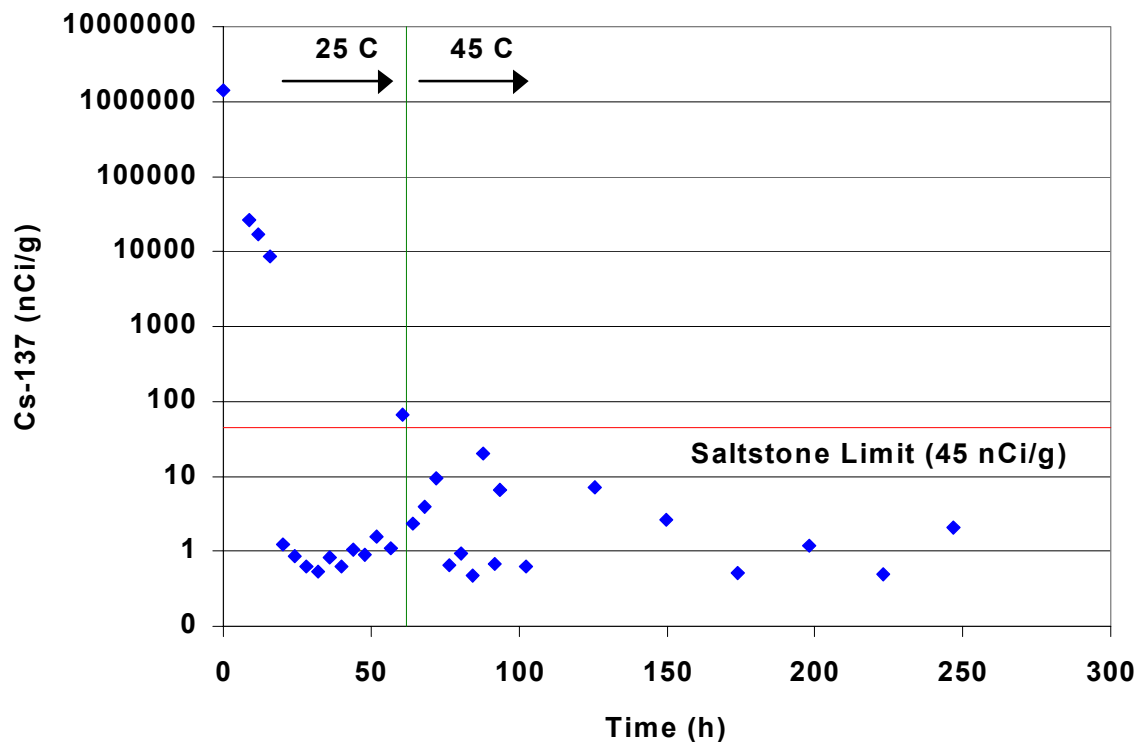


Fig. 2. Cesium removal performance from precipitation demonstration.

The monosodium titanate successfully removed the actinides (Pu, U) from solution yielding a solution with an average alpha dose below 0.214 nCi/g after treatment versus a process requirement of 18 nCi/g. Raising the temperature did not change the actinide removal performance.

The experiment demonstrated successful removal of strontium from the waste. This waste required a target DF of 19.3 to meet Saltstone requirements. During the entire experiment, the

decontamination factor averaged 45.6 after the equipment reached steady-state operation. The increase in temperature did not influence the strontium removal efficiency.

Throughout the duration of the testing we observed little, if any, evidence of foaming. We observed some minor frothing in the sample lines before reaching 45 °C, and the inter-stage overflow tube between the reactors plugged after 71 hours of operation. However, use of an inter-stage pump, included in the design for just such a contingency, allowed completion of the testing without further adverse hydraulic performance.

The slurry showed evidence of catalytic decomposition of the tetraphenylborate during the testing at 45 °C, giving a calculated benzene generation rate of 2.4 mg/L/h. This rate of reaction falls well within the allowable values for the process.

