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DECONTAMINATION OF SAVANNAH RIVER PLANT WASTE SUPERNATE

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Printed in the United States of America

Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161

Price: Printed Copy $3.50; Microfiche $2.25

662620

DP-1436
Distribution Category: UC-70

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Publication Date: August 1976

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ABSTRACT

Cesium-137, strontium-90, and plutonium were removed from 100-liter quantities of actual waste supernate by ion exchange in tests of the conceptual process for solidification of Savannah River Plant (SRP) radioactive waste. The supernate had been slurried with actual SRP sludges, and these sludges were separated from supernate by centrifugation and sand filtration. These steps simulated tank cleaning and isolating the insoluble waste sludge. Ion exchange reduced radioactivity in the supernate by a factor of 4×10^5 for ^{137}Cs, 5×10^3 for ^{90}Sr, and 300 for plutonium. These results were insensitive to flow rate over the range of rates studied. The process was monitored with an in-line gamma detector. Salt cake made from the decontaminated supernate would contain about 6 nanocuries/gram from the combined activities of ^{137}Cs, ^{90}Sr, and plutonium. ^{106}Ru was not removed; it will decay to a level below that of other nuclides in decontaminated salt stored for 12 years.

^{137}Cs, ^{90}Sr, and plutonium were eluted from the ion exchange columns, concentrated by evaporation of the eluate, and sorbed onto zeolite. This concentrated the radioactivity removed from supernate by a factor of 1500.

Carrier precipitation was tested for removing ^{90}Sr from supernate, but was not effective.

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INTRODUCTION

Methods for possible solidification and storage of high-level liquid radioactive waste at the Savannah River Plant (SRP) are being studied at the Savannah River Laboratory (SRL). One of several options under consideration is storage of solidified waste in an onsite retrievable surface storage facility until the waste can be shipped to a Federal repository.

SRP waste, stored in underground tanks, consists of an alkaline salt solution, a solid salt cake, and an insoluble sludge layer containing large amounts of iron, aluminum, manganese, uranium, and many other elements. Origins and characteristics of SRP waste, criteria for acceptable solid forms, and potential solid forms for SRP waste have been described in an earlier report.[1] That study was used to formulate the conceptual waste solidification process shown in Figure 1.

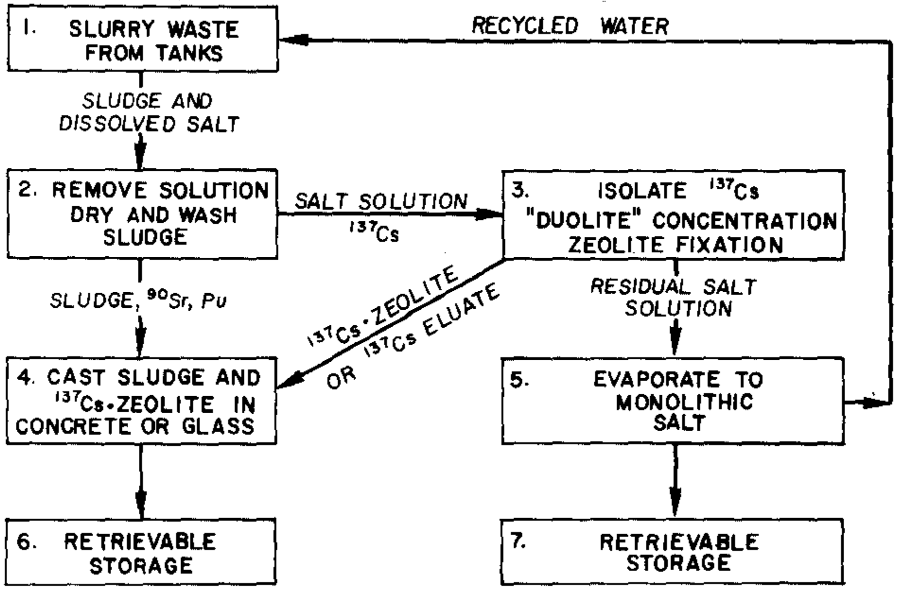

FIGURE 1. Conceptual Waste Solidification Process Divided into Modules

In the conceptual process, waste would be removed from a tank by dissolving the salt cake with water and sluicing the slurry of sludge and solution from the tank. Sludge and solution would be separated by centrifugation and filtration. ^{137}Cs, the principal biological hazard in solution, would be removed by an ion exchange process and sorption onto zeolite. The sludge would be washed to remove soluble salts; then the sludge would be dried and blended with cesium-loaded zeolite or concentrated cesium eluate for incorporation into either concrete or glass.

