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SEPARATION PROJECTS WITHIN THE U.S. DEPARTMENT OF ENERGY'S UNDERGROUND STORAGE TANK — INTEGRATED DEMONSTRATION

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ABSTRACT

The greatest technical and financial challenge facing the U.S. Department of Energy is the remediation of the 1×10^8 gal of high-level and low-level radioactive waste in the underground storage tanks (USTs) at its Hanford, Savannah River, Oak Ridge, Idaho, and Fernald sites. With current technologies, this remediation is estimated to cost at least 100 billion dollars. In an effort to reduce costs, improve safety, and minimize delays, the Underground Storage Tank—Integrated Demonstration was created for demonstration, testing, and evaluation of promising new technologies that can be used for UST remediation. These UST—ID demonstrations, which are typically at the pilot-plant scale, are performed by the Characterization and Waste Retrieval Program or by the Waste Processing and Disposal Program (WPDP). The results from these demonstrations will help to determine which processes will be used in the full-scale remediation of the USTs. This paper presents the current technical status of the WPDP projects that utilize separation technologies. These WPDP programs focused on three areas, which are the treatments of supernate and sludge as well as the destruction of nitrates/organics.

INTRODUCTION

The production of nuclear materials has been the primary mission of the U.S. Department of Energy (DOE) and its predecessor agencies for the past 5 decades. The DOE production facilities and laboratories have generated a considerable amount of radioactive and mixed wastes. Over 1×10^8 gal of waste are stored in 334 underground storage tanks (USTs) at Hanford, Savannah River, Oak Ridge, Idaho, and Fernald. The waste contains a wide variety of hazardous and nonhazardous chemical components such as sodium salts, acids, iron oxides, aluminum oxides, and heavy metals. Approximately half of the waste is composed of $NaNO_3$ and $NaNO_2$. The radionuclides are primarily transuranic (TRU) elements, ^{137}Cs, and ^{90}Sr. The temperatures of the waste range from near ambient to 93°C, and some tanks occasionally contain potentially explosive gas mixtures. Tank void-space radiation can be as high as 10,000 rad/h.

In 1989, the Office of Environmental Restoration and Waste Management (DOE—EM) was established to coordinate and manage the remediation, waste minimization, and environment compliances activities for the DOE sites. DOE—EM's Office of Technology Development (OTD) is responsible for providing new technologies which can reduce costs, improve safety, and minimize delays of the UST remediation. The OTD has divided the evolution of the new technologies into two phases: research and development (R&D) and demonstration, testing and evaluation (DT&E). The R&D efforts are directed primarily by the Efficient Separations and Processing—Integrated Program. After the new processes have completed their R&D phases, the Underground Storage Tank—Integrated Demonstration (UST—ID) performs DT&E on the most promising technologies. These UST—ID studies are performed by the Characterization and Waste Retrieval Program (CWRP) or by the Waste Processing and Disposal Program (WPDP). The results of these demonstrations are impacting the selection of processes that will be used during the full-scale remediation of the USTs. This remediation effort includes the characterizations of tanks structures and their waste, retrieval of the liquid and solid waste from the USTs, separation of the radioactive components from the waste, and disposal in final waste forms that meet the acceptance criteria of the individual waste repositories.

The WPDP is conducting several demonstrations that utilize separation technologies to assist in UST remediation effort. The objectives for these WPDP projects are the following:

1. Demonstrate the removal of radionuclides and chemical toxicity from low-level waste (especially alkaline supernate processing). Low level wastes will not contain any appreciable amounts of TRUs, and the radiation levels will be sufficiently low that the disposal requirements should be minimized.

2. Demonstrate the removal of the constituents contributing to excess volume of high-level waste (HLW) from HLW sludges and acidic wastes. HLW comes from fuel processing and contains TRUs. Its high radiation levels will require special disposal arrangements.

