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CESIUM REMOVAL FROM HIGH-pH, HIGH-SALT WASTEWATER USING CRYSTALLINE SILICOTITANATE SORBENT

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ABSTRACT

Treatment and disposal options for Department of Energy (DOE) underground storage tank waste at Hanford, Savannah River, Idaho National Engineering and Environmental Laboratory (INEEL), and Oak Ridge National Laboratory (ORNL) are limited by high gamma radiation fields that are produced by high concentrations of cesium in the waste. Treatment methods are needed to remove the cesium from the liquid waste and thus concentrate the cesium into high-activity, remote-handled waste forms. The treated liquids could then be processed and disposed of by more cost-effective means with less radiation exposure to workers.

A full-scale demonstration of one cesium removal technology has been conducted at ORNL. This demonstration utilizes a modular, mobile ion-exchange system and existing facilities for the off-gas system, secondary containment, and utilities. The ion-exchange material, crystalline silicotitanate (CST), was chosen on the basis of its effectiveness in laboratory tests. The CST, which was developed through a Cooperative Research and Development Agreement between DOE and private industry, has several advantages over current organic ion-exchange technologies. These advantages include (1) the ability to remove cesium in the presence of high concentrations of potassium, (2) a high affinity for cesium in both alkaline and acidic conditions, (3) physical stability over wide alkaline and acidic ranges, and (4) the elimination of large volumes of secondary waste required for regeneration of organic ion exchangers. Approximately 116,000 L of supernate was processed during the demonstration with ~1,142 Ci of ^{137}Cs removed from the supernate and loaded onto 265L (70 gal) of sorbent. The supernate processed had a high salt content, about 4 M $NaNO_3$, and a pH of 12 to 13. This paper discusses the results of the full-scale demonstration and compares these results with data from the laboratory tests.

INTRODUCTION

The Office of Science and Technology (OST) has established the Tank Focus Area (TFA) to manage and carry out an integrated national program of technology development for tank waste remediation. Department of Energy (DOE) underground storage tanks located at Hanford, the Savannah River Site (SRS), Idaho National Engineering and Environmental Laboratory (INEEL), and the Oak Ridge National Laboratory (ORNL) contain over 90 million gallons of high-level and low-level radioactive waste that

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must be remediated. Although the total volume is considered high-level waste (HLW), it is neither cost-effective nor practical to dispose of all of the waste to meet the requirements of the HLW repository program and the Nuclear Waste Policy Act[1] without some volume reduction. A large portion of the waste exists as a liquid solution (supernate) that contains soluble radionuclides, along with high concentrations of sodium and potassium salts. In general, the radionuclides constitute less than 5% of the waste, and cesium is the primary radionuclide contributing to the activity. As a result, the majority of the waste could be disposed of as low-level waste (LLW) if the radioactivity could be reduced below the Nuclear Regulatory Commission limits. The TFA Pretreatment Functional Area has been tasked with developing and demonstrating the chemical processes necessary for removing the radionuclides from the bulk liquid supernate and concentrating it into a small HLW fraction.

The Office of Waste Management (OWM) has also determined that waste management operations at ORNL would benefit from removing cesium from liquid-low level waste (LLLW). The LLLW supernate in the Melton Valley Storage Tanks (MVSTs) has become increasingly difficult to manage due to the increasing cesium concentrations in these tanks. This increasing cesium concentration is due to additions from radiochemical processing operations at ORNL, such as the processing of irradiated Mark 42 target fuel elements at the Radiochemical Engineering Development Center.

Since cesium removal is a priority for both OST and OWM, these two offices have jointly funded a Cesium Removal Demonstration (CsRD). This is a full-scale demonstration conducted at ORNL using actual radioactive supernate from the MVSTs. Approximately 116,000 L (31,000 gal) of supernate has been processed. The sorbent chosen for the CsRD, a crystalline silicotitanate (CST) ion-exchange material, was selected on the basis of its effectiveness in batch and small-scale column tests using both simulants and actual waste supernates from the MVSTs. This is the first large-scale demonstration of the new state-of-the-art ion sorbents being developed jointly by DOE and private industry. The primary objectives for the CsRD are to (1) process up to 100,000 L (25,000 gal) of radioactive supernate; (2) demonstrate the use of modular, mobile equipment in existing facilities; (3) evaluate decontamination for hands-on maintenance and possible transfer to other sites; (4) compare bench- and full-scale data; (5) provide loaded sorbent for vitrification studies; (6) concentrate the cesium on small-volume, solid waste forms and package these to meet the Waste Acceptance Criteria (WAC) for the Nevada Tests Site (NTS); and (7) provide a full-scale operational system for use in baseline activities at ORNL after the demonstration has been completed.

Although the CsRD is being conducted at ORNL, the information produced will also be useful to Hanford and SRS because the MVST supernate is similar in chemical and radiological composition to the underground storage tank wastes stored at those sites. Hanford has included ion exchange for concentration of radionuclides in its baseline plans, and SRS has expressed interest in ion exchange as an alternative to their tetraphenylborate process.