This report describes tests of supernate decontamination (Module 3, Figure 1). These tests were conducted in shielded, remotely operated cells so that large quantities (typically 50 to 150 liters) of highly radioactive SRP wastes could be used. Concurrent tests[2] of Module 2 provided clarified supernate for tests of Module 3. Two previous reports described laboratory-scale development of the ion exchange process used in Module 3.[3,4] Sludge solidification in concrete and glass (Module 4) has also been studied in the laboratory.[5,6]

TEST OBJECTIVES

The purpose of these tests was to show that waste supernate, separated from the plant sludge in Module 2, could be decontaminated to a degree that bulk storage of the salt produced by evaporating the supernate would be feasible. The salt could be stored with less rigorous containment than that required for high level waste. These tests included removal of ^{137}Cs, ^{90}Sr, and plutonium (all isotopes) from SRP waste supernate. No method was developed for large-scale removal of ^{106}Ru. Because ^{106}Ru has a half-life of 369 days, its activity in waste salt will have decreased by a factor of about 4000 after 12 years of storage. ^{106}Ru activity in salt cake will then be less than that of residual ^{137}Cs, ^{90}Sr, or plutonium.

At present, no guidelines have been established as to how much radioactivity could be allowed to remain in the salt cake before shipment to a Federal repository is required. A total activity of 10 nCi/g of salt (excluding ^{106}Ru) was chosen as a goal for this work. For comparison, the ^{40}K activity in natural KCl is 0.4 nCi/g.

These tests were designed to study:

- Effects of mixing supernates with various sludge types before ion exchange decontamination.
- Effects of flow rate and throughput on decontamination by ion exchange.

- Possible methods of removing ^{90}Sr during separation of sludge and supernate.

- The ability of an online gamma detector to monitor the ion exchange process.

- The effect of the 100-fold scale-up from previous laboratory-scale ion exchange tests.

EQUIPMENT

The ion exchange apparatus for these tests consisted of three 22-in.-long by 3-in.-diameter stainless steel columns. Two columns were each filled with 2.5 liters of *Duolite* ARC-359,* a phenolsulfonic acid cation exchange resin, 20 to 50 mesh. The third column contained 1.3 liters of *Chelex-100*** resin, 50 to 100 mesh. *Chelex-100* is an analytical grade of *Dowex* A-1,*** an iminodiacetate chelating resin. A stainless steel screen above the *Chelex-100* resin separated it from 4-mm-diameter glass beads, which filled the remainder of the column. All three resin beds were supported by 100-mesh stainless steel screens. Identical screens at the top of each column prevented loss of resin during backwashing. Column end fittings were sealed with O-rings. Vent tubes above each column allowed occasional bubbles to escape and prevented siphoning. The three columns were usually connected in series. However, the *Chelex-100* column was bypassed if ^{90}Sr had been removed earlier by carrier precipitation. Neither *Duolite* ARC-359 nor *Chelex-100* showed any sign of deterioration or buildup of radiation during the tests.

Figure 2 shows the columns, vent tubes, metering pump (Gorman Rupp), valves (Whitey, stainless steel, ball), and sample lines mounted on a steel frame. During operation, column effluent flowed continuously out of the sample line and into the cell drain. Samples of processed supernate were obtained by collecting this effluent stream in a clean vial.

In addition to samples that were analyzed offline, ^{137}Cs in effluent from the first *Duolite* column was detected by an online monitor. The effluent flowed through a 1/4-in. stainless steel line which passed through the wall of the shielded cell, past a Ge(Li) gamma detector, back into the cell, and into the second *Duolite* column. The Ge(Li) detector was interfaced with a PDP-15 computer† which calculated the fractional ^{137}Cs breakthrough each

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* Product of Diamond Shamrock.

** Product of Bio-Rad Laboratories.

*** Product of Dow Chemical Company.

† Digital Equipment Corporation, Maynard, MA.