3. Demonstrate technologies and strategies to maximize releasable or reusable fractions from the wastes.

4. Demonstrate technologies and strategies to minimize the requirements for pretreatment chemicals.

5. Develop improved waste forms to ensure compatibility of treated material with final waste forms.

These objectives and the WPDP strategic plan (1) were developed with assistance of the WPDP Steering Group, which is composed of senior advisors from several DOE laboratories.

RESULTS AND DISCUSSION

The current status of the WPDP projects will be discussed. These projects can be grouped into 3 technology areas: supernate processing, sludge processing, and nitrates/organics destruction.

SUPERNATE PROCESSING

Cesium—Extraction Compact Processing Unit (W. G. Richmond, Pacific Northwest Lab)

A compact processing unit (CPU) is a small, portable process unit that is designed to treat UST supernate at a rate of 2 to 5 gal/min. The current baseline alternative is a large centralized shielded-canyon structure. Since the CPU is the considerably smaller, treatment technologies can be deployed faster with a CPU. This program is responsible for the design and construction of a CPU for Cs removal from the UST supernate which will remove a significant portion of the radioactivity from the supernate. This separation will allow further processing to occur with a minimum of protective shielding. This CPU consists of four major subsystems: the containment system, the process system, the control system, and the process interface system. The principal design criteria (2) of the components for the Cs ion-exchange CPU are the following:

1. Capacity to process 1 × 10^6 gal of Hanford double-shell tank waste in 1 year.
2. Cs removal factor of 1 × 10^5.
3. Design that permits relocation using a construction crane and transport trailer.
4. Full compliance with all applicable federal and state regulations.

The CPU will filter the supernate in order to remove the solids, which will be transferred into a holding tank for alternative processing. The filtered supernate will be adjusted to optimum conditions for the ion-exchange process. The adjusted waste will then be pumped through 3 ion-exchange columns in series in a carousel arrangement to remove the Cs from the waste. This Cs-free waste will be returned to the tank farm. The CPU is currently planning to use the resorcinol-formaldehyde resin (3) that was developed at the Savannah River Technology Center (SRTC). The Cs-loaded resorcinol-formaldehyde resin can be regenerated using dilute HNO_3 to the remove the Cs. The waste with the high Cs concentration will be neutralized and will become a waste feed stream for the HLW vitrification process which will produce a borosilicate glass as the final waste form.

During FY 1994, this project has finalized the design of the Cs-extraction CPU. The first step in the Cs-extraction CPU procurement process was completed. A Commerce Business Daily announcement was issued to identify contractors with an interest in the Cs-extraction CPU Design/Fabrication contract.

Cesium-Extraction Testing (J. P. Bibler, Westinghouse Savannah River Company)

The primary mission of this project is to study the ion exchange properties of resorcinol-formaldehyde resin in the support of the Cs-extraction CPU project. This resin has shown promise in Cs removal from the high-caustic, high-sodium, radioactive wastes that are stored at Hanford, Savannah River, and Oak Ridge (OR). A 10-mL column with the resin was tested with a simulant of Tank 101-AW at Hanford. The new results are more conservative than the data from the earlier study that used 2-mL columns. Fifty percent breakthrough occurred at approximately 150 column volumes, which is comparable to the breakthrough with the 2-mL columns. Essentially 100% of the ^{137}Cs was eluted with a maximum of 20 column volumes of dilute HNO_3. In addition, resorcinol resin was used to process OR simulated supernate. 50% breakthrough occurred at 30 column volumes. It is believed that this low number of column volumes is due to the high potassium concentration in the OR feed. Currently, preparations are underway to test the resorcinol resin with real Savannah River Site supernate in a radioactive cave at the SRTC. Studies have shown that the resin is resistant to radiation up to 5×10^8 R.