SELECTION OF CRYSTALLINE SILICOTITANATE

The sorbent used for the CsRD was selected from state-of-the-art ion-exchange materials that are being developed primarily through the DOE-EM's Efficient Separations and Processing (ESP) Cross-Cutting Program. Six candidate ion-exchange materials were evaluated for potential use in the demonstration. These included (1) CS-100, an organic resin from Rohm & Haas; (2) crystalline silicotitanate IONSIV® IE-911, an inorganic sorbent from UOP Molecular Sieves; (3) potassium cobalt hexacyanoferrate (KCoFeC) an inorganic granular material from Eichrom Industries; (4) resorcinol-formaldehyde (RF), an organic resin from Boulder Scientific; (5) SuperLig 644C, an organic resin from IBC Advanced Technologies; and (6) WWL Web with SuperLig 644C, an organic resin bound in a proprietary organic web structure from 3M Corporation.

A bench-scale experimental system was set up in a hot cell at ORNL to generate the data necessary to evaluate the candidate sorbents. Details on the installation of the equipment in the hot cell and the procedures used have been previously reported.[2] Details of the tests, including run descriptions, breakthrough curves, elution curves, and operational problems, can be found in ORNL/TM-13363.[3] A summary of the breakthrough data for these runs is presented in Table 1.

Details on the ion-exchange selection criteria and the sorbent selection process can be found in ORNL/TM-13503.[4] Two of the sorbents, CS-100 and potassium cobalt hexacyanoferrate, were disqualified because their manufacturers could not provide a written guarantee that the sorbents would be available in the quantities necessary to complete the CsRD on schedule. Based on the discriminating criteria, CST IONSIV® IE-911 was selected for use in the CsRD. It was the only sorbent with no limiting operational problems and was rated first on all discrimination criteria except for kinetic performance.

CRYSTALLINE SILICOTITANATE DEVELOPMENT

Amorphous hydrous titanium oxide (HTO) materials were developed at Sandia in the 1960s and 1970s to prepare electroactive ceramic materials for defense applications. Work with HTO materials for high-level nuclear waste processing was initiated in 1975 at ORNL in collaboration with Sandia National Laboratories. The HTO absorbed most of the cationic radionuclides but had essentially no affinity for the highly soluble and radioactive cesium. About 1980, the program to develop amorphous HTO ion exchangers for application to nuclear wastes was concluded, based on DOE's decision to select glass, not ceramics, as the baseline waste form. Further development of HTO materials at Sandia for use as catalysts for coal liquefaction and other applications was continued through the DOE Fossil Energy Program. As part of this effort, a new class of exchangers called crystalline silicotitantes was prepared by personnel from Sandia and Texas A&M University. Testing demonstrated that this material had a

TABLE 1. A SUMMARY OF THE BREAKTHROUGH DATA FOR THE BENCH-SCALE HOT-CELL RUNS

Sorbent	Flow Rate (CV/h)[a]	CV to 1% Breakthrough	CV to 10% Breakthrough	CV to 50% Breakthrough
RF—Run 1	7	8	14	45
RF—Run 2	3	10	22	36
RF—Run 3	6	8	17	46
CS-100	3	3	8	17
SuperLig—Run 2	6	*	9	95
CST—Run 1	3	110	190	350
CST—Run 2	6	80	170	350
3M WWL	49	20	40	60
KCoFeC	9	90	225	225

[a]CV = column volumes (volume of sorbent in column).
* = immediate breakthrough

large affinity for cesium in the presence of high sodium concentrations.[4,5] A Cooperative Research and Development Agreement between Sandia and UOP was signed in March 1994, and UOP was awarded a license to produce and market the CST technology. In January 1995, UOP delivered the first engineered form of CST for evaluation; by December 1995, a final formulation had been selected and the material was declared commercially available. This material was given the name UOP IONSIV®Ion Exchanger Type IE-911. In January 1996, the UOP IONSIV® IE-911 was chosen for use in the CsRD at ORNL based on the results of bench-scale testing.[2,3] This granular sorbent has a particle size from 30 to 60 mesh and a bulk density of 1 g/cm^3. Other properties of the UOP IONSIVE® IE-911 have been described by Miller and Brown.[4]

DEMONSTRATION SYSTEM

Design specifications for the CsRD system were finalized and the contract for design and fabrication of the system was put out for competitive bid.[7] The CsRD system was designed to be a modular, mobile system and was supplied on three separate skids: (1) a feed tank skid, (2) an ion-exchange skid, and (3) a sorbent sluicing/drying skid. The feed skid contained a nominal 1890-L (500-gal) feed tank and two feed pumps. The ion-exchange skid contained a 25-μm stainless steel, backwashable filter and two ion-exchange columns. The ion-exchange vessels were ~30 cm ID by ~97 cm high and could be operated individually, in series, or in parallel. The sorbent sluicing/drying skid contained equipment used for remotely sluicing the sorbent to and from the ion-exchange columns as well as for remotely drying the sorbent after it had been sluiced from the columns. The piping and instrumentation drawing and an overhead view of the processing facility after the shielding had been put in place are presented in Figures 1 and 2, respectively. Details on the CsRD design, installation, and operation have been previously reported by Walker et al.[6]

