ATLANTIC RICHFIELD HANFORD COMPANY
QUARTERLY REPORT
TECHNOLOGY DEVELOPMENT FOR LONG-TERM
MANAGEMENT OF HANFORD HIGH-LEVEL WASTE
JANUARY 1976 THROUGH MARCH 1976

Edited by

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June 1976

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This document is the seventh in a series of quarterly reports on work in support of long-term management of Hanford high-level wastes. The work reported here was performed during the period January through March 1976. The specific topics discussed are grouped into the subject areas of:

- *Storage System Integrity and Engineered Improvements*
- *Waste Retrieval*
- *Waste Immobilization and Storage*
- *Contaminated Equipment Volume Reduction*
- *Analytical Methods Development*

Previous reports were published as ARH-ST-110 A through D and ARH-ST-132 A and B.

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INTRODUCTION

The high-level radioactive wastes resulting from Hanford operations must be stored or disposed of in a manner that at all times positively assures the protection of the public and the environment. As currently envisioned, long-term Hanford waste management plans are directed toward future removal of waste from underground storage tanks, conversion of the waste to an improved stabilized form, and final storage of the immobilized waste in a location that does not require controlled management.

The entries in this report under Storage System Integrity and Engineered Improvements are directed toward establishing a data base and methodology for determining the length of time that waste can safely continue to remain in the tanks. This information will also be used to evaluate engineered improvements that might extend the safe in-tank storage period. Identification, development, and demonstration of the technology necessary for retrieval of the waste from underground tanks are discussed under Waste Retrieval. The Immobilization and Storage section of the report summarizes technological developments in the conversion of retrieved waste to an immobilized form satisfactory for final storage and evaluates alternative engineered and final storage methods. Contaminated Equipment Volume Reduction addresses the development and demonstration of methods for reducing the volume of radioactively contaminated metallic items to a size and form suitable for final storage or disposal.

Analytical Methods Development deals with the design and construction of equipment able to analyze trace levels of radioactive and nonradioactive elements in various forms of Hanford waste.

The Energy Research and Development Administration, Division of Environmental Control Technology, sponsors the Contaminated Equipment Volume Reduction program; others are sponsored by the Division of Nuclear Fuel Cycle and Production.

SUMMARY

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STORAGE SYSTEM INTEGRITY AND ENGINEERED IMPROVEMENTS

The objectives of this task are:

- *Creation of data base and methodology to permit annual assessment of the safety of continued waste storage in the underground tanks.*
- *Identification, development, and testing of engineered improvements that could be utilized to extend the safe storage period of underground tanks.*
- *Definition of the Hanford groundwater flow system and determination of the extent of groundwater management necessary to control the rate and direction of radionuclide movement in the high-level-waste storage areas.*

STORAGE SYSTEM INTEGRITY

Salt Cake Stability - G. A. Beitel and L. E. Bruns

The avoidance of exothermic reactions between salt cake nitrates or nitrites and organic materials is a prerequisite for continued safe storage of Hanford high-level wastes. Studies and tests have been initiated to determine if such reactions are possible.

Shock sensitivity tests have shown no evidence of shock-initiated detonation susceptibility. Combustion limit tests conducted with mixtures of sodium nitrate and organic material (resin or charcoal) have shown that ease of combustion varies depending on the mixture and its moisture content.

A report was completed on tests conducted by Atlantic Richfield Hanford Company laboratories to determine the potential for an exothermic reaction between salt cake and organic

compounds. The tests documented in the report were conducted on one-gram samples at atmospheric pressure. The influence of sample size and confining pressure was not investigated. From the tests performed, it was concluded that:

- Below 380° C, sodium nitrate is stable and will not support combustion.
- At moisture concentrations above 22 weight percent of sample, exothermic reactions in even the most energetic and reactive compositions will not propagate.
- Fresh resin and paraffin are too volatile at temperatures below 380° C to enable a sodium nitrate supported combustion process to propagate at atmospheric pressure.
- Concentrations of sodium nitrate above 95 weight percent of sample or below 35 weight percent do not react with enough energy release to support combustion.

A final report was also completed that evaluates the results of detonation tests conducted by Stanford Research Institute - Poulter Laboratory and the deflagration tests conducted by ARHCO. The report arrives at the following eight conclusions, all contributing to confidence that the salt cake is stable in regard to exothermic reactions:

- Organics added to the waste tanks were not nitrated at the time of addition and therefore cannot have been subsequently transformed into detonable nitrated organics.
- Whatever organic has found its way into the tanks has been and will be essentially unaffected by radiation.
- Mixtures of either simulated salt cake or pure sodium nitrate with the types of organics that could have been added to the waste tanks are not detonable.

- The maximum amount of organic that could have been added to the waste tanks is less than 0.9 weight percent of the salt cake, a concentration far below the concentration required to support combustion.
- The many years during which the liquid high-level waste was boiling, and the subsequent evaporation - crystallization processing, have allowed many of the more volatile organics to be distilled off, further reducing the maximum expected concentration of organics.
- The occurrence of an explosive exothermic reaction of an organic in the waste tanks would require concentration and mixing of the organic material by an unknown and uncontrolled means and then would require that the mixture remain in that state long enough to be triggered by a shock wave, a totally unreasonable hypothesis.
- Even if a combustible concentration existed, temperatures generated by radionuclide decay in the salt are by design below 175° C and therefore would be incapable of igniting any of these mixtures.
- If any major organic solvents were present in concentrations theoretically adequate to support combustion, they probably would not burn. Because of their volatility, they are more difficult to react than charcoal, the normal fuel in sodium nitrate base explosives, and may be impossible to ignite.

A key factor yet to be established is the concentration of organic material within the salt cake. Special efforts are being made to incorporate determinations of total organic carbon content into the salt cake characterization program. In addi-

tion, consideration is being given to the testing of one-kilogram samples in sealed, pressure-limited containers, with sample carbon compositions determined by the results of the salt cake characterization program. These tests would be designed to determine pressure dependence of volatile organics, paraffin and resin, in combustion with sodium nitrate. Tests are also being considered for 50- to 100-kilogram samples to determine the effects of sample size on combustion.

No further testing is planned during the next report period, pending the results of the salt cake characterization study.

Containment Integrity - F. R. Vollert

The extended use of Hanford waste-storage tanks will greatly depend on how seriously corrosion and elevated temperatures affect the tanks. Experimental programs have been developed to investigate corrosion of the mild steel liner and reinforcing bars and deterioration of concrete exposed to the variety of waste compositions found in Hanford tanks. Study of the effects of elevated temperatures on the elastic properties of concrete used in the tanks is also in progress.

The second quarterly report on the effects of simulated waste solutions on the type of concrete used in Hanford tank construction was received from the Portland Cement Association Laboratories (PCAL) in Skokie, Illinois. Experiments are being conducted at three temperatures: 50, 100, and 150° C. Concrete specimens are immersed in the following ten solutions at each temperature:

saturated calcium hydroxide; 4 N, 7 N, and 10 N sodium hydroxide; 4 N, 7 N, and 10 N sodium hydroxide, each mixed with one N sodium nitrate and one N sodium nitrite; 4 N, 7 N, and 10 N sodium hydroxide, each mixed with 3 N sodium nitrate and 3 N sodium nitrite.

Tests are made to determine changes in length, weight, and sonic modulus. Evidence from the change-of-length tests indicates that temperature and the normality of the sodium hydroxide have the greatest relative effect on durability. Figure 1 shows the rate of change in specimen length in 7 N sodium hydroxide and with sodium nitrate and sodium nitrite added in the normalities shown. The specimens tested at 150° C increased in length between 0.070 and 0.130 inches after one month and were not measured after two months of exposure because of cracking and deterioration. They were considered to have "failed." At 100° C, the specimens increased between 0.030 and 0.060 inches after three months of exposure. By comparison, the maximum change at 50° C was 0.004 inches after three months exposure. Two specimens containing reinforcing bar reacted in essentially the same way as the unreinforced samples.

A program proposal for the testing of concrete cores from Hanford waste tanks was received from PCAL. The program will determine the strength and elastic properties of the concrete, determine how these properties are affected by test-sample size and shape, perform petrographic examinations of the concrete's microstructure, and correlate the results of these tests with the results of a thermal-effects program presently in progress at the laboratory. The size of the cores limits the size of the test samples to 3-by-3-inch cubes and 3-inch-diameter, 6-inch-long cylinders. Six-inch-diameter, 12-inch-long cylinders are being used in the current tests. This program will begin during transition fiscal year 1976.