Another key role of this project is to supervise resin degradation research at Clark Atlanta University. The objectives of this study are to determine the mechanism of degradation and possible ways to prevent this degradation during storage. In storage, the resorcinol resin has a shelf life of approximately 3-5 years. Resorcinol was subjected to a variety of atmospheres such as N_2, O_2, H_2O, and O_3 in an effort to induce degradation. Nuclear magnetic resonance spectroscopy, Fourier transform infrared spectroscopy, and electron spin resonance spectroscopy were used to examine the resin. Preliminary results indicate that the storage of the resin under a nitrogen atmosphere can prevent degradation due to oxygen attacks.

SKID Demonstration of Resorcinol (M. L. Meyer, Westinghouse Savannah River Company)

This project will conduct a large-scale demonstration of the resorcinol resin's Cs removal capabilities using the skid-mounted ion exchange demonstration (SKID) unit at the Westinghouse Savannah River Company. The SKID unit consists of 3 columns, ancillary valves, pumps, lines, and computer controls. Each ion-exchange column has an empty volume of 219 gal and a void volume of 142 gal when the resin is in place. A resin slurry tank is being added to the SKID unit to remove resin fines and to slurry the resin into the columns. The SKID unit can operate in an once-through mode as well as a regenerative mode. FY 1994 efforts have primarily focused on the installation of the SKID unit.

After the completion of the installation, H_2O tests will be conducted to study the hydraulic and pumping properties of resorcinol. This study will include slurring the resin in and out of the columns using process H_2O. Particle-size studies will monitor for any physical degradation of the resin due to transport to and from the column. Additional information will be obtained on the ability to transport the resin as a slurry and on the pressure drops across each column with the resorcinol resin in place. In early FY 1995, the Cs decontamination factors will be determined using simulants for the Oak Ridge Melton Valley Storage Tank (MVST), the Savannah River Site, and the Hanford tank 101-AW. In addition, the need for prefiltration will be examined using the Hanford simulant with simulated sludge solids. This project is directly supporting the Cs-extraction CPU and its proposed hot demonstration.

Mobile Evaporator and Concentrator (D. B. Chamberlain, Argonne National Lab)

The purpose of this program is to develop a mobile evaporator/concentrator CPU to remove the significant amount of H_2O that remains after the waste has been retrieved. Approximately 30 wt % of the UST waste inventory is H_2O. Retrieval of the UST waste will likely result in the addition of H_2O which could lead to a volume increase as high as three fold. While this additional water should not significantly impact the Cs-removal ion-exchange process, it may have detrimental effects on the thermal destruction processes for nitrates/organics. A considerable amount of energy in the thermal destruction processes will be used to boil the H_2O rather than to destroy nitrates and organics. In addition, dilute streams put an additional load on the wastes vitrifier. This project will develop a transportable, compact system to remove the majority of the water from the Cs-free supernate. This evaporator/concentrator system will be similar to the Cs-extraction CPU. The evaporator will remove the bulk of the H_2O, which can be recycled to support further retrieval operations and/or treated and discharged to a soil column if the release criteria have been met. Additional water removal will be completed by a concentrator that is specially designed to handle high solids concentration or slurries which can be directly sent to the equipment for nitrates/organics destruction.

The FY 1994 experiments will focus on the proof-of-principle for the mobile evaporator/concentrator. A diluted (1/3) simulated Hanford waste solution was tested in a laboratory-scale LICON evaporator. Prior to the dilution, this waste solution contained high concentrations of Na (10 M), Al (1 M), NO_3 (3.5 M), NO_2 (2.2 M), and OH (5.1 M) ions. 45 L of feed was evaporated to 11 L of concentrate in approximately 8.5 hours of operation which spanned three days. Therefore, a total concentration factor was greater than 4, and the boiling-point elevation was approximately 25°F (14°C). At the end of the test, the boiling temperature was 53°C at 676 mm Hg vacuum. The pH of the distillate was 9.5-11. No operational problems were observed. The tube bundles were examined at the end of the test; scale had not formed on the tubes, but a thin film (weighing 1.2 g) was observed. Additional experiments are being planned to further concentration this waste solution. In addition, the draft plan for the corrosion testing of candidate materials for the mobile evaporator CPU was issued. The corrosion testing procedures for materials selection were described. The first stage of testing will examine vapor/liquid junction and liquid immersion. Materials currently under consideration include 304L, 316L, AL-6XN, Incoloy 800, Inconel 625, Inconel 617, Incoloy 825, Inconel 690, Hastelloy G-30, and titanium. The corrosion rates in HNO_3, NaOH, and HNO_3-HF environments are being used as selection criteria. HNO_3-HF corrosion rates are of interest because CaF_2 may form some of the scale which will build up during evaporator operation. This scale is normally removed by flushing the unit with 1 M HNO_3.