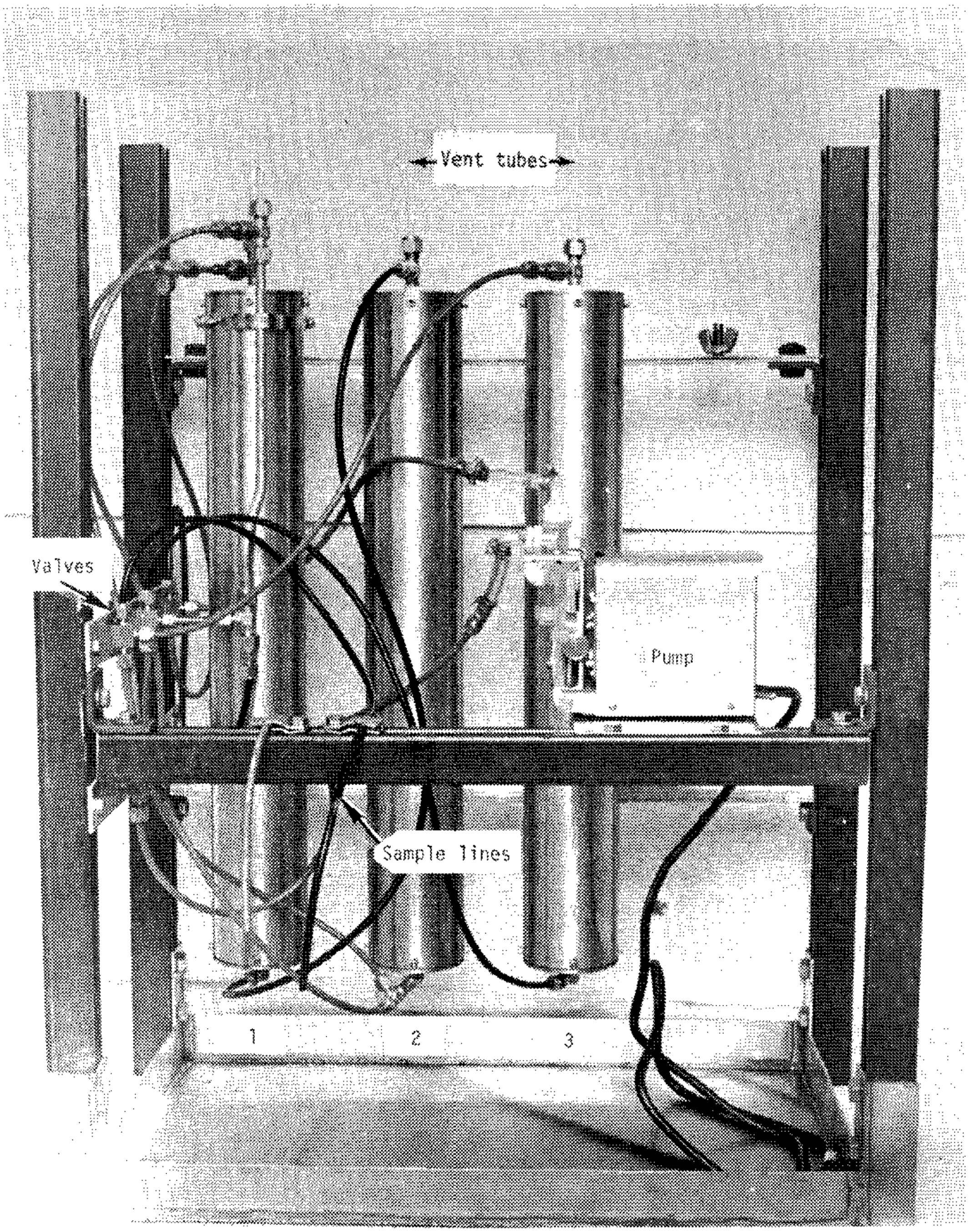

FIGURE 2. Ion Exchange Equipment

hour. Online monitoring of very low levels of ^{137}Cs in effluent from the second *Duolite* column was impractical because of interference from ^{106}Ru.

A 50-liter polyethylene bottle was used as the reservoir for radioactive supernate feed, and a 25-liter bottle held nonradioactive resin-conditioning solutions. After supernate had been processed, resins in the three columns were washed with 4 column volumes (CV)* of water to keep elutriant solutions from mixing with remaining supernate. Mixing would have caused $Al(OH)_3$ to precipitate and to plug the columns. Cesium and plutonium were eluted from the *Duolite* ARC-359 resin with 10 CV of 2M NH_4OH - 2M $(NH_4)_2CO_3$ solution. Strontium was eluted from the *Chelex-100* resin with 4 CV of 1M HNO_3 . The two resins were eluted separately because HNO_3 dissolves the *Duolite* resin, and the ammonia solution does not effectively remove ^{90}Sr from *Chelex-100.* A second wash with 2 CV of water removed the acid. All three columns were regenerated with 10 CV of 2M NaOH. Supernate and water for the first wash were pumped downflow through the columns. Resins were eluted and regenerated upflow, and the *Chelex-100* was washed (second wash) upflow.