CESIUM REMOVAL DEMONSTRATION RESULTS

A total of four CsRD runs were conducted. The first was a single-column, minimal-loading run designed to test the system and procedures on actual MVST supernate prior to the full-loading runs. A total of ~23 Ci of ^{137}Cs was loaded onto 38 L (10 gal) of sorbent. The sorbent from this run was shipped to the SRS for use in a demonstration to show that the sorbent can be incorporated in vitrified high-level waste that is suitable for ultimate disposal.

The second run consisted of a single 38-L column of sorbent operated at 3 bed volumes (BV) per hour. Approximately 18,028 L (4763 gal) of MVST supernate was processed prior to 50% breakthrough of ^{137}Cs and ~154 Ci of ^{137}Cs, was loaded on the sorbent.

The third run consisted of the operation of two 38-L columns of sorbent operated in series at a flow rate of 6 BV/h. Approximately 39,061 L (10,320 gal) of supernate was processed with the lead and lag columns reaching ~80 and ~47% breakthrough of ^{137}Cs, respectively. The lead column loaded a total of ~222 Ci of ^{137}Cs and the lag column loaded ~112 Ci of ^{137}Cs.

The fourth run was a three-column run with each column containing 38 L of sorbent. The system was initially started with two columns in series. The lead column (column 1) reached 55% breakthrough of ^{137}Cs after processing ~28,346 L (7489 gal) and loading ~253 Ci of ^{137}Cs. Column 1 was then removed from service, the lag column (column 2) was moved into the lead column position, and a column with new sorbent (column 3) was placed in the lag column position. Analyses indicate that column 2 loaded ~266 Ci of ^{137}Cs and column 3 loaded ~112 Ci of ^{137}Cs, for a total of ~631 Ci of ^{137}Cs removed during run 4.

During the entire operational period, ~ 115,757 L (30,583 gal) of supernate was processed and ~1142 Ci of ^{137}Cs was removed from that supernate and loaded onto 70 gal of sorbent. The average feed concentration for each of runs 2–4 is presented in Table 2. It should be noted that analyses for run 1 are not included because cross-contamination occurred between the samples for this run. Also, supernate from a different tank was added to the feed tank prior to run 4. This accounts for some of the concentration changes between run 3 anc 4. The EM-50 milestone to complete processing of up to