Comprehensive Sludge and Supernate Processing (B. Z. Egan, Oak Ridge National Lab)

The primary mission of this demonstration is to develop the preferred sludge/supernate processing flowsheet. In addition, a realistic performance assessment for each individual process will be determined, and the operating results which are needed for the pilot-plant design will be generated. This program is examining the separation, washing, and the acid dissolution of the solids; the partitioning of the transuranic (TRU) components from the acid solutions; and the pretreatment/separations processing of the supernate and soluble portions. Even though individual processes are known to partition or destroy specific components, combinations of these processes have not been evaluated. Once the compatibilities of the various processes are known, the optimum combination for the supernate/sludge processes will be determined based on cost and waste minimization. In order to perform realistic evaluations, approximately

5 L of actual sludge/supernate waste from the MVST was successfully transferred to a hot cell. This demonstration is the first time that liter quantities of actual UST waste are being used for DT&E of waste remediation technologies. The MVST waste is similar to the waste at Savannah River and Hanford since the wastes from the three sites were primarily produced by the same chemical processes and stored at a high pH.

The supernate studies have measured the distribution coefficients and percent removal of Cs from several ion-exchange materials. These ion exchangers and (their percent removal of Cs) included granular Duolite CS-100 (16.0%), granular resorcinol-formaldehyde resin (69.5%), fine powder crystalline silicotitanate (77.4%), and granular $K_2[CoFe(CN)_6]$ (98.7%). In addition, composite microspheres that contain $Na_2[CoFe(CN)_6]$ or $K_2[CoFe(CN)_6]$ in titanium hydrogen phosphate and hydrous titanium oxide will be tested. Future tests will examine the capabilities of various sorbents to remove Sr and Tc.

SLUDGE PROCESSING

Comprehensive Sludge and Supernate Processing (B. Z. Egan, Oak Ridge National Lab)

The sludge studies of this comprehensive supernate/sludge project have focused on the dissolution of the sludge solids. A 44-gram sample of wet MVST sludge contained 1056 μCi of ^{137}Cs; this sample was washed sequentially twice with 0.16 M NaOH, once with 0.5 M HNO_3, and twice with 3 M HNO_3. The dilute caustic washes removed about 63 μCi of the ^{137}Cs from the solid sludge. The dilute acid wash removed approximately 14 μCi of ^{137}Cs and trace amounts of Co and Eu. The 3 M HNO_3 washes removed a total of 436 μCi of ^{137}Cs so that 543 μCi remained with the 19 g of solid. It was noted that a gelatinous precipitate appeared in the leachate from the first 3 M HNO_3 wash after it was left for several days. This gelatinous material is postulated to be a form of silicic acid. Over 90% of the ^{60}Co and ^{154}Eu was removed from the solids after completion of the washes. The remaining solids from these washes were mixed with 100 mL of 6 M nitric acid for 5 days. After mixing, the HNO_3 concentration was 5.69 M. This acid leach removed an additional 212 μCi of ^{137}Cs along with 1.44 μCi of ^{154}Eu and 3.2 μCi of ^{60}Co. At this point in the leaching study, the cumulative percentages of ^{137}Cs, ^{154}Eu, and ^{60}Co removed were 69%, 97%, and 92%, respectively. The amount of cesium remaining in the sludge solids was significant even after leaching with 6 M HNO_3.

