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PROGRESS IN TREATMENT
OF A RADIOACTIVE CONDENSATE WASTE

By

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Process Demonstration and Analysis
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PROGRESS IN TREATMENT
OF A RADIOACTIVE CONDENSATE WASTE

INTRODUCTION

This report describes the experimental evaluation of a radioactive condensate waste decontamination process on a small engineering scale.

The management of wastes in nuclear fuels reprocessing plants requires consideration be given to six waste streams. These are the high-level wastes, the process condensates, the steam condensates, the cooling water, the cell drainage, and the laboratory wastes. Since the cell drainage and laboratory wastes are low volume but highly contaminated streams, they will probably be combined with the high level wastes. The resulting dilution of the high-level waste will be overcome by evaporation, and the volumes of the process condensate and steam condensate streams will be increased accordingly. The cooling water and steam condensate wastes will be segregated, discharged to holding basins for monitoring, and then either discharged to the environment or combined with the process condensate if excessive radiocontaminants are present; the most likely one to be contaminated is the steam condensate which is similar in composition to the process condensate. Thus, only the high-level wastes and process condensate waste will require routine treatment.

Process condensate is an intermediate-level waste both in volume and radionuclide content. To dispose this waste safely to the environment, decontamination factor requirements of 100 are ordinary and 1000 are not unusual. These decontamination factors must be routinely and economically achieved on a volume of waste which is greater than 95% of the total treatable volume from a plant.

Research and development studies at Hanford and Oak Ridge indicated that one way to obtain high capacity and high decontamination is by ion-exchange. These studies, generally conducted on wastes spiked with selected radionuclides, revealed many potential ion-exchange materials for a decontamination process. If actual waste was used, the samples were not of

sufficient size or the experiments were not conducted on a scale large enough to evaluate potential processes. To provide this information a program was considered necessary in which a condensate stream from a reprocessing plant would be used as feed for process studies.

SUMMARY

A waste stream decontamination process based on ion-exchange was selected for development. The process is centered on ion-exchange technology developed during recent years in the Hanford Laboratories and other atomic energy research centers. Early attempts at small engineering scale application of ion-exchange technology resulted in less than satisfactory results when compared to laboratory data. The difficulties were traced to nonradioactive impurities in the wastes. Steam stripping and filtering successfully removed these impurities, and excellent decontamination of all significant isotopes except ruthenium was achieved by ion-exchange. Satisfactory ruthenium decontamination was demonstrated on waste volumes up to about 1000 column volumes, and the capacity for other isotopes ranged from 10,000 to 20,000 column volumes. Improvement in ruthenium decontamination is needed to simplify operations and to improve economics.

Although a large scale integrated process is yet to be demonstrated, a cost estimate of an ion-exchange process was made. The estimate of \$7.50/1000 gal was based on a 2 ton/day fuels reprocessing plant which produces about 30,000 gal/day of condensate requiring treatment. The estimate was based on a process for alkaline condensate and it was assumed a process for acid condensate would cost about the same. This estimate is about \$10/1000 gal less than the estimated cost for reevaporation.

DISCUSSION

Purpose of Study

Figure 1 illustrates a nuclear process waste stream flow plan and highlights the fact that two waste treatment processes are required—one for the high-level wastes and one for the low-level wastes. The high-level waste is characterized by a high radionuclide content, a high salt content, and a relatively small volume.

FIGURE 1
Example of Waste Management Flow Plan

The low-level waste is of large volume and generally contains less than 0.01% of the fission products. The radioisotope concentration is about 10^{-2} to 10^{-3} μ curies of beta and gamma emitters per ml. This waste is mostly condensate and is formed when high-level solutions are concentrated in the reprocessing plant and when high-level wastes self boil in underground tanks. Waste solidification processes also will produce copious volumes of condensate that will require treatment even though much effort will be made to suppress radionuclide carryover.

Some condensate problems can be minimized. Reducing chemicals, such as NaNO_2 can be added to boiling, acidic solutions to suppress the formation of volatile ruthenium tetroxide. Recycle of a highly contaminated condensate for reevaporation or for dilution of a high-salt content stream is another possibility; however, use of condensate as a diluent may recycle trace quantities of troublesome chemicals such as degraded organic material. Even though condensate treatment requirements will be lowered by such techniques, a large volume will still have to be decontaminated before the waste can be released to the environment, and the decontamination must fit economically into the fuel cycle.