A glass kettle and condenser (Figure 3) were used to concentrate the radionuclides separated from waste supernate. About 1 liter of water was heated to boiling in the kettle, column elution was started, and eluate was pumped into the kettle. NH_3, CO_2, and H_2O were evaporated from the ion exchange column eluate, condensed, and discarded.[7] An acid scrubber prevented release of NH_3 into the cell. Evaporation rate matched the rate of elution, 2.5 liters/hr. At the end of an elution, the kettle contained about 2 liters of concentrate: a Na_2CO_3-$NaNO_3$ solution containing ^{137}Cs, ^{90}Sr, and plutonium. The concentrate from several ion exchange tests was accumulated and then passed through a column of zeolite, which immobilized the radionuclides.

DESCRIPTION OF TESTS

Supernate from Waste Tank 15H was decontaminated in the first three tests, and supernate from Tank 34F was used in the others (Table 1). Those supernates chosen were representative of waste from the two processing facilities (F and H) at SRP. These specific tanks were chosen because they contained waste supernates that were at least 10 years old; contained high levels of ^{137}Cs, ^{90}Sr, and plutonium; and were available for sampling. The composition of supernates was adjusted to a reference composition by adding nonradioactive salt solutions before each test (Table 2).

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* One column volume = 2.5 liters.