Since the Cs release is proportional to the amount of dissolved sludge, the Cs must be chemically bound. Originally, Cs was thought to be easily stripped/separated from the sludge. Future sludge studies will optimize the sludge dissolution parameters such as temperature and solution/solid ratio.

TRUEX Model Development (G. F. Vandegriff, Argonne National Lab)

The Center for TRUEX Technology Development at Argonne National Laboratory is continuing to perform R&D in an effort to broaden the applicability of the TRUEX process for use on HLW and TRU waste streams. The TRUEX process (4) is a solvent extraction process that uses octyl (phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide (CMPO) as the key ingredient. The TRUEX process can effectively separate the TRU components from aqueous nitrate and chloride solutions of acid dissolved sludge. A successful separation greatly reduces the amount of TRU waste that must be processed by the HLW vitrification plant. Most of the resulting solutions from the TRUEX process can be disposed as a low-level waste.

The Generic TRUEX Model (GTM) has developed to increase the utility of the TRUEX process. The GTM is a tool for designing site- and feed-specific TRUEX flowsheets and for estimating the space and cost requirements for installing a TRUEX process (5). The GTM has been recently enhanced through improvements in the thermodynamic modeling and computer codes. Further enhancements to the GTM during FY 1994 include the following: (1) the number of aqueous species whose concentrations can be calculated have been increased from 146 to over 200, and (2) the accuracy of the distribution ratio calculations and solvent loading has been improved by including the extraction due to tributylphosphate.

During the remainder of FY 1994, this project is comparing the data from TRUEX studies by the Power Reactor and Nuclear Fuel Development Corporation of Japan and the Radiochemical Engineering Development Center (REDC) at ORNL with the predicted results from the GTM. This project will also use the results from these pilot-plant-scale demonstrations to refine and validate the GTM. In addition, this work will enhance our understanding of the solution, extraction, and dissolution chemistry of HLW components.

TRUEX Data Collection and Model Validation (L. K. Felker, Oak Ridge National Lab)

The REDC at ORNL has conducted the first full-scale demonstration of the TRUEX process. A TRUEX flowsheet was developed with the assistance of ANL staff. A bank of continuous contactors known as the Solvent Extraction Test Facility was used for the TRUEX demonstration. The feed was a solution containing gram quantities of Pu, Am, and Cm from the dissolution of Savannah River Mark 42 targets which are highly irradiated PuO_2 targets. The experimental results from the three test runs were compared with the predictions from the GTM using a stage efficiency of 85%. Additional information is provided by another paper in this journal (6).

Commissariat á l'Energie Atomique Interchange (R. T. Jubin, Oak Ridge National Lab)

The Commissariat á l'Energie Atomique (CEA) has long-term development program on the separation of long-lived radionuclides. Of special interest, CEA has developed the DIAMEX process, which uses diamides as the extractants for the actinides in an acidic waste stream. Technical interchange included the placement of an ORNL staff member at the CEA in early 1994 so the DIAMEX process (7) can be compared to similar DOE technologies such as TRUEX process. During the course of the interchange, ORNL researcher will be directly involved in three key R&D areas for the DIAMEX process. The first area is to develop a method to control or suppress the extraction of Mo(VI) by maloamides. The removal of Mo is important in the control of third phase formation. The second task focuses on the hydraulic problems that were observed during the hot demonstration of the DIAMEX process in the CYRANO hot cell. Flow problems occurred in the mixers settlers of the extraction bank. Therefore, the number of stages that could be utilized was limited. The third task will addressed the use of centrifugal contactors for the DIAMEX process. The rapid contactors will be evaluated as a means to possibly utilize kinetics effects to improve decontamination factors. This collaboration is beneficial to he CEA and DOE, and current discussions may lead to the assignment of a CEA researcher to a DOE laboratory in 1995.

NITRATES/ORGANICS DESTRUCTION

The projects removes (or separates) the nitrates/organics from the waste streams. The presence of nitrates/organics may adversely effect other separation process such Sr and TRU removal.