Metal-clad specimens are being considered for use during the next report period, instead of the plain concrete specimens now in use. The metal cladding will be cracked to allow only

FIGURE 1

CHANGE IN CONCRETE SPECIMEN
LENGTH CAUSED BY CHEMICALS
AND TEMPERATURE

a limited exposure of the concrete to the test solutions. This type of specimen is intended to simulate the condition of a cracked steel liner in the concrete tanks. Periodic testing will determine the extent of penetration of the solution behind the cladding and the behavior of the corrosion products in the crack.

ENGINEERED IMPROVEMENTS

Engineering Technology Survey - P. F. Mercier

This activity is based on the principle that the extended safe storage period in the underground tanks can be assured if an impervious barrier enveloping each tank can be developed.

A literature search covering the past ten years of documented methods and techniques for controlling groundwater in subsurface construction was conducted by the Battelle-Pacific Northwest Laboratories Technical Library. The resulting nine reports describe techniques utilizing a variety of soil additives which vary with soil particle size. Materials so far revealed as useful stabilizers include phenolic alcohols, lime-slurry, cement grout, silicate chemicals, chrome-lignins, bentonite clay, organic monomers, and asphalt emulsions. Combinations of the above have also been used successfully.

Seventeen companies specializing in the application of soil-stabilization engineering techniques to the design and construction of foundations, tunnels, mine shafts, and dams were asked for an expression of interest in developing a technique to provide a barrier to liquid migration in the soil surrounding the underground tanks. Discussions with the responding companies will be conducted to initiate a demonstration program in FY 1977.

Safety Analysis of Stored Solidified Waste - D. J. Brown

The purpose of this analysis, to be initiated next quarter, will be to determine the effects upon the biosphere of continued storage of the solidified waste in the single-shell tanks. The analysis will consider the radiologic impact upon man resulting from loss of containment through failure of the storage system as the result of an accident, sabotage, or natural phenomena.

A study as complex as this will use data and results from the numerous programs completed or in progress that are related to the safe storage of the solidified waste. Typical are hydrogeologic site characterization; studies of radionuclide - sediment reactions, plant - radionuclide interaction, animal - radionuclide interaction, water table management, atmospheric science records, and seismological history; and tank structural and seismic analysis.

The final report of the study will define the radiologic impact on man at the tank farm boundaries, the Hanford Site boundaries, and at specific offsite locations.

RADIONUCLIDE MIGRATION IN GROUNDWATER - R. A. Deju and R. E. Gephart

The objectives of this subtask are:

- *To conduct the field research necessary to better define the potential migration pathways of radionuclides leaving the high-level-waste storage areas through groundwater.*
- *To evaluate principal interactions between waste, sediment, and groundwater that are significant in the long-term isolation and/or transport of radioactive waste solutions.*

- *To determine the extent of radionuclide contamination in the Hanford groundwater environment.*

This program involves a three-part study. First, a geophysical gravity model is being developed and analyzed to identify the transmissive character of various geologic units below the water table. Second, the geophysical study is being complemented by the preparation of strip logs for approximately 100 wells. These strip logs are being used to prepare cross sections along portions of the Hanford Reservation. Using these cross sections, the hydrologic properties of each hydrologic unit will be identified. Third, past hydrologic records of water level, contaminant, and chemistry patterns will be used to determine the extent of contaminant migration through the groundwater pathway and the various sedimentary units that will be delineated with geophysics and strip logs.

During the report period, effort continued in interpreting the results of gravity readings taken on 350 stations over the Hanford Reservation. All gravity corrections have been compiled. Five gravity profiles have been constructed to determine the location of buried stream channels. The work to date reveals one major anticlinal structure, the Umtanum-Gable Anticline, separating two synclinal structures. This anticline appears to be a double anticline with a minor intervening syncline. Near the anticlinal axis, severe breaching has probably taken place. This breaching could provide a pathway connecting the upper sediment to lower confining aquifers.

These data are being prepared for an initial three-dimensional model of the Hanford Reservation. The model will assume that the subsurface of the Reservation consists of unsaturated, unconsolidated material separated by the water table from saturated, unconsolidated, and poorly consolidated material. The lower boundary of the saturated zone is assumed to be the

basalt surface. This three-dimensional model will assist in evaluating not only the location, but also the thickness of permeable buried streams of potential significance in the transport of radionuclides leaving the high-level-waste storage areas.

Samples of sediment materials from wells on the Hanford Reservation were used in the preparation of strip logs. A strip log consists of well samples taken at various depths that are attached to a sheet of cardboard in a continuous strip showing the vertical distribution of sedimentary material. A series of such strip logs from many wells will permit evaluation of stratigraphic variations. The sedimentary logs of the Ringold Formation and the glaciofluviatile material will, therefore, permit study of the movement of the groundwater and associated radionuclides in the unconfined aquifer. Strips have been prepared for 114 wells. One hundred and ten strips have been studied under the microscope and the sediment has been classified. Hydrologic characteristics will be ascribed to each individual sediment type.

Existing information on Hanford waste compositions and absorption properties of sediments was compiled. Additional information from the literature concerning trace element distribution during salt crystallization, weathering reactions of aluminosilicate minerals, and mass-transport models was assembled. This material will be used to identify gaps in our understanding of waste - sediment - groundwater reactions. Emphasis is being placed on determining which factors are important and which can be neglected. The effort will help in planning of subsequent laboratory experiments.

By the end of next report period, the modeling of gravity data should be nearly completed and a report on lithological strip logs should be issued. Work in assessing the chemistry of contaminant migration will continue.

LONG-TERM GROUNDWATER MANAGEMENT - R. C. Arnett, R. A. Deju,
R. E. Gephart, R. K. Ledgerwood

The objectives of this subtask are:

- *To identify potential ways in which the groundwater can come into contact with the high-level waste.*
- *To identify existing computer codes and models useful in predicting consequences of events identified in the objective above.*
- *To determine the data-gathering requirements needed for long-term prediction of subsurface contaminant migration from high-level-waste-storage areas into and through the groundwater flow system.*
- *To examine potential impact to the high-level-waste storage areas resulting from past, present, and projected activities at Hanford; potentially hazardous contamination accidents; and potential or existing agricultural development adjacent to or within the Hanford Reservation.*

A program has been initiated to evaluate computer models and data required for long-term groundwater management within waste-storage areas. A rather comprehensive analysis of finite element techniques as applied to groundwater flow problems is being made. Groundwater flow models available commercially are being examined for their usefulness at Hanford and are being compared to existing models developed onsite. Areas where significant deficiencies are noted in the computer modeling effort will be slated for additional development work. By recognizing all data requirements and having available the most suitable computer systems to handle this information, maximum credibility should be achieved in predictive efforts.

Twelve different computer codes potentially useful in this effort were identified during the report period and are being studied. These codes include work by the Illinois State Water Survey; Desert Research Institute of Las Vegas, Nevada; the U. S. Geological Survey (USGS); Lawrence Livermore Laboratories of Livermore, California; and a number of private companies and universities. Discussions were held during the report period with modelers from the USGS, the United States Bureau of Reclamation, and private industry to determine the availability and applicability of offsite groundwater codes to the Hanford groundwater management program.

The USGS has developed both three-dimensional and two-dimensional codes. These codes were made available to ARHCO and are presently being examined. The U. S. Bureau of Reclamation has developed a code that integrates flow in the partially saturated zone with flow in the saturated zone. Although their model is primarily used to trace salinity changes in the soil, portions of the work could be applicable to the Hanford groundwater flow system.

Most model development in private industry is of the three-dimensional variety, with emphasis on multiphase, multi-component flow models. Tests will be conducted in subsequent months to determine which models presently available commercially could be adapted to the groundwater management effort at Hanford.

Work was also conducted in conjunction with Battelle-Pacific Northwest Laboratories and Boeing Computer Services Richland, Incorporated, to determine what level of modeling capability is needed to support ARHCO's waste management effort, and which commercially available models appear most promising.

Drilling specifications for three research wells in the Cold Creek Valley were completed and a contract was awarded. Operations were started March 1, 1976. One well was completed just south of Umtanum Ridge and finished at 120 feet below ground surface, in the uppermost basalt. Two additional wells will be drilled. These wells will be used to determine the amount of water recharging the unconfined aquifer flow system and to evaluate the impact of increased irrigation west of the Hanford Reservation boundary. It remains to be determined whether such irrigation is likely to have an impact on the radionuclide plumes beneath the high-level-waste storage facilities.