Ion Exchange Demonstration

The demonstration of the crystalline silicotitanate process met all the design objectives.

The IONSIV[®] IE-911 effectively removed cesium from the SRS radioactive waste as shown in Figure 3. The 1.5-cm diameter x 160-cm long column removed more than 99.999% of the cesium in 75 liters of diluted Tank 44F waste. All of the treated waste met Saltstone process requirements. The radioactive cesium loading reached 376 Ci/L on the loaded IONSIV[®] IE-911, producing an estimated dose rate of 0.12 Mrad/h or 15% of that expected in process operations.

The cesium removal exceeded predictions through most of the test at sampling points located 10, 85, and 160 cm down the column. Removal after 10 cm exceeded predictions for the first 50 hours of the test and lagged the prediction for the remainder of the test. Filtration removed the majority of the Cs-137 found after 85 and 160 cm of column length, suggesting the radioactivity resulted from fines from the IONSIV[®] IE-911.

Gas generated due to radiolysis from the absorbed cesium did not remain as bubbles in the column during loading (i.e., when liquid flowed through the column). At the end to the test, personnel terminated flow and gas bubbles accumulated at a rate of 0.034 mL/h.

Leaching and precipitation of a niobium from the IONSIV[®] IE-911 posed a problem with column plugging. During sodium hydroxide pretreatment of the packed column, the leached material plugged the test column. Personnel removed the blockage by back flushing the column. (Subsequent research efforts and coordination with the vendor resulted in a revised manufacturing process that dramatically reduces the leaching of niobium.)