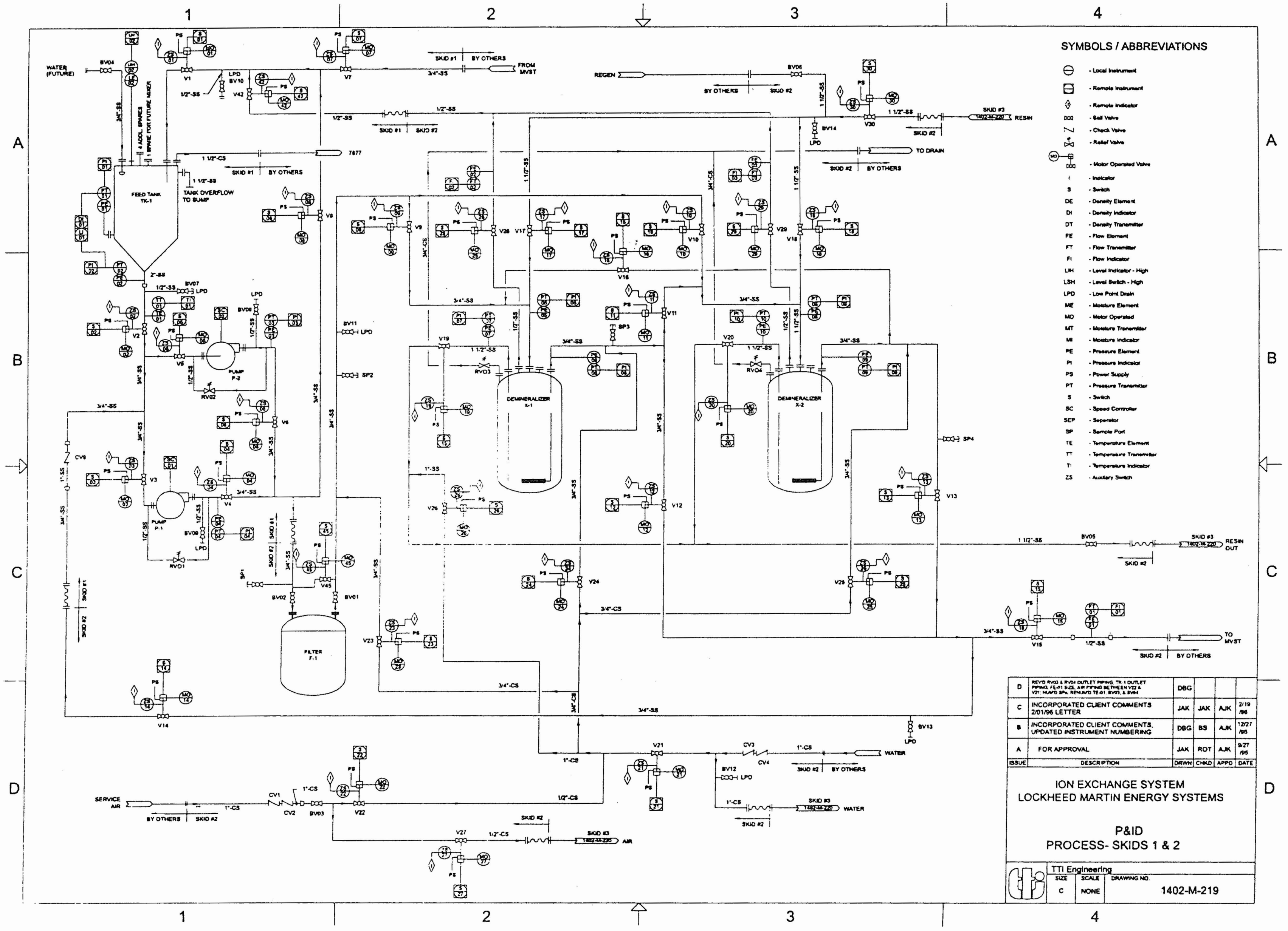

FIGURE 1. Piping and instrument diagram for the CsRD feed tank skid and the ion-exchange skid.

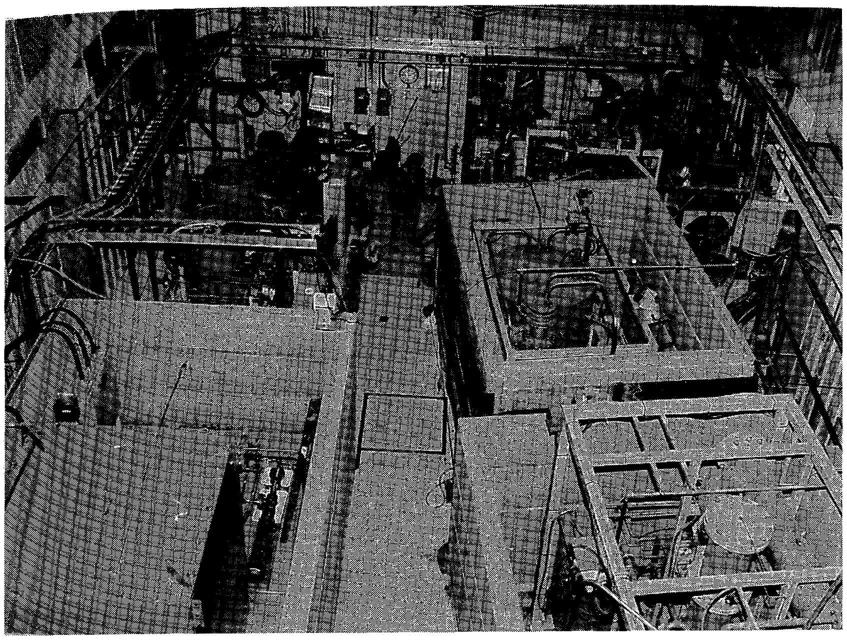

FIGURE 2. Photograph of the CsRD processing facility after shielding has been put in place.

94,625 L (25,000 gal) of MVST supernate by July 31, 1997, was met, as was the EM-30 milestone to remove at least 520 Ci of radioactive cesium by June 30, 1997. The sorbent from the last three runs will be stored at the ORNL solid waste storage area until it can be shipped off-site for permanent disposal. Laboratory analysis has shown that the loaded sorbent is nonhazardous, and no further stabilization is necessary prior to disposal.

Cesium Removal Demonstration Run # 1

The first CsRD run was a limited-loading run with the goal of loading ~25 Ci of ^{137}Cs prior to shutting down. The primary reason the first run was limited to ~25 Ci of ^{137}Cs was to provide information for later runs, when loadings would be much higher (i.e., >200 Ci of ^{137}Cs per column and as much as 1000 Ci of ^{137}Cs present in the processing building) and the risk of radiation exposure to individuals would be much greater. The information to be provided included verification of (1) operational procedures during an actual run with radioactive supernate, (2) ion-exchange system functionality during an actual run with radioactive supernate, (3) performance of the sorbent sluicing/drying system with cesium-loaded sorbent,