WASTE RETRIEVAL

The objective of this task is to develop and demonstrate the technology necessary for retrieval of the waste material (salts, sludges, liquids, and accumulated debris) from the underground storage tanks. This technology will provide the Hanford High-Level Radioactive Waste Management Program with the option of removing the stored wastes to permit their conversion to a more acceptable product form.

The waste retrieval task was restructured at the beginning of the current report period. The restructuring was deemed necessary to increase the certainty that the final program objective would be accomplished on schedule. The program now consists of the following five subtasks:

- Concept and equipment development
- Engineering studies and conceptual design

- Engineering design
- Fabrication and construction
- Prototype test operations.

CONCEPT AND EQUIPMENT DEVELOPMENT - I. E. Reep

The objectives of this subtask are:

- *The definition of areas of the waste retrieval task which will require technology development and evaluation.*
- *Identification of potential retrieval techniques.*
- *Preliminary testing of the identified retrieval techniques to establish feasibility.*
- *Evaluation and reporting on engineering studies and designs performed in support of this subtask.*

The major activities of the concept and equipment development subtask are scheduled to be completed by the close of FY 1976. Potential retrieval methods have been identified and their feasibility has been established in "bench-scale" tests.

Hydraulic Retrieval - N. M. Arnold

The objective of this activity is to evaluate the feasibility of in-tank waste retrieval using hydraulic techniques. Two Scumbuster® (Vaughan Company, Inc., Montesano, Washington) agitator pumps are being evaluated for their ability to slurry salt cake in its own interstitial liquor and pump the resulting slurry out of the tank. This technique could

also be used for the retrieval of sludge if the moisture content is sufficiently high. A hydraulic retrieval test loop has been installed for use with the larger, 100-horsepower pump, which has successfully slurried and pumped synthetic salt cake containing up to 70 weight percent included solids.

Work is in progress on the design and fabrication of a mobile stand for use in testing the ability of a modified 60-horsepower Scumbuster® pump to remove solid radioactive wastes from the 75-foot-diameter Hanford waste storage tanks.

One objective of the test is to determine the pumping and flow characteristics of synthetic salt cake slurries as a function of the percentage of solids in the slurry. One possible application of the hydraulic concept being tested is illustrated in Figure 2.

ENGINEERING STUDIES AND CONCEPTUAL DESIGN - I. E. Reep

This subtask consists of:

- *Engineering studies in problem areas requiring further definition, and*
- *Conceptual designs for the further development of concepts and equipment identified as feasible in the Concept and Equipment Development subtask.*

A number of engineering study reports have been received from consultants. These include an engineering study and cost estimate for a waste retrieval system; a design study of a salt cake retrieval system; a study evaluating the adaptability of

FIGURE 2

SLURRY POOL METHOD OF RETRIEVAL

standard mechanical, hydraulic, and pneumatic devices to salt cake and sludge retrieval at Hanford; and a study on the high-level waste management technology development program. This information will provide a basis for the conceptual design of a full-scale waste retrieval system.

Waste Retrieval System - H. A. Wallskog

The objective of this activity is to provide the conceptual design of a full-scale, completely integrated waste retrieval system having the capability of retrieving waste from any of the 75-foot-diameter Hanford waste-storage tanks.

Preparation of functional design criteria and conceptual design of such a waste retrieval system was initiated during the report period. A contract with Programmed and Remote Systems Corporation (PaR), St. Paul, Minnesota, to execute engineering design studies and conceptual designs, received preprocurement approval. One concept under consideration, using mechanical excavation (clamshell shovel) and mechanical removal (bucket and elevator), is illustrated in Figure 3. A hydraulic retrieval option for this system is illustrated in Figure 2.

Prototype Testing Facility - N. M. Arnold

The objective of this activity is the conceptual design of a facility for use in testing and demonstrating the capability of a complete waste retrieval system in a nonradioactive environment. Design of the cold test stand, a metal structure that would permit limited testing of retrieval system components, has been completed and is being held for future reference.

V7606-2.2

FIGURE 3

COMPOSITE SECTION OF A CONCEPTUAL
WASTE RETRIEVAL SYSTEM

The cold test stand will be constructed only if lengthy delays are expected in completing the prototype test facility.

Conceptual design of the prototype test facility, which is part of the Long-Term Waste Management Equipment Test Facility (described under Contaminated Equipment Volume Reduction), was initiated by Vitro Engineering during the report period. The conceptual design will be completed during the next report period.

IMMOBILIZATION AND STORAGE

The objectives of this task are:

- *Identification, development, and demonstration of technologies for converting high-level waste to an immobilized form satisfactory for long-term or final storage.*
- *Evaluation of alternative engineered and final storage methods.*

AQUEOUS SILICATE PROCESS

The conversion of fission product wastes to solid, insoluble products suitable for long-term or final storage is being investigated through the Aqueous Silicate process. This process utilizes reactions of clay with waste to form stable products. Basically, three processing options are available in the Aqueous Silicate process, as described in previous reports of this series. During this report period development was directed exclusively toward the high-temperature version - Clay Calcination. In this process dry waste materials (such as salt cake) or other dehydrated wastes are reacted with clay at

600° to 800° C to form water- and nitrate-free sodium aluminosilicate powders. These powders may then be incorporated with binders or sintered to form clay calcination products.

Effect of Temperature on Properties of Sintered Clay
Calcination Products - G. S. Barney

The best products yet produced by the Clay Calcination process were obtained when sintering temperatures were increased to approximately 1200° C.

Three types of synthetic waste - salt cake, Purex sludge, and Redox sludge (both sludge compositions reported previously⁽¹⁾) - were each reacted with kaolin at 750° C. An optimum clay/waste ratio (determined from previous work) was used in these reaction mixtures. After the reaction, the powdered products were pressed into pellets and sintered at two different temperatures (1150° and 1222° C) to determine the effect of these sintering temperatures on density, volume, abrasion resistance, and leach rate of the sintered products. (Previous sintering temperatures had been limited to $\leq$ 1000° C.) In addition, the two types of sludge wastes were sintered without clay to permit their properties to be compared to products from the same sludges that were reacted with clay. Several products were also identically prepared to determine the reproducibility of the data.

The properties of these products are shown in Table I. Most products were greatly improved when sintered at the higher temperature (1220° C). For Redox sludge sintered at 1150° C, however, the leach rates show essentially no improvement over

TABLE I

PROPERTIES OF SINTERED CLAY CALCINATION PRODUCTS

Waste Type	Percent Waste in Reaction Mixture ^a	Sintering		Density, g/ml	Abrasion Resistance ^b mg/min	Volume Increase, % ^c	Leach Rate, g/cm ² -day ^d	
		Time hrs.	Temp °C				1 Day	13 Days
Redox Sludge	100	4	1220	1.62		-37	1.0×10^{-1}	1.6×10^{-3}
Purex Sludge	100	4	1220	1.87		-61	5.3×10^{-3}	8.6×10^{-6}
Redox Sludge	50	2	1150	1.19	325	127	2.1×10^{-3}	1.0×10^{-4}
Redox Sludge	50	4	1220	2.26	0.23	19	7.3×10^{-6}	$<8.4 \times 10^{-7}$
Redox Sludge	50	4	1220	2.37	0.23	14	8.6×10^{-6}	$<8.5 \times 10^{-7}$
Purex Sludge	50	4	1150	1.47	418	30	6.2×10^{-3}	
Purex Sludge	50	4	1220	1.46	16	31	4.4×10^{-3}	1.4×10^{-5}
Purex Sludge	50	4	1220	1.50	16	28	3.1×10^{-3}	1.3×10^{-5}
Salt Cake	30	2	1150	1.96	32	95	9.7×10^{-4}	5.4×10^{-5}
Salt Cake	30	2	1150	1.96	32	95	9.5×10^{-4}	5.7×10^{-5}
Salt Cake	30	4	1220	2.47	0.15	55		

^a Kaolin was used in each sample (Georgia Kaolin - Astra Glaze).

^b Weight of material abraded per minute.

^c Calculated using the following densities: Redox Sludge = 1.97 g/ml
Purex Sludge = 1.40 g/ml
Salt Cake = 1.6 g/ml

^d Based on amount of Cs⁺ leached.

those of similar products previously sintered at 1000° C.⁽¹⁾ This temperature-sensitive effect is illustrated in Figure 4.

The Redox sludge sintered without prior reaction with clay yielded a very poor product. The leach rates were very high and the sample disintegrated during leaching. Reaction of this sludge with clay greatly improved the properties of products from the sludge. However, leach rates were only slightly improved when Purex sludge was reacted with clay. This surprising result will be studied further.