Multiple effect evaporation is one processing technique that can be used for waste treatment. The success of a reevaporation process is assured and information is available to predict the cost of the process. The technique is flexible and simple; the potential of any other treatment process may be measured against reevaporation. The incentive for developing alternate processes is primarily economics. Since approximately 10,000 to 15,000 gal of condensate are produced per ton of fuel reprocessed by conventional aqueous processes, appreciable savings can be achieved by lowered unit processing costs.

Application of ion-exchange technology developed at Hanford and other Atomic Energy Commission sites appeared to be a method of lowering unit waste processing costs. Technology developed at Hanford before 1959 on the removal of radioisotopes from waste streams was directed to a better understanding of the ion-exchange and other processes that occur in the soils at Hanford. An outgrowth of this work was the discovery that

certain minerals can selectively remove radioisotopes from wastes. (1, 2) Laboratory work was initiated to evaluate one of the minerals, clinoptilolite, for removing radiostrontium and radiocesium from condensate wastes. (3) At the same time, the need to study a treatment process on a small engineering scale was recognized and the design and installation of appropriate equipment was started.

Waste Pretreatment

Organic matter in the waste, especially in emulsified form, tends to be mechanically removed by the ion-exchange beds. The emulsion globules fill the void spaces in the bed and cause excessive pressure drop after only a fraction of the adsorption capacity of the bed is used. Plugging of the bed by organic material is characterized by the formation of a distinct layer of an orange colored, wax-like material in the top inch of the bed.

Significant quantities of NH_4^{+*} reduce the efficiency of cesium removal because Cs^+ is similar to NH_4^+ . Early during the waste treatment experiments, the presence of NH_4^+ had not been recognized; consequently, much work was done on removing only the organic matter. Two methods were tried: activated carbon adsorption and membrane separation. (4) The first method successfully removed both soluble and emulsified organic, although there were several limitations to its use. Membrane separation was expected to remove only emulsified organic. The method worked with synthetic or simulated waste, but the emulsion in the actual waste was too stable for separation. When it was determined that NH_4^+ was also a constituent of the condensate waste, it was realized that a method for removing both organic matter and NH_4^+ was necessary to increase the life of ion-exchange beds. Steam stripping had been used for removing organic material from plutonium and uranium stream, (5, 6) but only with concentrations where large evaporation factors were used. NH_4^+ removal has been demonstrated with conventional deaerators from boiler feed waters using a similar technique. (7)

* It is recognized that either NH_3 or NH_4^+ will exist in the waste system depending on the pH and the stoichiometry of the stream. NH_4^+ has been used throughout this report for consistency.

A steam stripper for the removal of gross amounts of NH_4^+ and organic impurities from condensate waste was designed and fabricated. The unit was operated at a boil-off rate of about 5% of the feed flow rate. In general, the stripper removed > 97% of the NH_4^+ and organic impurities. Some of the radioiodine and radoruthenium are volatilized and travel with the overhead stream. These isotopes concentrate in the organic phase, and the aqueous phase can be released to the environs without any further special treatment.

Although the steam stripper bottom streams are very clear, residual organic matter is present as a filterable particulate. A filtering step is considered necessary to protect ion-exchange beds that are to be regenerated and reused.

Radioisotope Forms Present in Waste

With nonradioactive impurities reduced to relatively low concentrations in the steam stripper bottoms, the waste can be decontaminated by ion-exchange adsorption. Choice of adsorbent is dependent upon the ionic forms of the radioisotopes. The cesium in the waste is present almost entirely as Cs^+ . The waste leaving the stripper in the bottom stream has a pH of 5 to 7. At this pH the strontium, cerium, and zirconium are primarily positively charged cations; however, 1 to 3% are nonionized. The pH must be reduced to less than 4 to ionize these elements more completely. Ruthenium is present primarily in complexed forms. These complexes of ruthenium are chiefly anions.

Cesium Removal

Cs^+ can be removed from steam stripper bottoms by conventional cation exchange resins. The capacity of a strong acid cation resin for removing cesium from this waste stream is dependent upon the residual Na^+ and NH_4^+ . The mineral clinoptilolite has been studied for some time at Hanford and has been used to remove cesium from the steam stripper bottom stream. In general, more waste can be treated by clinoptilolite than by a strong acid cation resin before Cs^{137} concentration in the effluent stream increases. The greater capacity of clinoptilolite is due to its crystal

structure. The crystal structure is near optimum dimensions for selective cesium removal even when larger amounts of NH_4^+ and Na^+ are present. Little work was done with synthetic alumino-silicate materials, but it is believed that they have cesium capacities comparable to clinoptilolite although they may not be as selective. These materials are limited to near neutral solutions, since high or low pH degrades most of the alumino-silicate zeolites.