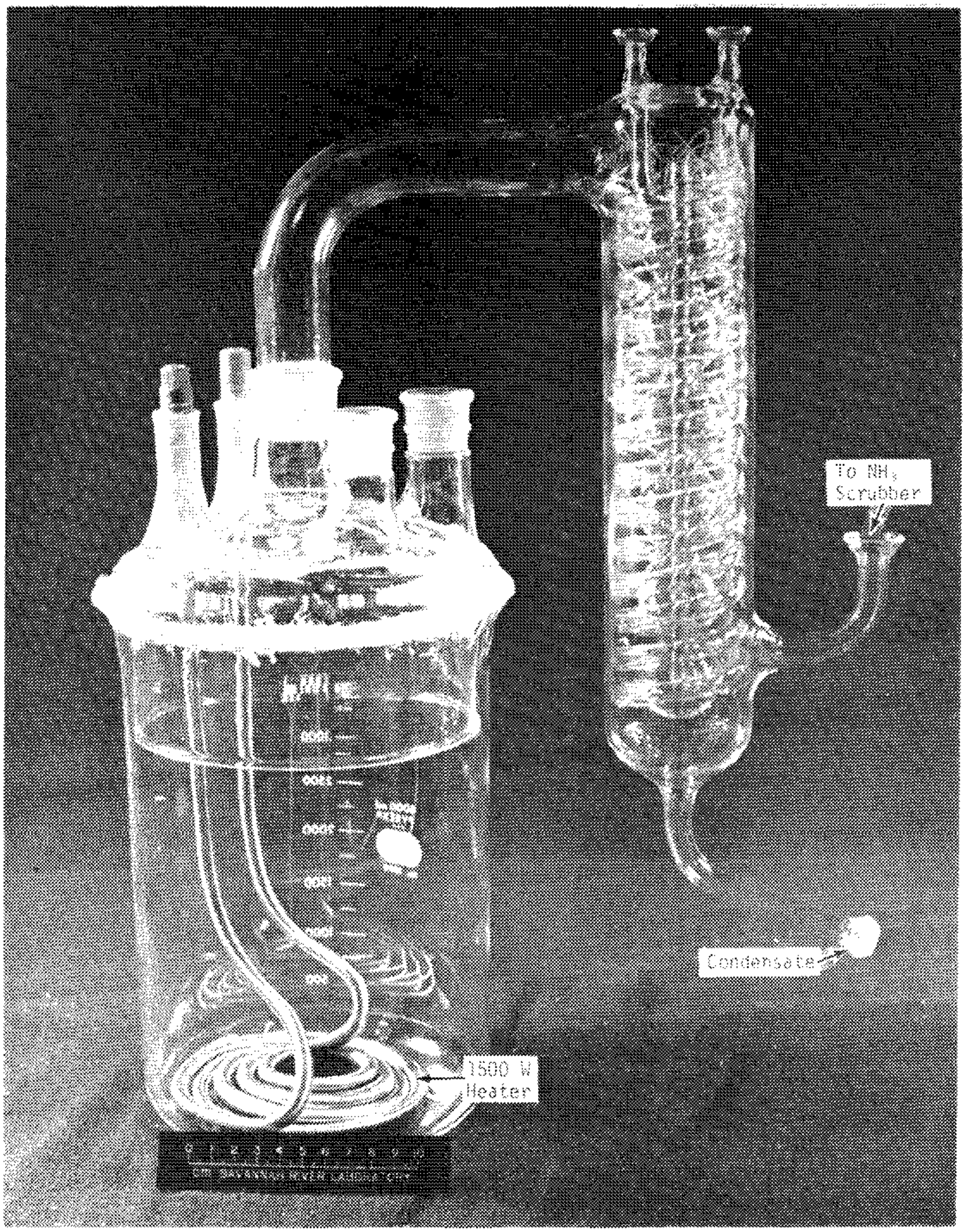

FIGURE 3. Eluate Evaporator and Condenser

TABLE 1

Summary of Ion Exchange Tests

		Supernate		
Test	*Sludge*[a] *Composition*	*Volume, CV*	*Flow Rate, CV/hr*	^{90}*Sr Removal*
1	None	20	1	*Chelex-100*
2	Fe, Al, Mn, U	20	1	*Chelex-100*
3	Fe, Al, Mn, U	30	2	$Sr_3(PO_4)_2$
4	Fe, U, Mn, Na	40	2	$Fe(OH)_3$
5	Fe, U, Na, Al	60	3	$CaCO_3$
6	Al, U, Fe, Hg	40	1	*Chelex-100*
7	Al, U, Fe, Ca	40	2	HZO-1[c]
8	Al, Fe, Ca, Mn	40	1	*Chelex-100*
9	None	20	1	*Chelex-100*

---

[a] Principal cations (Reference 2).

[b] Tank 15H supernate used in Tests 1 through 3. Tank 34F supernate used in Tests 4 through 9. One column volume (CV) = 2.5 liters.

[c] Hydrated zirconia gel from Bio-Rad Laboratories.

TABLE 2

Supernate Compositions, Molar

	Tank 15H	*Tank 34F*	*Reference Composition*[a]
$NaNO_3$	3.4	2.4	2.2
$NaNO_2$	1.2	2.6	1.1
NaOH	1.4	5.9	0.75[b]
$NaAlO_2$	0.54	0.44	0.5
Na_2CO_3	0.09	0.01	0.3
Na_2SO_4	0.04	0.01	0.3

---

[a] Expected for a mixture of supernate and dissolved salt cake.

[b] Actual OH^- concentration in adjusted supernates was 0.75 to 1.0M

Supernates were adjusted so that effects of mixing supernates with different sludges and effects of varying the ion exchange process conditions could be determined. Sludges for these tests were also representative of the different sludge types in the waste tank farm.

About 3 liters of plant sludge was slurried with each 50 liters of adjusted supernate. Supernate and sludge remained in contact for at least 24 hr. During this time, radioactive elements from the sludge were expected to regain their equilibrium concentrations in the supernate.

Sludge and supernate were separated by two passes through a centrifuge.[2] In some tests (Table 1), a ^{90}Sr carrier precipitant was added between the two centrifuge passes. After the first pass, 250 ml of a solution containing the carrier was poured into the sludge-supernate mixture. The second centrifuge pass then removed remaining sludge and precipitate. A filter consisting of two 4-ft-long sand and anthracite coal beds removed the remaining particulates. Solids were not detectable in the filtrate, which was then decontaminated by ion exchange.

TEST RESULTS

Removal of ^{137}Cs and Plutonium by Ion Exchange

Cesium-137

^{137}Cs decontamination factors (DF)* were consistent throughout all nine tests (Table 3). Slurrying supernate with various sludges did not affect cesium retention by the *Duolite* ARC-359 resin. Flow rates up to 3 CV/hr did not degrade separation. The overall average DF of 4×10^5 was in excellent agreement with previous results; the 100-fold scaleup[3] caused no adverse effects.

In the fifth test, enough supernate was processed to obtain a ^{137}Cs breakthrough profile from both *Duolite* columns (Figure 4). The relation between ^{137}Cs in effluent from the first column (monitored online) and that from the second column could be used to control a plant-scale decontamination process. For a specified final ^{137}Cs DF, supernate would be processed until the corresponding fraction of ^{137}Cs appeared in first column effluent. ^{137}Cs coming through the second column cannot be monitored directly online because of interference from ^{106}Ru. Effluent monitoring will be necessary in a plant process because waste supernates of different concentrations will be decontaminated. ^{137}Cs retention depends on the concentration of both Na^+ and OH^- ions in waste supernate.[3]

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* Decontamination Factor = Activity in Feed/Activity in Product.