Cesium Volatility During Clay Calcination Reactions -

C. H. Delegard

During the last report period two cesium volatility determinations were made for clay calcination reactions with salt cake. During this report period, studies of the cesium and ruthenium volatility of clay calcination reactions of salt cake were conducted using both synthetic salt cake and genuine salt cake from the 102-SX waste tank. Charges containing from 25 to 50 weight percent salt cake (the balance of the charge being kaolin) were reacted at temperatures beginning at 650° C and ending at 800° to 1000° C. The quantities of cesium and ruthenium reporting to the off-gas train for these reactions were determined.

The salt cake content of the reactant charge had a significant effect on the amount of cesium found in the off-gas, as shown in Table II. It is clear that as the weight fraction of salt cake in the reaction mixture approaches and exceeds the stoichiometry necessary to form the crystalline nepheline product (~ 0.42 weight fraction), volatilized cesium increases

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FIGURE 4

CLAY CALCINATION AND SINTERING TREATMENT
OF REDOX SLUDGE: EFFECT ON PRODUCT LEACH RATES

TABLE II

CESIUM VOLATILIZED AS A FUNCTION OF SALT
CAKE CONTENT IN CLAY CALCINATION REACTIONS (1000° C)

<u>Salt Cake</u>	<u>Fraction Cesium Volatilized</u>				
	<u>0.25^a</u>	<u>0.30^a</u>	<u>0.35^a</u>	<u>0.40^a</u>	<u>0.50^a</u>
Synthetic	0.0076	0.052	0.030	0.1	0.54
102-SX	0.0064	0.021	0.033	0.065	

^aWeight fraction salt cake in reaction mixtures.

dramatically. Ruthenium volatilized from the genuine salt cake in these experiments ranged from 30 to 60 percent and showed no apparent correlation with clay/salt cake ratio.

Experiments also showed that volatilized cesium could be decreased significantly by lowering the final reaction temperature from 1000 to 900 to 800° C. Table III shows the observed cesium-volatility behavior as a function of reaction temperature. Again, ruthenium volatilities were high (~ 50 percent).

TABLE III

CESIUM VOLATILIZED AS A FUNCTION OF
REACTION TEMPERATURE IN CLAY CALCINATION REACTIONS

<u>Salt Cake</u>	<u>Fraction Cesium Volatilized</u>		
	<u>800° C</u>	<u>900° C</u>	<u>1000° C</u>
Synthetic, 0.25 ^a	0.00047	0.0012	0.0076
Synthetic, 0.40 ^a	0.013	0.045	0.14
102-SX, 0.40 ^a	0.0078		0.065

^aWeight fraction salt cake in reaction mixtures.

Leach rate data (not shown) clearly indicate that increasing salt cake content in the reaction mixture increases cesium leach rates as rapidly as it increases cesium volatility.

SILICATE MELT PROCESS - M. J. Kupfer, C. W. Hobbick, W. W. Schulz

The objective of this subtask is to develop and demonstrate processes for incorporation of Hanford high-level wastes into immobile silicate-based glass forms. Present laboratory-scale glass-making activities are aimed at (a) development and demonstration of suitable processes for conversion of Hanford sludges and terminal liquors to glasses, and (b) construction and operation of a small-scale (10 - 25 kg of glass per day) all-electric, continuous melter for eventual hot-cell use with high-level waste.

In previous work the effects of various chemical additives on the viscosity and durability of low-temperature ($< 1000^{\circ}\text{C}$) Redox sludge-glass melts were evaluated. Tests were initiated to define the optimum formulations for high-melting ($> 1000^{\circ}\text{C}$) Redox sludge-glasses.

Low-Temperature Glasses Made with Redox Sludge

Based on results of earlier scouting tests and using the same synthetic sludge composition, four candidate glass formulations for fixation of Redox sludge at 1000°C were chosen for further detailed evaluation.⁽²⁾

Because of the difficulties encountered in producing acceptable glasses at 1000°C using 40 weight percent of the

highly refractory, high-chromate Redox sludge, the maximum sludge content used in the four formulations was 32 weight percent. Lithium oxide was used in all of the formulations because it reduces viscosity markedly, enabling complete dissolution of all of the components at 1000° C, and permitting easy pouring.

The formulas produce visually acceptable brown-black homogeneous glasses. There is no Na_2CrO_4 phase separation from the melts as was observed with earlier Redox formulations.⁽²⁾ The range of concentrations of the glass-forming ingredients is narrow, however, and differs primarily in the relative amounts of flux (Li_2O and $\text{Na}_2\text{B}_4\text{O}_7$) and CaO .

Leach-rate data and qualitative observations on viscosity are given in Table IV for these Redox glass formulations. Typical leach rates in deionized water (25° C) range from about 10^{-6} to 10^{-5} g/cm²-day after 120 hours of leaching. As indicated by the data, addition of CaO to the Redox sludge formulations appears to do little to improve product durability (leachability).

Low-Temperature Glasses Made with Purex Sludge

Eight glass formulations were tested for fixation of Purex sludge, the synthetic composition of which is given in Table V.

Glass compositions and leach-rate test results are given in Table VI. Half the formulations were made identical to those for Redox sludge-glass (Table IV) to test the feasibility of using the same glass-making formulations for immobilization of either Redox or Purex sludge or mixtures of the two.

TABLE IV
 REDOX SLUDGE-GLASS FORMULATIONS
 (LOW-TEMPERATURE GLASSES)

Formula	Sludge	Components, wt. %				Viscosity at 1000° C 1-5 ^b	Leach Rates, g/cm ² -day			
		SiO ₂	Na ₂ B ₄ O ₇ ^a	Li ₂ O	CaO		Based on Sr		Based on Cs	
							24 hrs.	120 hrs.	24 hrs.	120 hrs.
1	32	32	32	4	-	2	3.6×10^{-6}	2.1×10^{-6}	2.4×10^{-5}	1.1×10^{-5}
2	30	32	26	5	7	2	2.7×10^{-5}	6.9×10^{-6}	2.0×10^{-5}	5.0×10^{-6}
3	30	35	23	7	5	1-2	$< 2.7 \times 10^{-6}$	1.0×10^{-6}	$< 3.9 \times 10^{-6}$	2.4×10^{-6}
4	30	35	25	4	6	2-3	1.4×10^{-5}	5.6×10^{-6}	1.6×10^{-5}	6.4×10^{-6}

^aNa₂B₄O₇ = 31% Na₂O, 69% B₂O₃

^b1-runny, 2-very pourable, 3-pourable, 4-hardly pours, 5-melted, but thick

TABLE VCOMPOSITION OF SYNTHETIC PUREX
SLUDGE FOR GLASS-FIXATION EXPERIMENTS

<u>COMPONENT</u>	<u>WEIGHT %</u>
Fe_2O_3	38.2
Na_2CO_3	26.7
Al_2O_3	15.5
SiO_2	8.4
MnO_2	3.4
CaO	2.4
SrO	1.7
Trace Elements	<u>3.7</u>
	100.0

TABLE VI
PUREX SLUDGE-GLASS LEACH RESULTS
(LOW-TEMPERATURE GLASSES)

Formula	Sludge	Components, wt. %				Leach Rates, g/cm ² -day			
		SiO ₂	Na ₂ B ₄ O ₇	Li ₂ O	CaO	Based on Sr		Based on Cs	
						24 hrs.	120 hrs.	24 hrs.	120 hrs.
1	32	32	32	4	-	< 3.5 x 10 ⁻⁶	8.8 x 10 ⁻⁷	4.4 x 10 ⁻⁵	1.7 x 10 ⁻⁵
1a	40	28.25	28.25	3.5	-	< 2.6 x 10 ⁻⁶	< 6.6 x 10 ⁻⁷	3.5 x 10 ⁻⁵	1.7 x 10 ⁻⁵
2	30	32	26	5	7	3.1 x 10 ⁻⁵	1.3 x 10 ⁻⁵	1.0 x 10 ⁻⁴	4.8 x 10 ⁻⁵
2a	40	27.4	22.3	4.3	6	1.6 x 10 ⁻⁵	6.5 x 10 ⁻⁶	4.8 x 10 ⁻⁵	2.5 x 10 ⁻⁵
3	30	35	23	7	5	9.9 x 10 ⁻⁶	2.8 x 10 ⁻⁶	7.8 x 10 ⁻⁵	4.3 x 10 ⁻⁵
3a	40	30	19.7	6	4.3	9.1 x 10 ⁻⁶	5.9 x 10 ⁻⁶	2.8 x 10 ⁻⁵	1.5 x 10 ⁻⁵
4	30	35	25	4	6	2.6 x 10 ⁻⁵	1.2 x 10 ⁻⁵	1.1 x 10 ⁻⁴	5.4 x 10 ⁻⁵
4a	40	30	21.4	3.4	5.2	8.6 x 10 ⁻⁶	2.9 x 10 ⁻⁶	1.8 x 10 ⁻⁵	4.8 x 10 ⁻⁶

Substitution of synthetic Purex sludge for Redox sludge in the formulations in Table IV results in visually acceptable glasses. The viscosities of the melts at 1000° C are much lower for the Purex formulations, however. This is expected because of the relatively lower Al_2O_3 content of the Purex sludge, making it much less refractory than the Redox sludge.