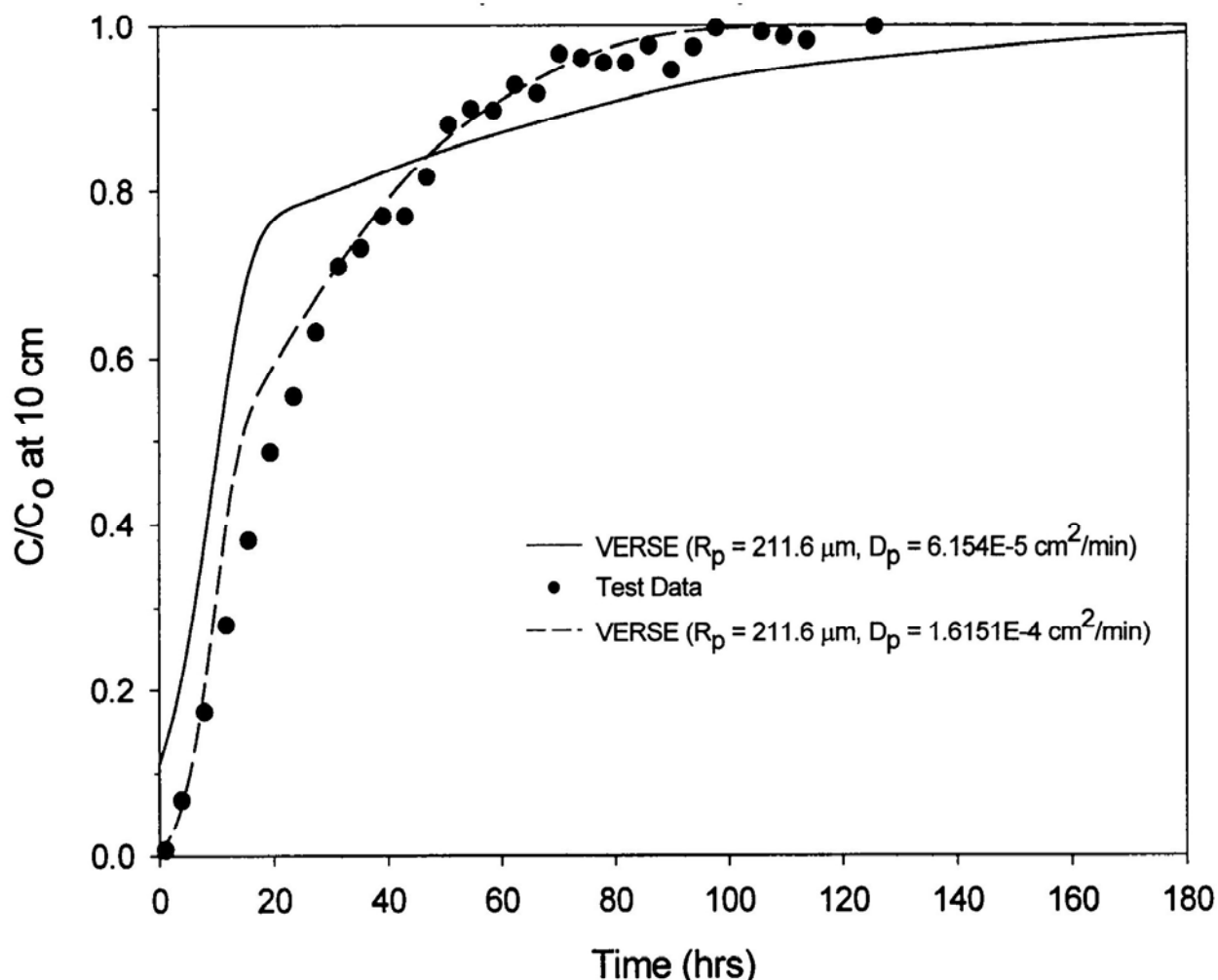


Fig. 3. Cesium removal performance from crystalline silicotitanate demonstration.

The strontium and actinide removal efficiency met process requirements although the poor mixing conditions in the batch contacting yielded poorer than expected performance.

Solvent Extraction Demonstration

The demonstration of the solvent extraction process met all the design objectives.

The process effectively removed cesium from the waste to concentrations below the Saltstone process requirement of 45 nCi/g as shown in Figure 4. (As with the demonstration of the other processes, researchers also performed experiments with simulated wastes prior to the work with actual waste samples. Figure IV also shows performance data for two of those demonstrations.) Decontamination Factors (DFs) reached as high as 2 million during stable hydraulic conditions in the 2-cm contactor apparatus. The composite waste DF averaged 511,000 over the first 34 hours and 40,000 for the entire 48-hour test. The test successfully stripped the solvent of cesium with an average solvent DF of 154,000 for the entire test.