TABLE 2. AVERAGE FEED CONCENTRATION FOR THE CsRD RUNS 2–4

	Run 2	**Run 3**	**Run 4**
^{137}Cs (Bq/mL)	417,500	398,000	490,000
^{134}Cs (Bq/mL)	5,650	5,170	13,000
^{60}Co (Bq/mL)	395	372	320
^{90}Sr (Bq/mL)	1,675	1,280	823
Ba (mg/L)	0.618	0.776	0.31
Ca (mg/L)	2.41	4.25	71.7
Cr (mg/L)	3.27	3.82	4.03
Cs (mg/L)	0.966	1.79	0.678
Hg (mg/L)	0.0811		
K (mg/L)	16,150	16,700	20,100
Mg (mg/L)	<0.075	0.341	0.25
Ne (mg/L)	100,000	88,000	-
NO$_3$ (mg/L)	270,000		
Pb (mg/L)	2.4	0.623	0.35
Rb (mg/L)	2.64	4.08	2.19
Sr (mg/L)	1.143	1.24	0.43
U (mg/L)	8.86	11.7	
Zn (mg/L)	0.852	0.842	0.98
Ph	12.7	12.7	-

(4) the ability to sluice loaded sorbent if operational problems resulted in the sorbent remaining in the column for a long period of time, (5) remote transfer equipment and procedures utilizing cesium-loaded sorbent, (6) transportation procedures and equipment using cesium-loaded sorbent, and (7) operation of the remote sampler, as well as (8) measurement of radiation levels during sluicing and transfer of the loaded sorbent to assist in determining the expected radiation levels at higher loadings. In addition, the first run served to provide material for use by SRS for a vitrification demonstration and for training SRS personnel at ORNL on the remote transfer of the radioactive sorbent.

Cesium Removal Demonstration Run # 2

Column X-1 was loaded with ~38 L of conditioned CST sorbent on February 13, 1997. Flow of W-29 supernate at 3 BV/h to this column was started on February 16 at ~10:00 a.m., initiating CsRD run #2. Flow to the column was terminated on February 23 at ~12:30 p.m. after processing ~18,028 L (4763 gal) of supernate. There were no major operational problems during this run.

Breakthrough curves for ^{137}Cs and ^{134}Cs during run #2 are presented in Figure 3. As can be seen, the breakthrough curves for ^{137}Cs and ^{134}Cs are essentially identical. For both ^{137}Cs and ^{134}Cs, 1 and 50% breakthroughs occur at ~75 and 475 BV, respectively. The sorbent loaded ~154 Ci of ^{137}Cs and 2.1 Ci of ^{134}Cs. Breakthrough curves for ^{60}Co, ^{90}Sr, and uranium are presented in Figure 4. Essentially all of the ^{90}Sr is removed from the supernate, and the CST does not load ^{60}Co. The uranium is loaded by the sorbent, with 50% breakthrough occurring after ~120 BV of supernate had been processed. Approximately 2.5 h was required to dry the sorbent with ambient air after it had been remotely sluiced from the column. After the sorbent had been dried and prior to sealing the drum, 38 L (10 gal) of vermiculite was remotely added to the top of the drum to ensure that no free liquid was present. This was done to ensure that the Waste Acceptance Criteria (WAC) for the NTS were met.

Cesium Removal Demonstration Run #3

Approximately 38 L of conditioned CST sorbent was added to each ion-exchange column. Supernate processing for the third run was initiated with the CsRD system being operated with two columns in series at a flow rate of ~ 3.8 L/min (6 BV/h for each individual column and 3 BV/h for the two columns in series). The system was shut down after processing approximately 11,635 L (3074 gal) of supernate. This event was scheduled to determine the effect a prolonged shutdown would have on the breakthrough curve. The system was restarted at a flow rate of ~3.8 L/min after being shut down for ~17 days and ~39,061 L (10,320 gal) of supernate was processed prior to the termination of run #3..

Breakthrough curves for ^{137}Cs, ^{134}Cs, and total cesium are presented for the columns in series and for the lead column for the third CsRD run, respectively, in Figures 5 and 6. As can be seen, the ^{137}Cs, ^{134}Cs, and total cesium track each other well. This is to be expected because ion-exchange processes do not significantly differentiate between isotopes. From Figure 5, it can be seen that 1% breakthrough of ^{137}Cs and ^{134}Cs occurred after ~75–80 BV had been processed and ~47% breakthrough occurred after ~470 BV had been processed. Both of these compare well with the second CsRD run, which was also conducted at 3 BV/h. The ^{137}Cs loadings for both columns during the third run totaled ~334 Ci, which averages 4.4 Ci/L. This also compares favorably with the second run, which totaled 4.1 Ci/L. A slightly higher capacity per unit volume of sorbent could be expected in the third run because the fractional breakthrough in this run was slightly less than that in the second run.