Leach rates for the Purex glasses based on strontium are nearly the same as those found for Redox glasses (Table IV). Surprisingly, however, rates are significantly lower for formula 1 which does not contain lime (CaO) in the formulation. Leach rates based on cesium are about 2 - 20 times higher for the Purex glasses than for the Redox glasses. There is also a slight but unexpected improvement in leach rates based on cesium or strontium when the Purex sludge content is increased from 30 weight percent to 40 weight percent (compare rates for 1 vs. 1a, 2 vs. 2a, etc. Table VI).

Viscosities and electrical conductivities of both Redox and Purex sludge-glasses will be determined in the near future in order to establish the feasibility of using identical glass formulations for both sludges in a continuous electric melter.

High Temperature Glasses Made with Redox Sludge

Development of high-melting (> 1000° C) Redox sludge-glass formulations continued with definition of reactant proportion ranges necessary for production of dense, single-phase homogeneous glasses at 1200° C. Suitable reactant proportion ranges for immobilization of 22 - 40 weight percent synthetic

Redox sludge are as follows:

$\text{Na}_2\text{O} + \text{B}_2\text{O}_3$:	26 - 38 weight percent
Range of B_2O_3 :	9 - 20 weight percent
Range of Na_2O :	14 - 20 weight percent
Remainder:	SiO_2

As little as 5 weight percent B_2O_3 can be used if Na_2O content is 20 weight percent and the weight percent of sludge does not exceed 27.

Formulations outside of these range boundaries produced poorly melted specimens or products containing a separate water-soluble Na_2CrO_4 phase.

Leach rates in deionized water (25° C) were determined for selected specimens prepared using the range boundaries defined above. After 120 hours of leaching the rate based on strontium ranged from 3.7×10^{-7} to 1.3×10^{-6} g/cm²-day. Leachability showed little or no dependence on sludge content.

Leach rates based on cesium will be determined in the near future for these formulations. Substitution of Purex sludge for Redox sludge in the formulations will also be evaluated.

OTHER IMMOBILIZATION CONCEPTS

The objective of this subtask is to identify, develop, and evaluate alternative processes (other than the Aqueous Silicate or Silicate Melt processes) for conversion of Hanford high-level waste to immobile forms suitable for long-term storage.

The primary immobilization techniques to be investigated in the laboratory include, but are not limited to:

- *Glass sintering*
- *Concrete*
- *Pressed concrete (at hot and ambient temperatures)*

In previous work, scouting studies were initiated to determine the application of a process for sintering synthetic sludge into a glass matrix.

Glass Sintering - M. J. Kupfer

Determination of optimum sintering temperatures for cold-pressed pellets of synthetic Redox sludge powder mixed (50:50) with various crushed glass powders was described in the previous progress report.⁽¹⁾ During the present report period optimum sintering temperatures were determined for 50:50 mixtures of synthetic Purex sludge and crushed glass and also for sludge/glass mixtures containing 75 weight percent synthetic Redox or Purex sludge. Generally the specimens were very hard but quite porous. Some specimens were cracked or slightly flaked. The volume of a typical sintered product containing 75 weight percent sludge was only about 55 percent of the volume of the original sludge powder.

Leach rates were determined for specimens containing 50 and 75 weight percent Redox or Purex sludge sintered in the optimum temperature ranges. The rates after 120 hours leaching in deionized water (25° C) ranged from 6.0×10^{-6} to 3.5×10^{-4} g/cm²-day when based on strontium. In general, leachabilities were lowest for products sintered at the higher temperatures. The sintered Purex products leached at slightly lower rates than the Redox products. Surprisingly, leach rates were not dependent upon sludge or on the type of glass frit used.

Leach rates based on cesium, however, were extremely high (about 10^{-2} g/cm²-day). There is evidently little or no solid-state reaction between cesium and the glass structure.

Fixation of Sludge in Cement - M. J. Kupfer

Fixation of synthetic Redox sludge in portland and high aluminum cement was described earlier.⁽²⁾ Scouting tests are now complete for fixation of synthetic Purex sludge in cement. Table VII gives the composition (on an anhydrous basis) of synthetic Purex sludge material prepared for use in the tests. The sludge powder was mixed thoroughly with types I or III portland cement or with calcium aluminate cement using sludge/cement weight ratios of 30:70 and 40:60. Water content was adjusted to give an ideal consistency. The pastes were placed in plastic molds, tamped to remove air spaces, and cured for 28 days in a humid atmosphere.

The Purex cement specimens were leached in deionized water at room temperature using the International Atomic Energy Agency proposed standard leaching technique. After 1000 hours of leaching, the rates based on strontium ranged from 1.2×10^{-4} to 3.6×10^{-4} g/cm²-day. Products containing 40 weight percent sludge leached at only slightly higher rates than products containing 30 weight percent sludge. There was no apparent relationship between leachability and the type of cement used. The leach rates for the Purex products were similar to those observed earlier for similar products containing simulated Redox sludge. A few Purex cement samples exhibited rates about 50 percent lower than equivalent Redox cement samples, however.

Rates based on cesium, after 1000 hours leaching, were determined for cement products containing 40 weight percent sludge. The rates ranged from 1.5×10^{-3} to 3.0×10^{-3} g/cm²-

TABLE VII
COMPOSITION OF SYNTHETIC,
WASHED, DRIED PUREX SLUDGE^a

<u>Component</u>	<u>Weight Percent</u>
Fe	22.4
Al	8.3
Si	6.4
Na	4.8
Mn	2.5
Sr	1.0
Ca	0.5
PO ₄ ³⁻	2.2
H ₂ O (measured)	26.0
O ₂ (calculated)	26.0

^aThe Purex hydrated oxide sludge waste was prepared by adjustment to pH 9 of an aqueous solution of Fe(NO₃)₂, Mn(NO₃)₂, and Sr(NO₃)₂ containing a slurry of Al₂O₃·3H₂O, CaHPO₄, and cancrinite. The precipitate was washed three times with water and dried at 110° C.

day, or were about a factor of 10 higher than rates based on strontium.

The effectiveness of sludge fixation in cement is reported to be improved by cold-pressing the cement-sludge paste before curing. Cement-pressing techniques (both hot pressing and cold pressing) have been described in detail by workers at Pennsylvania State University.^(3,4) They have reported products showing adequate resistance to hot-water leaching, low porosity, and compressive strengths which are five to ten times those of normal concrete.

To explore this possible process variation, synthetic Redox or Purex sludge and Portland cement (type I or III) or calcium aluminate cement were mixed together (one part sludge/two parts cement) in powder form, a small amount of water was added (about 1/3 of that used for normal cement pastes), and then the mixed wet "paste" was cold pressed at 20,000 psi. The pellets were subsequently cured in a humid atmosphere (> 95 percent) for 28 days.

The volume of a typical product was about 40 percent less than the volume of an unpressed specimen containing an equivalent amount of sludge, thus representing an increase in volume of about 1.6 times the volume of the sludge.

Pressed product leach rates (based on strontium) in deionized water after 1000 hours of leaching ranged from 3×10^{-4} to 6×10^{-4} g/cm²-day. The rates were nearly equivalent for products containing the three different cements. Purex and Redox sludge specimens exhibited nearly equivalent leach rates.

Although the product volume was significantly improved (reduced) by the cold-pressing technique, the product's resistance to strontium leaching apparently was not. Final judgment will be reserved, however, until compressive strengths for pressed cement forms can be determined and compared to those for unpressed specimens.

RADIONUCLIDE REMOVAL STUDIES

These studies are concerned with devising, developing, and demonstrating processes for removal of all long-lived radionuclides from Hanford salt cake solutions and, possibly, from terminal liquors. The overall objective is to develop and demonstrate technology for partitioning such high-level wastes into a small volume of highly radioactive waste requiring further immobilization for long-term storage and a large volume of chemical or very low-level wastes which can be safely stored or disposed of by inexpensive means.