Nitrate to Ammonia & Ceramic (NAC) Process (A. J. Mattus, Oak Ridge National Lab)

Bench-scale feasibility studies (8) using Hanford and Oak Ridge tank waste simulants have demonstrated that a new low-temperature (50-90°C) process can convert 90 to 99% of the NO_3 to NH_3. This nitrate destruction/separation is necessary because nitrate causes leaching in some proposed wastes forms and produces off gas problems in other potential waste forms. The destruction of the nitrates significantly reduces the final waste volume. In the NAC process, Al powders or shot was used to convert alkaline, NO_3-based supernate to NH_3 and an alumina-silica-based ceramic solid. The final NO_3-free ceramic product was then calcined, pressed, and sintered. Actual MVST supernate has been used in the NAC process. Leach tests were performed on the ceramic pellets from this test, and the results indicate that ^{137}Cs, ^{134}Cs, ^{85}Sr, ^{90}Sr, ^{60}Co, and ^{57}Co are firmly fixed. In accordance with the U.S. Nuclear Regulatory Commission's ANS 16.1 test procedure, no Sr or Co was detected after 30 days of leaching. The leach index (negative logarithm of the diffusion coefficient) of ^{137}Cs is 13, and the ^{137}Cs leach rate is slowly decreasing with time. The NRC requires a leach index of greater than 6. The diffusion coefficients of ^{137}Cs in the ceramic pellets and in glass should be comparable.

The NAC project is currently examining waste-form properties such as Na leach rate. In addition, Florida International University (FIU) will be optimizing microwave processes for the drying, calcining, and sintering of the NAC ceramic product into a final waste form. FIU is also studying the nitrate conversion kinetics using a pilot scale system.

Finally, alternative compositions of the final waste forms are being evaluated. Synthetic NAC solids with additives for glass formation have undergone melting tests in crucibles to determine the presence of any off-gas from the melt. Observations indicate that a flowable glass can be prepared.

Calcination/Dissolution Process Development (S. A. Colby, Westinghouse Hanford Company)

Two full-scale tests at the Westinghouse Science and Technology Center have successfully demonstrated the feasibility of plasma-arc technology to calcine simulated

Hanford tank waste. The primary objective of these trials was to identify the combination of chemical dissolution and thermal processing that will destroy nitrates and organics. The major benefit of the plasma-arc process is the separation of the TRU waste into a relatively small volume.

The first continuous calcining test (9) used 290 gal of feedstock in 5 h. The feedstock simulated the HLW sludge in tank 101-SY at Hanford. Approximately 95% of the NO_3 and NO_2 were thermally destroyed. The off-gas products from this destruction were primarily N_2 and O_2 with low concentrations of NO_x, CO, and CO_2. The volume reduction from the feedstock to the final product was approximately 80%. Process power consumption was 1000 kw per gpm of feedstock. The final product consisted of 37-38% by weight NaOH, 38-44% Na_2CO_3, 16-22% $NaAlO_2$, and trace amounts of various heavy metal oxides and phosphates. After the final report on the second test is issued, this project will have successfully completed its demonstration phase. Finally, calcination is currently a reference process for the full-scale remediation plan at Hanford.

CONCLUSIONS

The greatest technical and financial challenge facing DOE is the remediation of the USTs. In an effort to reduce remediation costs, improve safety, and minimize delays, the WPDP has been conducting DT&E on new separation and treatment technologies. The WPDP separation projects for FY 1994 examined supernate and sludge treatment, and nitrates/organics destruction. The supernate projects were primarily concerned with the development of the Cs-extraction and evaporator/concentrator CPUs. The objectives of the sludge studies were to develop a complete system-level plan for handling sludge/supernate and to evaluate the TRUEX and DIAMEX solvent extraction processes for acidic TRU waste streams. The projects on nitrates/organics destruction include the calcination/dissolution process and the NAC process. The results of the WPDP projects will impact the selection of the processes for the full-scale remediation of the USTs.

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