TABLE 3

Removal of ^{137}Cs and Plutonium by *Duolite* ARC-359 Resin

Test Number	^{137}*Cs, dis/(min-ml)*		^{137}*Cs DF*	*Pu, dis/(min-ml)*		*Pu DF*
	Feed	*Product*[a]		*Feed*	*Product*	
1	1.2×10^9	3200	3.7×10^5	3,000	50	60
2	1.2×10^9	1500	8.0×10^5	16,000	2000	8
3	1.2×10^9	2800	4.2×10^5	10,000	2000	5
4	1.1×10^9	2800	3.9×10^5	38,000	1000	38
5	1.1×10^9	3000	3.7×10^5	600[c]	2000	-
6	1.1×10^9	2600	4.2×10^5	200[c]	1500	-
7	1.1×10^9	3000	3.7×10^5	30,000	1000	30
8	1.1×10^9	1800	6.1×10^5	30,000	700	42
9	1.1×10^9	3700	2.9×10^5	1.5×10^7[b]	5×10^4	300

---

[a] Average over entire test. Test 5, average over first 40 column volumes.

[b] Saturated with ^{238}Pu.

[c] Counted after standing several weeks. $Al(OH)_3$ precipitate probably carried plutonium out of solution.

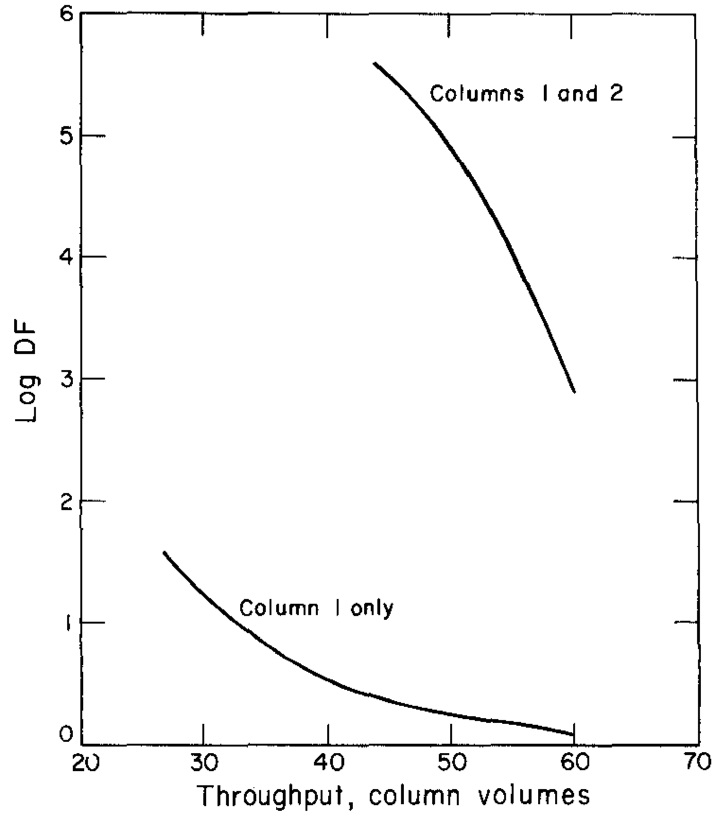

FIGURE 4. ^{137}Cs Decontamination as a Function of Throughput

Plutonium

Plutonium was also removed from waste supernate by *Duolite* ARC-359 resin (Table 3). Product activities for Tests 2 through 8 averaged about 1500 dis/(min-ml).

Activity in samples taken immediately after the beginning of a test (before the supernate could have flowed through both columns) was often as high as that in samples taken at the end of the test. Thus, cell contamination limited the experimental DF's. To measure a reliable plutonium DF, feed for the last test was saturated with ^{238}Pu. The DF measured in this test (300) agreed well with previous test results.[4] A DF of 300 would give a product containing about 100 dis/(min-ml). If no sample contamination is assumed, the 1500 dis/(min-ml) of alpha activity is still well below the 22,000 dis/(min-g) level currently in effect for trench storage of alpha-contaminated waste. The limit for trench storage is based on naturally occurring ^{226}Ra concentrations in soil.[8]

Removal of ^{90}Sr by Ion Exchange and Carrier Precipitation

^{90}Sr was removed from waste supernate by the ion exchange column containing *Chelex-100* resin (Table 4). One exception occurred in Test 2, probably because the resin was not properly conditioned after the first test. After Test 2, the amount of HNO_3 used to elute the column was increased from 2.5 liters to 10 liters; DF values were consistently high.