Current studies are focused on:

- *Screening and testing, with synthetic waste solutions containing appropriate radiotracers, various sorption and ion exchange processes and procedures for removal of ^{137}Cs , ^{90}Sr , ^{99}Tc , and actinides.*
- *Testing radionuclide removal procedures with actual waste solutions as they become available.*

Process Development Studies for Cesium Removal -

J. E. Hammelman, W. W. Schulz

Cesium removal by Duolite ARC-359® (Diamond Shamrock Chemical Company, Cleveland, Ohio) ion exchange resin was investigated and engineering design calculations were made showing column size and performance. The major process design variables are cesium feed concentration and hydroxide ion concentration. Results indicate that cesium/sodium salts (eluate) can be concentrated by a factor of greater than 50 relative to feed salts.

The equilibrium data used to calculate column performance are shown in Figures 5, 6, and 7 which show K, the equilibrium

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FIGURE 5

K_{Cs} AND K_{Sr} FROM SYNTHETIC SALT CAKE SOLUTION
[Na^+] = 7 g-equivalents/liter

FIGURE 6

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K_{Cs} AND K_{Sr} FROM SYNTHETIC SALT CAKE SOLUTION
 $[OH] = 1$ Molar

FIGURE 7

K_{Cs} AND K_{Sr} AS A FUNCTION OF AQUEOUS EQUILIBRIUM
CESIUM OR STRONTIUM CONCENTRATION RESPECTIVELY

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loading constant, as a function of varying levels of nitrate, hydroxide, and solute. These curves have been generated from previously reported laboratory data and with an equilibrium loading constant, K_j , defined as:

$$K_j = \frac{Q_j C_{Na}}{C_j Q_{Na}}$$

Q_j = solute j concentration on resin (g-equivalents/kg resin)

C_j = solute j concentration in solution (g-equivalents/liter)

Q_{Na} = solute Na concentration on resin (g-equivalents/kg resin)

C_{Na} = solute Na concentration in solution (g-equivalents/liter)

Strontium Phosphate Scavenging from Synthetic Solutions -

W. W. Schulz

Previous work with both synthetic and actual waste solutions has demonstrated the effectiveness of precipitating inert strontium phosphate for removal of ^{90}Sr from salt cake solutions. A statistically designed (Plackett-Burman design) experiment was performed during the report period to screen six scavenging variables to determine which, if any, affected the carrying of ^{90}Sr by the inert strontium phosphate precipitate. Variables tested were (1) precipitation temperature, (2) digestion time, (3) hydroxide concentration, (4) carbonate concentration, (5) phosphate concentration, and (6) concentration of added inert strontium. The experimental procedure involved adding $\text{Sr}(\text{NO}_3)_2$ to NaNO_3 - NaOH - Na_2CO_3 - Na_3PO_4 solutions containing ^{85}Sr at either 25° C or 60° C and stirring the resulting precipitate for either 0.5 or 6.0 hours. The ^{85}Sr content of the final scavenged solution was determined, and the strontium decontamination factor (DF) was calculated.

The strontium DF results were used to estimate experimental error and to determine which of the variables was

significant. This analysis showed that, over the range studied, the strontium DF was determined by the concentrations of $\text{Sr}(\text{NO}_3)_2$ and Na_3PO_4 and the digestion temperature and was independent of digestion time and the concentrations of NaOH and Na_2CO_3 .

Strontium and Technetium Removal from Actual Waste Samples -
W. W. Schulz

For removal of ^{90}Sr , actinides, and possibly other long-lived cations (e.g., ^{93}Zr) investigators are currently considering scavenging salt cake solutions by precipitation of inert $\text{Sr}_3(\text{PO}_4)_2$, sorption on Sandia Titanate beds, or combinations of both methods. Results of multiple scavenging of actual salt cake solutions at both low (0.04 - 0.2 M) and high ($\sim$ one M) NaOH concentrations are recorded in Table VIII. The ^{90}Sr concentration of solutions prepared by dissolving Tank 105-S and 105-B salt cakes was reduced to less than one $\mu\text{Ci/liter}$, tentative removal criteria, by a single precipitation of 0.00225 M $\text{Sr}_3(\text{PO}_4)_2$. On the other hand, four consecutive precipitations of 0.00225 M $\text{Sr}_3(\text{PO}_4)_2$ reduced the ^{90}Sr concentration of solutions from Tanks 102-SX and 103-SX to only 3 to 6 $\mu\text{Ci/liter}$.

Sorption of ^{90}Sr on Sandia Titanate Resin. Results obtained previously both at Hanford and at Sandia Laboratories - Albuquerque have demonstrated that Sandia Titanate ion exchanger has a great affinity for ^{90}Sr and other cationic radionuclides in alkaline salt cake solutions and terminal liquors.

Further evidence attesting to this affinity for ^{90}Sr is presented in Table IX. According to these results, the ^{90}Sr concentration of two-fold scavenged Tank 102-SX and 103-SX salt cake solutions was reduced to below 1.0 $\mu\text{Ci/liter}$ by passage of these solutions through one bed of the titanate

TABLE VIII
SCAVENGING OF STRONTIUM FROM
ACTUAL SALT CAKE SOLUTIONS^a

Tank Number	Initial ⁹⁰ Sr μCi/l	OH ⁻ M	Scavenged Solution, ⁹⁰ Sr, μCi/l				Volume Solids Scav 1 ^b
			Scav 1	Scav 2	Scav 3	Scav 4	
105-B	61.0	0.2	0.93				0.6
105-S	22.9	0.2	0.13				0.3
		0.2 ^c	0.26				
		2.1	0.59				0.3
102-SX	1820	0.17	76.3	25.6	14.5	6.36	0.25
		1.0 ^{c,d}		17.2			
103-SX	1780	0.04	68.6		12.5	2.79	0.25
		0.04 ^e	21.6	11.9	4.54	5.76	0.45
		1.0	61.9	8.02	3.49	3.56	0.25
		0.85 ^{c,d}		16.2			

^a Unless otherwise indicated, salt cake solution adjusted, if necessary, to indicated OH⁻ concentration, and made 0.025 M H₃PO₄ and 0.0075 M Sr(NO₃)₂; resulting precipitate digested one hour at 25° C and then centrifuged. This scavenging procedure repeated indicated number of times.

^b Wet centrifuged solids from 30 ml salt cake solution at 0.00225 M Sr₃(PO₄)₂.

^c 250-ml scale experiments.

^d At 60° C.

^e With 0.015 M Sr(NO₃)₂.

TABLE IX
REMOVAL OF ^{90}Sr FROM SALT CAKE SOLUTIONS
BY SORPTION ON SANDIA TITANATE^a

Salt Cake Solution		Column 1			Column 2		
Tank Number	NaOH M	Feed ^{90}Sr $\mu\text{Ci}/\ell$	Col. Vols	Effluent ^{90}Sr $\mu\text{Ci}/\ell$	Feed ^{90}Sr $\mu\text{Ci}/\ell$	Col. Vols	Effluent ^{90}Sr $\mu\text{Ci}/\ell$
105-S	1.0 ^b	18.3	22	< 0.084			
102-SX	1.0 ^c	17.2	23	0.238			
103-SX	0.85 ^c	16.2	15	0.516	0.516	15	0.470

^aEach resin bed 1.05-cm diameter x 11.5-cm high. Downflow at one column volume/hour at 25° C.

^bPreceded by passage of solution through three 10-ml beds of Duolite ARC-350® resin.

^cPreceded by duplicate precipitations of 0.00225 M $\text{Sr}_3(\text{PO}_4)_2$ (Table VIII) and passage of scavenged solution through three 10-ml beds of Duolite ARC-359® resin.

exchanger. Tests to determine effectiveness of Sandia Titanate resin for removal of ^{90}Sr from unscavenged Tk 102-SX and 103-SX salt cake solutions are in progress. As noted in Table IX, passage of unscavenged Tk 105-S salt cake solution through a bed of Sandia Titanate resin easily reduced its ^{90}Sr concentration below one $\mu\text{Ci/liter}$.

Removal of ^{99}Tc . For ^{99}Tc the current goal is to devise procedures for reducing the ^{99}Tc concentration of salt cake to 5 $\mu\text{Ci/liter}$ or less. This goal can be easily achieved, as indicated by the data in Table X, by passage of the salt cake solution through a bed of strong-base anion exchange resin. Dowex MSA-1® (Dow Chemical Company) is expected to be a superior resin for TcO_4^- removal because of its macroporous structure and associated more rapid exchange rate.