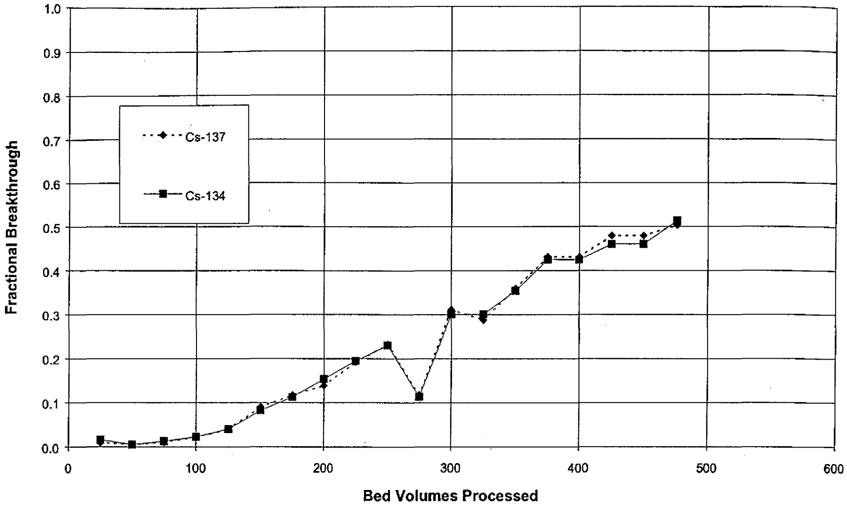

FIGURE 3. Breakthrough curves for ^{137}Cs and ^{134}Cs during the second CsRD run.

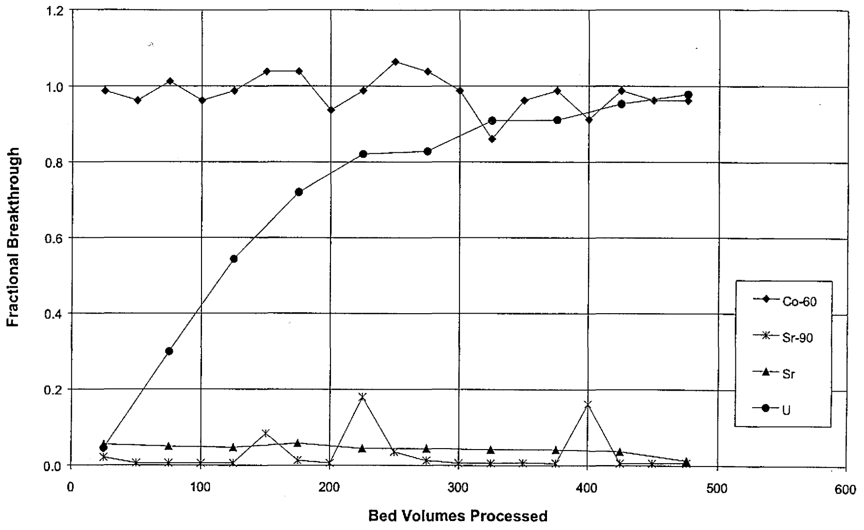

FIGURE 4. Breakthrough curves for ^{60}Co, ^{90}Sr, Sr, and U for the second CsRD run.

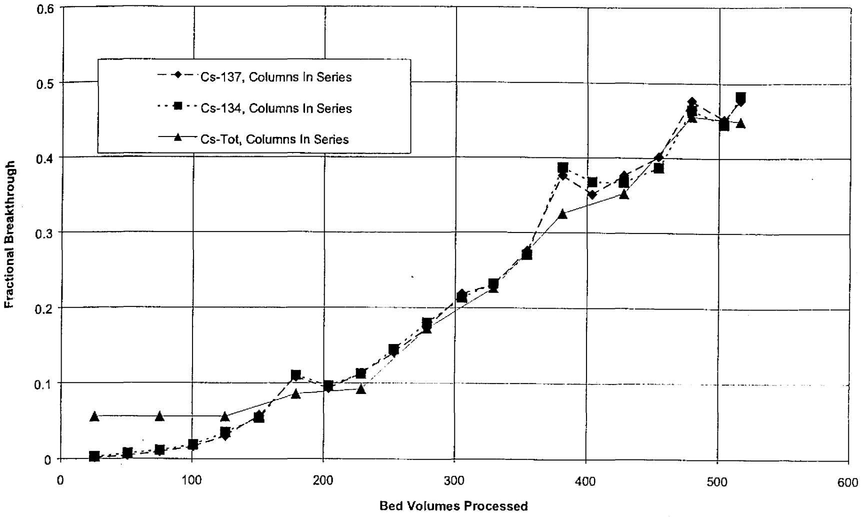

FIGURE 5. Breakthrough curves for ^{137}Cs, ^{134}Cs, and total Cs for the two columns in series for the third CsRD run.

As can be seen in Figure 6, the fractional breakthrough for the first column in series during the third CsRD run was taken to about 80%. This column loaded ~222 Ci of ^{137}Cs. Also, the effect of the ~10-day shutdown, with the sorbent allowed to sit in the supernate solution, can be seen in this figure. This scheduled shutdown occurred at ~300 BV. The shutdown did not have a negative impact on the loading of ^{137}Cs after the system was restarted. This indicates that in full-scale operations, the ion-exchange process could be shut down for several days without decreasing the expected loading, which adds considerable flexibility to the operation. Breakthrough curves for Sr, Co, and U were typical of those in the second CsRD run. Data from this run also indicated that the CST loads Pb, Ba, and Zn.