Strontium, Cerium, and Zirconium Removal

The radioisotopes of strontium, cerium, and zirconium can also be removed by cation exchange. The efficiency of removal, however, is significantly related to the pH of the feed. Strontium, for example, can be removed with a decontamination factor of 500 when the steam stripped waste at a pH of 6 to 7 is passed through a bed of strong acid cation resin. However, if the pH is reduced to 4 or less with HNO_3 , the decontamination factor is increased to 5000. Cerium and zirconium radioisotopes are also removed under the conditions which remove strontium effectively.

Ruthenium Removal

Ruthenium removal is difficult by an adsorption process. In view of the complex chemistry of ruthenium this is not without expectation. In a separations plant, ruthenium is present in solutions of high HNO_3 concentration and significant nitrite and nitrate complexes of the element are formed. When the acid solutions are neutralized with caustic, water-soluble or colloidal complexed polymeric species of ruthenium may be formed. It is exceedingly difficult and time consuming to determine the exact form in which the ruthenium is present. Adsorption studies showed, however, that 10 to 20% of the ruthenium was removed when the waste was passed through a bed of strong acid cation resin. When waste was passed through a bed of strong base anion exchange resin, ruthenium removal was 80 to 90%. When the two types of resin were used in series or together in mixed beds, >99% of the ruthenium was removed during the early part of an experiment. While the initial removal efficiency for ruthenium was good, the capacity of the two bed system for ruthenium was relatively low. The

reduction in efficiency of ruthenium removal appears to be a function of non-radioactive anion concentration.

Integrated Treatment Process

A possible process for treating a waste such as Purex tank farm condensate is illustrated in Figure 2. The waste containing NH_4^+ and organic material along with a number of radioisotopes is fed to a steam stripper tower. The overhead vapors, representing about 5% of the waste, contain most of the NH_4^+ and organic impurities. Some radioiodine and radoruthenium are also present. The organic phase, which represents about 0.02% of the waste, contains most of the ruthenium and iodine in the overhead stream, and further processing may be necessary. The aqueous phase contains most of the NH_4^+ and almost none of the radioisotopes; it can be disposed to the environment without further treatment.

The bulk of the waste leaves the stripping tower as bottoms. This stream is cooled and is passed through a precoated filter. A 100 x 400 mesh cesium-selective adsorbent such as clinoptilolite or Decalso is used as a filter aid. This filtering step removes particulate matter and radiocesium. The adsorbent cake is discharged for burial whenever its cesium capacity is exhausted or whenever the pressure drop across the filter reaches a predetermined point. The filtered waste is then passed through a strong base anion bed which removes an appreciable amount of the ruthenium along with NO_3^- and NO_2^- . Periodically this bed is regenerated with caustic. The regenerant solution is discharged to underground storage. Since alkali in excess of the stoichiometric amount must be used, the regenerant solution could be used for neutralizing high-level acid waste. The waste leaving the anion bed is acidified to a pH of 4 before it enters the cation bed which removes strontium, cerium, zirconium, residual cesium, and other cationic radioisotopes. The cation resin has an extremely high capacity for these radioisotopes, and can economically be discharged for burial. The cation bed effluent is sufficiently decontaminated that it can be safely discharged to the environment. If the residual acid needs to be neutralized, the effluent can be combined with the aqueous overhead stream containing NH_4^+ .

FIGURE 2
Condensate Decontamination Flow Diagram

Treatment Costs

Evaporation is an available method for the treatment of condensate streams; the primary incentive for research and development of other treatment processes is economics. Unit costs for evaporative processes in service at several sites are available;⁽⁸⁾ and, since evaporative processes are widely used, cost estimates can be made with reasonable accuracy for proposed evaporative processes. Table I, lists these costs; a plot of these data (Appendix E, Figure E-1) reflects that as the capacity of an evaporative system increases, the cost levels-off at \$15 to \$20 per 1000 gal treated. These numbers simply reflect the basic cost of the heat load and, unless the cost of heat can be reduced markedly, little can be done to lower the costs of evaporative plants.

TABLE I
EVAPORATOR COST COMPARISON

<u>Site</u>	<u>Volume Treated/Day, gal</u>	<u>Cost, \$/1000 gal *</u>
Brookhaven	1000	103
Knolls	3000	65
Bettis	4600	38
Hanford Study	30,000	17 (est.)

* All costs normalized by amortizing the capital investment over 20 years.

The price for steam alone at Hanford is \$1/1000 lb or \$8.30/1000 gal of waste evaporated if a 1 to 1 conversion is attained. Actual conversion is probably 2 lb of steam consumed for every 1.25 lb of water evaporated.