Removal of Sodium by Solvent Extraction - G. S. Barney

As an alternative backup technology to the radionuclide removal processes discussed above, studies were continued to evaluate solvent extraction as a means of separating inert sodium salts from salt cake and terminal liquor wastes.

The search for an extractant specific for sodium salts led to the choice of crown ethers having the 15-crown-5 ring. (Crown ethers with the larger 18-crown-6 ring extract appreciable amounts of both cesium and strontium, along with sodium salts, although strontium extraction can be greatly reduced by adding small amounts of EDTA to complex the Sr^{++} ion.)

Two crown ethers having the 15-crown-5 ring have been tested as possible extractants. They are 15-crown-5 and t-butylbenzo-15-crown-5. Many solvent extraction experiments have shown that the 15-crown-5 is quite soluble in the aqueous phase of the salt cake solution. This high aqueous solubility probably eliminates 15-crown-5 from consideration as a practi-

TABLE X
REMOVAL OF ^{99}Tc FROM SALT CAKE SOLUTIONS BY
SORPTION ON ANION EXCHANGE RESINS

Salt Cake Solution		Resin Type	Column Run ^a		Col. Vols
Tank Number	NaOH M		Feed ^{99}Tc $\mu\text{Ci}/\ell$	Effluent ^{99}Tc $\mu\text{Ci}/\ell$	
105-S					
Test 1	1.0	Dowex 1-X4 ^b	9.25	0.689	24
Test 2	1.0	Dowex MSA-1 ^c	5.60	0.0769	13
102-SX	1.0	Dowex MSA-1	13.3	0.346	22.5
103-SX	1.0	Dowex MSA-1	9.3	0.114	13

^aColumns were 1.05-cm diameter x 11.5-cm high; downflow at $\sim 25^\circ \text{C}$ and $\sim$ one column volume/hour.

^bMicroporous 20-50 mesh, NO_3^- -form.

^cMacroporous, 20-50 mesh, OH^- -form.

cal extractant. However, the t-butylbenzo-15-crown-5 is not soluble in the aqueous phase because of its large, hydrophobic side group on the polyether ring. It therefore appears to be a reasonable choice.

Of the solvents tested, those having the most desirable properties are: 0-dichlorobenzene, 1,2,4-trichlorobenzene, and, possibly, chloroform. Extraction coefficients (E_a^O 's) for sodium, cesium, and strontium have been determined for 1.0 M t-butylbenzo-15-crown-5 in 1,2,4-trichlorobenzene. Various dilutions of a synthetic salt cake solution were equilibrated with this extractant to determine the effect of Na^+ concentration on E_a^O . The results are shown in Table XI.

TABLE XI
EFFECT OF Na^+ CONCENTRATION ON E_a^{Oa}

<u>$[Na^+]$, M</u>	<u>$10^2 \times E_a^O$</u>		
	<u>Na</u>	<u>Cs</u>	<u>Sr</u>
5.1	8.3	0.77	7.1
3.9	7.2	0.67	8.2
2.8	4.2	0.44	11
1.5	4.4	0.61	20

^aExtraction coefficients (E_a^O 's) determined for 1.0 M t-butylbenzo-15-crown-5 extractant in 1,2,4-trichlorobenzene solvent.

Extraction coefficients for sodium are about ten times greater than for cesium. This difference in E_a^O is probably great enough to permit an effective separation of sodium from cesium. Although strontium is strongly extracted by the organic phase, strontium extraction can be prevented by the addition of a small amount of complexing agent such as EDTA. Most actual waste solutions will already contain strontium complexing agents from various processes.

The extractant must be able to remove the major types of sodium salts contained in the waste if it is to be effective. Therefore, the extraction of NaNO_3 , NaNO_2 , and NaOH with 15-crown-5 in butyl alcohol was tested. Separate aqueous solutions of the salts (each at 1.0 M) were contacted with this extractant and the concentration of sodium in the organic phase was measured. The results (not shown) indicated that each of the salts is extracted by the organic phase, with extraction coefficients which decrease in the order:

This is the predicted order since large anions such as NO_3^- have a low charge density and are therefore more easily extracted into solvents having relatively low dielectric constants.

Stripping sodium salts from the organic phase with water appears to be feasible. In tests using 0.76 M 15-crown-5 in butanol the concentration of sodium in the organic phase was reduced from 0.45 M to < 0.0038 M after two contacts with water (equal volumes of water and organic).

Process Development for Solids - Liquid Separation - P. C. Ely

A planning document is being prepared with the objective of presenting timetables, proposed funding, and experimental programs designed to provide for separation of solids from solutions in a radionuclide removal process.

The many different solids - liquid separation requirements result in the following possible separation steps:

- Clarification of salt cake solution prior to processing for radionuclide removal. This step is required only if surges of high-solids-content salt cake solution from the recovery operation are to be expected.
- Precipitate removal. This step is required only if a strontium phosphate precipitation process is used.
- Polishing of salt cake solution prior to ion exchange processing. This step will be required for all proposed radionuclide removal flowsheets.
- Polishing of salt cake solution prior to discharge from the radionuclide removal process. This step will be required only if contaminated solids pass through the ion exchange columns.
- Thickening of sludge prior to processing. This step will be required only if there is excessive solution present in recovered sludge.
- Thickening of sludge after it has been washed. This step will be required only if a sludge washing process is used.

The techniques being considered for the various possible solids - liquid separation steps are listed below. Each of these techniques will remain in the program only as long as it remains viable for one of the possible separation steps.

- Gravity sedimentation
- Dissolved air floatation
- Centrifugation
- Cross flow filtration
- Ultrafiltration
- High-gradient magnetic separation

The characterization and initial solids - liquid separation feasibility work will be performed by the ARHCO laboratories, with some preliminary solids - liquid separation feasibility work being performed offsite. The techniques and equipment demonstrated to be effective in these preliminary tests will ultimately be pilot-plant tested to obtain final process design information and to assess the remote operability of the equipment.

ENGINEERING ANALYSIS OF WASTE STORAGE SYSTEMS

Heat Transfer Analysis of Immobilized Waste Storage Systems - D. D. Chen

Heat generated from the storage of immobilized radionuclide wastes in subsurface vaults or underground double-shell tanks may be harmful to concrete and steel structures. The present objective of this subtask is to evaluate the maximum permissible heat dissipation capacity of these alternative storage systems.

In preliminary work, heat removal from arrays of canisters filled with glass waste was calculated. Modifications to an existing computer program are under way to permit a more definitive prediction of the temperature distribution in double-shell tanks.

During the coming quarter emphasis will be placed on the analysis of double-shell tanks as they might conceptually be used for the bin-like storage of immobilized terminal liquor or sludge wastes in glass-marble form.

CONTAMINATED EQUIPMENT VOLUME REDUCTION

The objective of this task is to develop and demonstrate methods for reducing the volume of radioactively contaminated metallic items to a size and form suitable for interim or final storage. Although the problem of disposition of contaminated equipment exists throughout the nuclear industry, this program is focusing its attention on the contaminated decommissioned equipment presently located in the chemical processing areas at Hanford.

DECONTAMINATION EVALUATION - G. A. Beitel

The purpose of this subtask is to evaluate costs and secondary waste generation associated with equipment decontamination as a precursor to volume reduction. A decontamination test on a Purex dissolver has been planned to provide this information.

During February 1976, the equipment to be used in the decontamination test was assembled and a work plan was formulated. A complete radiation survey of the dissolver was made. The surface radiation dose ranged from 300 mR/hour near the top to a maximum of 5 R/hour in several spots near the bottom. The intent of this test is to reduce the surface dose to less than 50 mR/hour, maximum, at any point on the vessel. The first decontamination cycle, a caustic permanganate soak with a steam sparge, followed by an oxalic acid neutralization soak, was begun in early March 1976. No decontamination results are yet available.

SIZE-REDUCTION EVALUATION - G. A. Beitel

Several tools have been selected for testing as size-reduction alternatives. These include the plasma arc furnace torch, the arc saw, and the radio-frequency plasma arc torch.

A demolition demonstration was conducted by Schlienger, Inc., San Rafael, California, on the remaining half of the heat exchanger used in the plasma arc furnace torch demonstration described previously.⁽¹⁾ The test conducted by Schlienger involved the use of an innovative tool known as an arc saw, a high power (low voltage, high current) spark erosion machine. The demonstration was completed in February 1976.