TABLE 4

Removal of ^{90}Sr by Ion Exchange and Carrier Precipitation

Test Number	*Sorbent*	*^{90}Sr, dis/(min-ml)* Feed	*Product*	*^{90}Sr DF*
1	*Chelex-100*	1.5×10^6	2000	7500
2	*Chelex-100*	1.3×10^6	2×10^5	7[a]
3	0.01M $Sr_3(PO_4)_2$	1.3×10^6	1×10^5	13
4	0.005M $Fe(OH)_3$	2.3×10^6	6×10^4	38
5	0.01M $CaCO_3$	2.3×10^6	3×10^5	8
6	*Chelex-100*	2.3×10^6	550	4200
7	0.1 vol % HZO-1[b]	2.3×10^6	5×10^5	5
8	*Chelex-100*	2.3×10^6	300	7700
9	*Chelex-100*	1.8×10^6	1000	1800

---

[a] Resin not regenerated properly after Test 1.

[b] Hydrated zirconia gel from Bio-Rad Laboratories.

During supernate decontamination, the amount of ^{90}Sr in the effluent from the *Chelex-100* column did not increase as a function of throughput in any test. Flow rate was 2 resin bed volumes/hr (2.5 liters/hr) in all tests.

Analysis of eluate from the *Chelex-100* column showed that metals such as calcium, iron, manganese, and zinc are sorbed in addition to strontium. Sorption of these metals will load the resin to capacity faster than if only strontium were present. Some of these metals are more tightly bound to the resin than strontium; therefore, more acid is required to elute these metals from the resin than to elute strontium.

In a plant operation, online monitoring of ^{90}Sr will not be possible because its decay produces no gamma rays. Because the distribution coefficient for strontium on *Chelex-100* is much higher than that for cesium on *Duolite* ARC-359, eluting and regenerating *Chelex-100* resin at the same time the *Duolite* is conditioned will ensure that column operation is conservative and that ^{90}Sr does not pass through the decontamination process and contaminate the final salt cake.

Carrier precipitation was tested for removal of ^{90}Sr from supernate in Tests 3, 4, 5, and 7. (Carriers used are listed in Table 1.) Carrier precipitation during centrifugation would require less equipment and is a simpler process to operate than ion exchange; however, the precipitate would increase the volume of high-level waste sludge to be stored at least 10%. None of the four carriers tested gave a satisfactory DF.

Decontaminated Salt Cake

The radioactivity which would remain in a salt made by evaporating decontaminated supernate can be calculated from results in Tables 3 and 4. A liter of adjusted supernate contains 400 g of salt. After decay of ^{106}Ru, the residual activities in salt would be 3.1 nCi ^{137}Cs/g, 0.6 nCi ^{90}Sr/g, and <1.7 nCi Pu/g. The total residual activity, 5.4 nCi/g, would be about 14 times greater than ^{40}K activity in natural potassium chloride.

Radionuclide Fixation Onto Sorbents

In the conceptual waste solidification process (Figure 1) ^{137}Cs from the *Duolite* ARC-359 columns is sorbed onto zeolite for solidification in concrete. If glass is used as a final solidification matrix, concentrated column eluate will go directly to the glass works and will not be sorbed onto zeolite.

Eluate from the *Duolite* ion exchange process was concentrated by boiling to remove NH_3, CO_2, and H_2O. This concentrated eluate was used to test ^{137}Cs sorption onto columns containing four solid sorbents. Effluent from the sorbent columns was monitored by on-line gamma counting. Plutonium and ^{90}Sr could not be counted with the online system. Their sorption onto *Linde* AW-500* was shown previously.[4]

As shown in Table 5, *Linde* AW-500 and *Zeolon* 900** zeolites sorbed ^{137}Cs equally well. Each column held 100 ml of zeolite. Including concentration of ^{137}Cs by the *Duolite* columns and by eluate evaporation, each zeolite bed sorbed ^{137}Cs from about 150 liters of SRP supernate. The ^{137}Cs concentration factor was about 1500. (Sorption of ^{137}Cs from supernate directly onto zeolite gives a concentration factor of only 37.[3]) After being loaded with ^{137}Cs, the *Linde* AW-500 and the *Zeolon* 900 columns each emitted gamma radiation >4000 rad/hr at 3 in. *Zeolon* 500** and *Vermiculite*† were not acceptable sorbents for ^{137}Cs in these tests.

TABLE 5

Fixation of ^{137}Cs Onto Sorbents

	Composition	*Size*	^{137}Cs *Sorption*[a]
Linde AW-500	Chabazite	20 to 50 Mesh	100% retention for 70 CV 60% retention for 80 CV
Zeolon 900	Synthetic Mordenite	20 to 50 Mesh	100% retention for 70 CV 60% retention for 80 CV
Zeolon 500	80% Chabazite 20% Erionite	1/16 in. Pellets	No retention after 8 CV
Vermiculite	-	20 to 50 Mesh	No retention after 2 CV

a. Each column volume (100 ml) of concentrate contained ^{137}Cs from about 2 liters of SRP waste supernate.