Costs for flocculation and precipitation treatment processes at Rocky Flats and Los Alamos were estimated at \$9 and \$12 per 1000 gal treated, respectively (adjusted for a 20 year amortization).⁽⁸⁾ An estimate for a 15,000 gal/day flocculation plant at Hanford indicated costs would be about \$9 per 1000 gal. These chemical treatment processes are applicable to low-level wastes which contain appreciable amounts of inorganic salts; in fact,

successful decontamination depends on the presence of these salts. (These processes would not be useful to condensate streams unless additional chemicals were added; however, their costs are included in this discussion for comparison purposes.)

The estimated cost for the process shown in Figure 2 is \$7.50/1000 gal of waste treated which is less than one-half of that estimated for an evaporative process. The \$7.50/1000 gal figure includes utilities, chemicals, adsorbents, labor, adsorbent burial, maintenance, and plant amortization. Labor costs are based on using one operator half time (84 hr/wk, 52 wk/yr). It is assumed that he can be productively used elsewhere the other half time. Maintenance is estimated at 5%/yr of the \$359,000 investment, and amortization of this capital over 20 yr adds another 5%/yr.

It is assumed that 2 ft³ of precoat zeolite adsorbent and 1 ft³ of cation resin will be used for each 150,000 gal of waste treated. Anion resin loss is estimated to be about 2% per cycle. The effluent from the anion resin is to be acidified with HNO₃ to a pH of 3.5 to 4 before being fed to the cation resin bed. Caustic soda will be used to regenerate the anion bed and to neutralize the effluent from the cation bed. The regeneration of strong base anion resins requires a large excess of caustic soda; consequently, the anion bed regenerant can be used for neutralizing high-level wastes. Therefore, no charge was made for disposing of the regenerant solution. A charge of \$3.20/ft³ was assigned for burial of used adsorbents; this includes a storage container and burial space. Appendix E contains a summary of cost information.

Future Work

Future effort will be directed toward demonstrating an integrated process for decontaminating alkaline tank farm condensate by combining the techniques described in this report. Additional demonstration runs of treatment processes for other type wastes will follow along with the evaluation of other process techniques. The pilot scale demonstration runs will be based on chemical flow sheets and on supporting information from laboratory studies. The long term objective of this development effort is to provide integrated waste treatment processes so that most low- and intermediate-level wastes can be safely and economically disposed to most environs.

Research effort is being directed toward studying ruthenium and improving removal of ruthenium from wastes. Ruthenium removal is especially difficult due to its many species and their interactions with other constituents in the wastes. Studies are aimed at selection of ion-exchangers that may improve ruthenium's decontamination factor, or that may have a high capacity for NO_3^- and NO_2^- which appear to limit ruthenium capacity. Adsorption of ruthenium on preformed flocs, minerals, charcoal, and metal surfaces will also be examined.

Electrochemical reactions of ruthenium in wastes are also being examined, particularly electrodialysis data obtained with a laboratory electrodialysis unit indicate that ruthenium can be separated and concentrated. An engineering scale unit is being installed in the Waste Treatment Pilot Plant and will be used with other processing equipment to study ways of decontaminating low- and intermediate-level wastes.

DESCRIPTION OF PUREX TANK FARM CONDENSATE WASTE

Source of Waste

A simplified flow diagram of a Purex tank farm waste facility at Hanford is shown in Figure 3. Neutralized high-level radioactive wastes and organic wash wastes are pumped from the Purex Plant to a diversion box from which they can be routed to any of six underground storage tanks. Vapors produced by radioactive decay heat in the stored waste are routed to an entrainment removal vessel and then to one of three surface condensers. The liquid collected in the entrainment vessel drains to one of the storage tanks. The vapor condensate from the condenser is routed to a 40,000 gal underground retention tank. The condenser cooling water is sent to a swamp (surface disposal pond); the condenser is vented through a deentrainment tower and then to a local stack; any deentrained liquid is discharged to a crib (a subsurface excavation filled with gravel and covered by earth).

Most of the aqueous condensate collected in the 40,000 gal tank is returned to the high-level waste storage tanks for volume control. Pumps are provided to allow future recycling of condensate to the Purex Plant, if desired. Much of the aqueous condensate and any organic matter is periodically overflowed through a proportional sampler pit to a crib.

The waste used in the study described in this report is the aqueous phase obtained from the lower part of the retention tank (241-A-417).