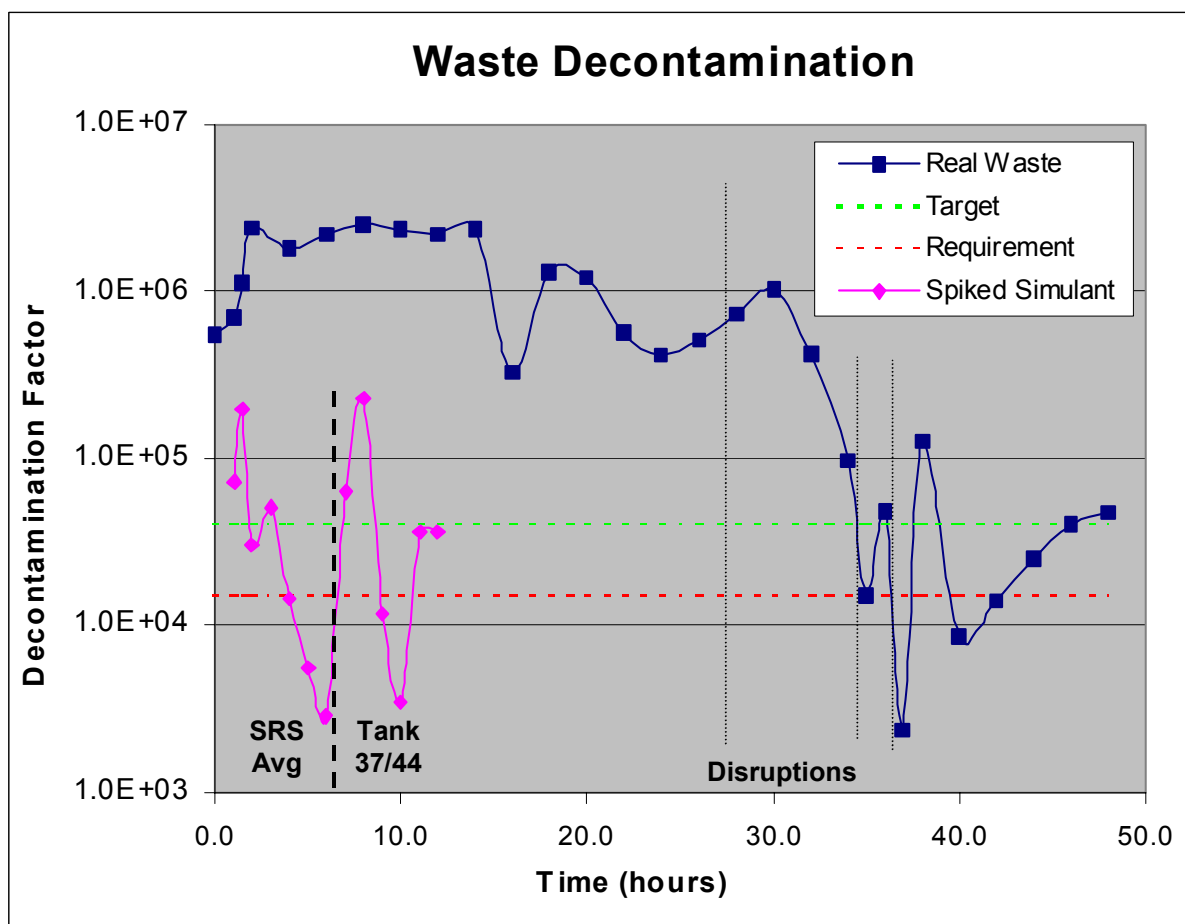


Fig. 4. Cesium removal performance from Caustic Side Solvent Extraction Demonstration.

The tests demonstrated hydraulic stability of the contactor array over a range of process conditions. Table IV provides the demonstrated extraction and strip section stage efficiencies of greater than 80% for all tests.

| <u>Feed</u> | Table IV. Stage Efficiencies | |
|--|--------------------------------------|---------------------------------|
| | <u>Extraction Stage Efficiencies</u> | <u>Strip Stage Efficiencies</u> |
| Mockup | 86 ± 6% | 96 ± 6% |
| Spiked Simulated Tank 37H/44F Waste | 94 ± 6% | 82 ± 6% |
| Tank 37H/44F Waste | 84 ± 4% | 82 ± 4% |

Hydraulic capacity of the 2-cm contactor apparatus proved 10% higher with simulated waste compared to either Tank 37H/44F simulated or high level waste. This difference in performance occurred due to differences in viscosity and density between the two waste compositions. The planning failed to account for these variances and, hence, the experiments included three hydraulic interruptions as researchers increased flows during the demonstration beyond the

limitations of the equipment. Nevertheless, researchers demonstrated the ability to recover from process disruptions and achieve the required decontamination levels.

SUMMARY

The three demonstrations successfully explored the operation of the processes allowing evaluation of each option against the performance requirements for treatment of high level waste at the Savannah River Site. Each process used available waste samples selected to pose as great a challenge as practical for the underlying chemistry. All three technologies satisfied the operational requirements. Subsequently, the Department of Energy conducted a comparative assessment of the entire research and development effort for each process option. In conducting the assessment, the Department also solicited and received opinions from the Defense Nuclear Facility Safety Board and from a special committee established by the National Research Council of the National Academy of Sciences. The Department identified the solvent extraction technology as the preferred technology, citing its robust nature and the well-developed expertise in deploying solvent extraction within nuclear facilities.(17)

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