Cesium Removal Demonstration Run #4

The fourth and final CsRD run was a three-column run, with each column containing 38 L of sorbent. The lead column (column 1) reached 55% breakthrough of ^{137}Cs after processing ~28,346 L (7489 gal) and loading ~253 Ci of ^{137}Cs. Column 1 was then removed from service, the lag column (column 2) was moved into the lead column position, and a column with new sorbent (column 3) was placed in the lag column position. Column 2 loaded ~266 Ci of ^{137}Cs and column 3 loaded ~112 Ci of ^{137}Cs, for a total of ~631 Ci of ^{137}Cs removed during run 4. The flow rate during the initial portion of the run was 3.8 L/min (3 BV/h for the system), and it was raised to 7.6 L/min (6 BV/h for the system) for the final portion of the run.

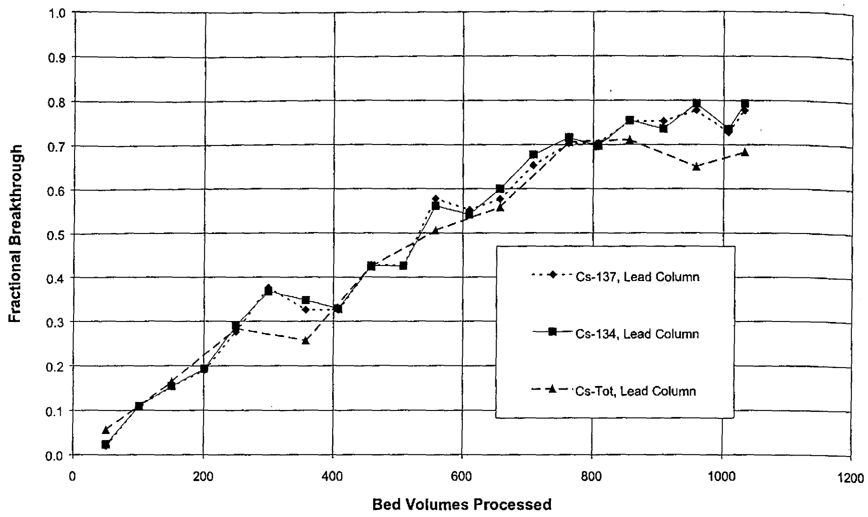

FIGURE 6. Breakthrough curves for ^{137}Cs, ^{134}Cs, and total Cs for the lead column for the third CsRD run.

Approximately 590 BV was required to reach 50% ^{137}Cs breakthrough in the lead column. This compares with ~470 BV required for 50% loading of ^{137}Cs in the lead column during run number 3. The discrepancy in the capacity for ^{137}Cs between runs 3 and 4 can be attributed to the differences in feed concentrations. After the third CsRD run had been completed, insufficient supernate existed in the W-29 MVST feeding the CsRD system; therefore, two MVSTs were combined to provide adequate supernate to complete run 4. A summary of the supernate concentrations for the three runs can be found in Table 2. The concentration of radioactive cesium was higher in run 4 than in run 3; however, the total cesium concentration was less. Therefore, one would expect to load more radioactive cesium during run 4. The total Sr was also less in this feed, which may have also resulted in layer breakthrough values at 50%. The loading curves for ^{134}Cs, total Cs, ^{90}Sr, total Sr, ^{60}Co, U, Ba, Pb, Rb, and Zn were typical of those previously presented for earlier CsRD runs.

A graph of the cumulative volume of supernate processed during the fourth run versus the ^{137}Cs fractional breakthrough is presented in Figure 7. The high fractional breakthrough for column 2 in the lag position at the initiation of this run can probably be attributed to residual loaded sorbent remaining in the lag column from the previous run. Since the inlet concentration to the lag column was very low at the beginning of the run prior to breakthrough from the lead column, any residual loaded sorbent from the previous run would tend to skew the fractional breakthrough for the lag column to the high side initially.

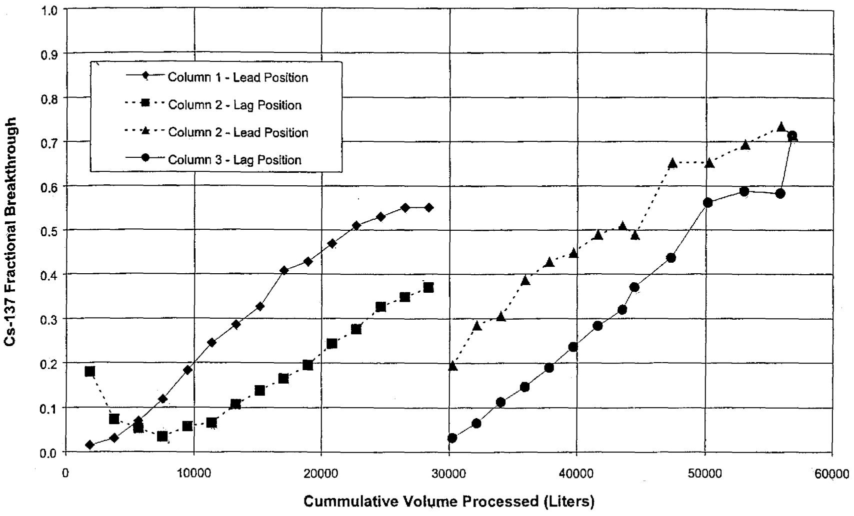

FIGURE 7. Graph of the cumulative volume of supernate processed during the fourth CsRD run versus the ^{137}Cs breakthrough.