The results of the arc saw demonstration were impressive and promised improved size-reduction capability over any instrument observed to date. The 2000-kg heat exchanger was cut into about 40 pieces (not counting hundreds of short pipe segments), averaging about 40 kg each, in 100-120 minutes of cutting time. Even so, the cutting time was much longer than would have been required if either the saw or the vessel had been more maneuverable (improved maneuverability is possible). The nominal cutting power was 100 kilowatts. During the entire demonstration, jamming of the saw was never encountered, even when cutting through a variety of materials, multiple thicknesses simultaneously, and loose pieces and tubes. Since there is no mechanical contact between the saw and work piece, no mechanical forces are transferred, and, once a piece is cut off, it merely falls away. Even when the vessel shifted during the cutting operation, the arc saw merely slowed down in its vertical cutting speed and cut a wider kerf to accommodate the

shift. The arc saw used in the demonstration had a 36-inch-diameter, 1/4-inch-thick blade, and made cuts up to 12 inches deep and approximately 3/8 inch wide.

VOLUME REDUCTION EVALUATION - G. A. Beitel

This task attempts to determine whether radioactively contaminated equipment can be safely melted and cast as a product acceptable for final storage. The specific objective is the design of a low-maintenance, remotely operated meltdown system incorporating maximum fume control. In support of this work experimental meltdown of contaminated stainless steel pipe samples has yielded promising decontamination results.

Hot laboratory meltdown studies, using pieces of contaminated stainless steel pipe, continued during the report period. A decontamination study was performed using a flux made from a flint glass cullet whose approximate composition was 55 weight percent silica, 19 weight percent calcium oxide, 15 weight percent aluminum oxide, 7 weight percent boric oxide, 3 weight percent magnesium oxide, and one weight percent sodium oxide. Contaminated sections of a stainless steel pipe from a process facility were melted in the molten flux. The stainless steel samples weighed approximately 200 gm; the ratio of metal to flux in three runs was 1:1, 1:2, and 2:3.

The decontamination factors were very interesting. The isotopes present at detectable levels ($\sim$ one nCi/gm) with half lives greater than 5.5 years (^{90}Sr , ^{137}Cs , and ^{241}Am) experienced decontamination factors of about 200 (except for a unique incident involving ^{90}Sr where no ^{90}Sr was detectable in the flux).

Those isotopes with half lives less than 5.5 years (^{60}Co , ^{106}Ru , ^{125}Sb , ^{144}Ce , and ^{154} , ^{155}Eu) became fairly uniformly distributed between the flux and the ingot. These results are shown in Table XII. If this behavior can be extended to other transuranics (it is the anticipated behavior with plutonium) then it should be possible to reduce almost all metallic waste to non-transuranic waste. Although interesting, such a decontamination ability is of no real benefit unless the weight of the slag can be held to a few percent of the metal and a system can be devised to provide a 99 percent, or better, separation of slag and metal.

The decision was made to proceed with the design and acquisition of a water-cooled, copper crucible vacuum furnace. Negotiations are under way to obtain a design contract with Schlienger, Inc., of San Rafael, California.

LONG-TERM WASTE MANAGEMENT EQUIPMENT TEST FACILITY (formerly
WASTE MANAGEMENT ENGINEERING PROTOTYPE AND TEST FACILITY) -

J. M. Davis

The Energy Research and Development Administration has asked Atlantic Richfield Hanford Company to proceed with the conceptual design of a Long-Term Waste Management Equipment Test Facility. The facility will consist of an office building for approximately 200 engineers and scientists, and a high-bay building in which equipment will be tested and developed for use in the various long-term waste management programs undertaken by ARHCO.

The conceptual design plan and functional design criteria documents were approved by ERDA's Richland Operations Office late in January, and Vitro Engineering started conceptual design early in February 1976. The documentation of the conceptual design will be completed by the end of June 1976.

TABLE XII

DECONTAMINATION FACTORS FOR CULLET FLUX

Radionuclides Present with Less than 5.5 Yr Half Life

Radionuclide (quantity in μCi)	Sample I			Sample II			Sample III		
	Ingot (295 gm)	Cullet (200 gm)	Fumes	Ingot (180 gm)	Cullet (180 gm)	Fumes	Ingot (185 gm)	Cullet (90 gm)	Fumes
^{60}Co	5.5	1.6	Not determined	1.4	0.4	ND	0.7	0.2	ND
^{106}Ru	63.6	5.4		83.3	5.4	0.6	19.5	5.1	ND
^{125}Sb	3.5	ND		1.7	1.3	0.4	1.5	ND	0.2
^{144}Ce	0.8	12.6		2.0	212.4	ND	0.1	17.7	ND
$^{154} \& ^{155}\text{Eu}$	ND ^a	4.8		ND	9.2	ND	ND	0.8	ND
Total	73.4	24.4		88.4	228.7	1.0	21.8	23.8	.2
Decontamination Factor	1.3			3.6			2		

Radionuclides Present with Greater than 5.5 Yr Half Life

	Sample I			Sample II			Sample III		
	Ingot	Cullet	Fumes	Ingot	Cullet	Fumes	Ingot	Cullet	Fumes
^{90}Sr ^b	1.2	107.4	Not determined	0.5	96.5	0.1	7.8	ND	0.1
^{137}Cs ^c	0.2	49.8		0.5	66.1	9.1	0.1	8.5	8.2
^{241}Am	1.9	524		4.6	806.4	ND	0.3	15.8	ND
Total	3.3	681.2		5.6	969.0	9.2	8.2	24.3	8.3
Decontamination Factor	200			160			5		

^a None detected^b Values include a small fraction of ^{89}Sr ^c Values include a small fraction of ^{134}Cs

ANALYTICAL METHODS DEVELOPMENT

SUBCRITICAL ASSEMBLY FOR LOW-LEVEL FISSILE ANALYSIS - W. H. Zimmer

The primary objective of this project is the analysis of low levels of fissile material. A particular need exists at Hanford for the nondestructive assay of fissile material, in any media, at a level as low as 10 nanograms fissile material (specifically ^{239}Pu) per gram of sample, a factor of 19 below the maximum fissile concentration for discardable waste. The secondary task of this project is analysis of long-lived beta-emitting fission products and nonradioactive elements through the use of standard neutron activation analysis techniques.

The californium multiplier (CFX-40) was the neutron source chosen for this project because it is capable of multiplying the spontaneous neutron flux from 40 miligrams of ^{252}Cf to a flux density sufficient to attain program goals in a subcritical assembly. Samples will be irradiated briefly in the core of the assembly, causing the fissile contents of the sample to fission. A neutron counter (DNC) will count the delayed fission-product neutrons to determine fissile content of the sample. The neutron flux of the CFX-40 will also activate long-lived beta-emitting fission products and nonradioactive elements, permitting detection of these waste constituents at the part-per-billion level without additional investment of money or manpower. A pneumatic sample-transfer system, using molded capsules as sample-transfer vehicles (rabbits), will connect the CFX-40 with the DNC and other detector equipment.

Several phases of long-term, high-level-waste management at Hanford will benefit from this analytical capability. These include projects in waste retrieval, characterization, and immobilization, as well as efforts to gather information on the long-term fixation of radionuclides in soil.

IRT Corporation of San Diego, California, was chosen to design and fabricate the CFX-DNC system. Their contract with ARHCO for performance of this work was under review by the ERDA-Richland Operations Office throughout the report period.

The United States National Bureau of Standards 1.3-inch-diameter pneumatic transfer capsule was adopted as the sample-transfer vehicle. Marlex BMB 5040® (Phillips Petroleum, Incorporated, Bartlesville, Oklahoma) ethylene copolymer was chosen as the capsule molding material on the basis of its extremely low uranium content. Uranium in the capsule material limits the assay of sample concentrations of fissile material. A molding order for 10,000 capsules was awarded to the Wilks Precision Instrument Co., Inc., of Rockville, Maryland.

The pneumatic transfer system will be purchased from Reactor Experiments, Inc., of San Carlos, California. The system will contain an automatic sample loader, an irradiation terminus, and four detector termini. All points will be connected by flexible polyethylene tubing. A central processing unit (CPU) designed to meet project requirements will control the transfer system. Specifications for a computer to fulfill this function were released for competitive bidding.

During the next report period, design and procurement will begin for several CFX-DNC system support components. The pneumatic transfer system and CPU will be installed. Scheduling will be completed for the series of steps required for acquisition of 40 milligrams of ²⁵²Cf by April 1977.

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