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* Product of Union Carbide Company.

** Product of Norton Chemical Company.

† Product of Zonolite Company, Travelers Rest, SC.

APPENDIX: RADIONUCLIDE ANALYSES

Analyses of supernate which was decontaminated in these tests were complicated by high salt concentrations and relatively high ^{106}Ru activity in the samples. Methods of determining ^{137}Cs, ^{90}Sr, and alpha activity should be helpful in future work on supernate decontamination.

Cesium-137

Cesium-137 can be determined directly by gamma counting of the 661-KeV gamma ray of its ^{137}Ba daughter. Because radioactivity of ^{137}Cs in SRP waste supernate is about 1000 times greater than that of any other radionuclide, analysis is straightforward. After a ^{137}Cs DF of 10^5, however, ^{106}Ru is the dominant activity. Background from ^{106}Ru-^{106}Pd interferes with the ^{137}Cs analysis. This background can be reduced 90 to 95% by oxidizing ruthenium complexes in the supernate to volatile RuO_4, which is expelled from solution by boiling.

Supernate samples were neutralized by slowly adding concentrated H_2SO_4. The solutions were then made 1M in H_2SO_4. NO_2^- was destroyed by heating the acid solutions until they became clear. The solutions were cooled and made strongly oxidizing with a few crystals of periodic acid. Potassium permanganate was added as an indicator. Because RuO_4 reacts rapidly with organic materials, the beaker was covered with a piece of filter paper to minimize hood contamination. Solutions were then boiled 15 to 20 min until the permanganate was destroyed. Removal of ^{106}Ru in this manner allowed reliable ^{137}Cs analyses to be made.

Strontium-90

^{90}Sr activity was determined by carrier precipitation and liquid scintillation counting. Ingrowth of its ^{90}Y daughter showed that pure ^{90}Sr fractions had been obtained.

One ml of 0.1M $Sr(NO_3)_2$ carrier was added to 1 ml of supernate. About 10 ml of fuming HNO_3 was added, and the mixture was chilled in ice water for 20 min to precipitate $Sr(NO_3)_2$. The mixture was centrifuged, and the liquid was decanted. The $Sr(NO_3)_2$ was dissolved with 1 ml of water and reprecipitated with fuming HNO_3. Four precipitations were usually enough to separate ^{90}Sr from ^{106}Ru and ^{137}Cs. Gross gamma counting provided an initial check on purity of the precipitate. $Sr(NO_3)_2$ was dissolved in

2 ml of water and extracted with 2 ml of HDEHP (hexyl di-2-ethyl hexyl phosphoric acid) to ensure separation of ^{90}Y. One ml of the ^{90}Sr (aqueous) phase was then counted by liquid scintillation. Chemical yield was measured by titrating an aliquot of the remaining aqueous phase with EDTA and NH_3-arsenazo indicator.

One channel of the scintillation counter was set to detect beta particles below 0.5 MeV, the end point of the ^{90}Sr beta spectrum. A second channel detected beta particles having higher energy. Because ^{106}Rh (from ^{106}Ru) emits a 3.5-MeV beta particle, the ratio of first to second channel counts indicated the purity of the ^{90}Sr. A ratio of 10 was marginal; a ratio of 100 was good.

^{137}Cs also emits a 0.5 MeV beta particle. Observation of ^{90}Y grow-in was necessary to ensure that ^{90}Sr,rather than ^{137}Cs, was being counted.

Plutonium

Measurement of alpha particle activity was complicated by the large amounts of salt in the waste supernate. To reduce absorption of alpha particles by the salts, the salt was deposited in a very thin layer over the surface of the planchet.

A large drop of water was placed in the center of a 1.5-in.-diameter steel planchet. Ten μl of supernate was added to the water, and the pipette tip was used to distribute the solution over the planchet surface. A known activity of plutonium was sometimes added so that alpha absorption on a typical planchet could be measured. The planchet was then dried slowly. The dried planchet was gradually heated to red heat in a burner flame. This gradual heating was important to minimize spattering as the $NaNO_2$ and $NaNO_3$ decomposed. A thin, black film formed and was then fixed with a drop of collodion. Counting planchets containing a known alpha activity showed that, on carefully made plates, counting efficiency was decreased only about 20% by the salt.

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