BENCH-SCALE SUPPORT

Bench-scale tests were conducted with actual supernate and equipment used to provide data in the sorbent selection process for the CsRD. In addition, a 25-μm filter was placed upstream of the ion-exchange column(s) in the bench-scale simulation to better simulate the full-scale demonstration. The primary objectives of the bench-scale tests were (1) to allow direct comparisons of the breakthrough curves for the bench- and full-scale systems using the same feed and similar operating conditions and (2) to provide samples of loaded sorbent for analysis to document that the loaded CST sorbent from the CsRD was not hazardous as defined by the Toxicity Characteristic Leaching Procedure (TCLP) for Resource Conservation and Recovery Act (RCRA) metals.

Five bench-scale simulations with small columns were conducted. Comparisons of the ^{137}Cs breakthrough curves for the full- and bench-scale runs are provided in Figure 8 for the 3-BV/h runs. As can be seen, very good agreement exists between the bench- and full-scale data, indicating that laboratory-scale column data can be successfully used to design full-scale ion-exchange systems. It should be noted that there was good agreement between the bench- and full-scale runs at 6 BV/h. Moreover, less scatter was noted in the bench-scale data than in the data from the full-scale CsRD system. This can be attributed to the longer counting times used in the analysis of the samples from the bench-scale operations.

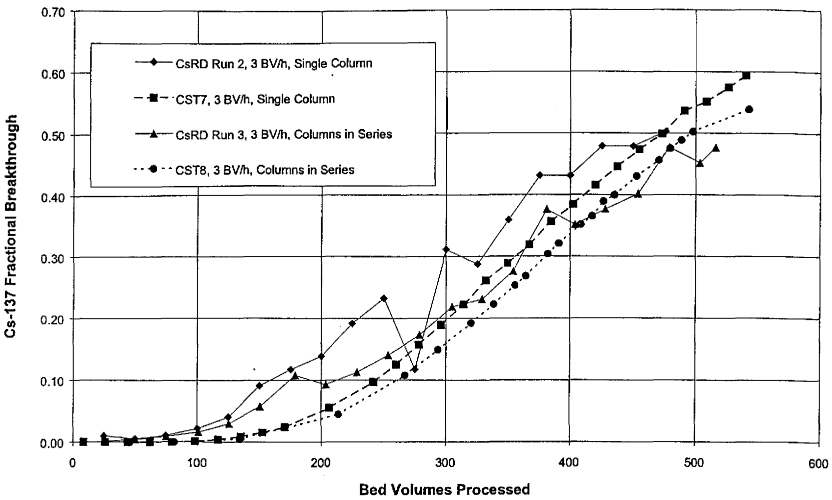

FIGURE 8. Comparison of the ^{137}Cs breakthrough curves for the full- and bench-scale runs at 3 BV/

Solid samples of the loaded sorbents from the bench runs were taken and subjected to the TCLP for RCRA metals. Analytical results from these procedures indicated that the concentrations of the RCRA metals in the leachate from the TCLP were much less than the regulatory limit set for hazardous waste. This meets part of the WAC required for permanent disposal at NTS.

SUMMARY

A full-scale Cesium Removal Demonstration (CsRD) has been jointly funded by the DOE Office of Science and Technology and the Office of Waste Management. The CsRD was conducted at ORNL using actual radioactive supernate from the MVSTs. The sorbent chosen for the demonstration, a crystalline silicotitanate (CST) ion-exchange material, was selected on the basis of its effectiveness in batch and small-scale column tests using both simulants and actual waste supernates from the MVSTs. This is the first large-scale demonstration of the new state-of-the-art ion-exchange sorbents being developed jointly by DOE and private industry.

A total of four CsRD runs were conducted. During the entire operational period, ~ 115,757 L (30,583 gal) of supernate was processed and ~1142 Ci of ^{137}Cs was removed from that supernate and loaded onto 265L (70 gal) of CST sorbent. The CST also loaded Sr, U, Pb, Ba and Zn. Laboratory analysis of the spent sorbent has shown that it is nonhazardous, and no further stabilization is necessary prior to disposal. Data analysis also indicated that the cesium breakthrough curves for the bench- and tl full-scale columns agreed very well, suggesting that the bench-scale columns can be used to predict scale-up.

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