Radioisotopes Present in Purex Tank Farm Condensate

The significant radioisotopes found in the Purex tank farm aqueous condensate waste are listed in Table II. Also listed is the maximum permissible concentration of each radioisotope allowable in potable waters (MPC_w) (only one radioisotope present). The last column in Table II lists the decontamination factor required of a treatment process to produce the MPC_w value. Isotopes requiring the highest decontamination factors are Sr^{90} and Cs^{137} .

TABLE II

RADIOISOTOPES PRESENT

IN PUREX TANK FARM AQUEOUS CONDENSATE WASTE

<u>Radioisotope</u>	<u>Half-Life^(a)</u>	<u>Typical Concentration, μcuries/ml</u>	<u>$MPC_w^{(b)}$, μcuries/ml</u>	<u>Decontamination Factor Required</u>
Sr^{90}	28 yr	10^{-4}	1×10^{-6}	>100
Cs^{137}	30 yr	10^{-2}	2×10^{-4}	>50
Zr^{95}	65 d	10^{-2}	6×10^{-4}	>17
Ru^{106}	1 yr	10^{-3}	1×10^{-4}	>10
Ce^{144}	285 d	10^{-3}	1×10^{-4}	>10
Sr^{89}	50 d	10^{-3}	1×10^{-4}	>10
Nb^{95}	35 d	10^{-2}	1×10^{-3}	>10
Y^{91}	59 d	10^{-3}	3×10^{-4}	>3
I^{131}	8 d	10^{-5}	2×10^{-5}	--

(a) Half-lives given are for parent substances only except for Nb^{95} which is listed separately. Nb^{95} was the only daughter radioisotope present in sufficient concentration and with long enough half-life to be considered in this study.

(b) Maximum Permissible Concentration in Water for Occupational Exposure (168 hr wk) from National Bureau of Standards Handbook 69 (June 5, 1959).

Nonradioactive Substances Present in Purex Tank Farm Condensate

The significant nonradioactive substances found in the Purex tank Farm condensate during normal operation are listed in Table III. The organic matter present in solution as a very stable emulsion does not change the decontamination ability of ion exchange materials noticeably; however, it does plug packed ion exchange beds. NH_4^+ because of its similarity to Cs^+ , reduces ion exchange removal of Cs^{137} . NO_3^- and NO_2^- form complexes with ruthenium and inhibit its removal.

TABLE III

NONRADIOACTIVE SUBSTANCES PRESENT PUREX TANK FARM AQUEOUS CONDENSATE WASTE, pH 9-10

	Typical Concentrations, mg/liter
Butyl Phosphates	30 to 200
Hydrocarbons	10 to 70
NH_4^+	35 to 200
Na^+	1 to 2
NO_3^-	1 to 5
NO_2^-	5 to 10

Periodic, but infrequent, addition of unneutralized waste to the high-level waste tanks results in HNO_3 and HNO_2 in the vapors and condensate. Spot samples of condensate taken about a day after unneutralized waste addition had a pH of 2.7, an iron concentration of 3 mg/liter (the collection tank is made of carbon steel), NO_3^- concentration of 260 mg/liter, and NO_2^- concentration of 1100 mg/liter. Normal practice when this occurs is to add an excess of caustic to the storage tank to minimize corrosion.

EXPERIMENTAL PROGRAM

Description of Experimental Equipment

Waste for the experimental program was taken from the Purex tank farm to a waste treatment pilot plant in stainless steel drums or in an 1800 gal tank truck.

Pyrex glass pipe having nominal 1, 2, and 4 in. diameters was used to contain adsorbents for fixed bed adsorption studies. The 4 in. glass pipe was used only for an activated carbon pretreatment. The glass pipe columns were provided with shop fabricated end fittings that held 24 by 110 twilled Dutch weave stainless steel filter cloth in the lower fittings to support the adsorbent beds. Ball valves on both the upper and lower end fittings allowed the columns to be filled with or emptied of adsorbent. The waste was stored in a 75 gal stainless steel feed tank and was pumped by a positive displacement diaphragm pump through columns. The column effluent was discharged out the column bottom to a local crib. The system was provided with instruments to measure temperature, flow, and pressure drop across adsorbent beds.

The removal of NH_4^+ and organic matter from the bulk of the waste was found necessary, so a small steam stripper was designed and fabricated. A stripping column was fabricated from an 8 ft length of 2 in. stainless steel (SS) pipe packed with 1/4 in. SS Raschig rings. A reboiler was fabricated from a 2 ft length of 4 in. SS pipe containing a steam coil made from 10 ft of 3/8 in. SS tubing. A 400 gal SS feed tank was added as was a second diaphragm pump to pump waste to the steam stripper. The stripper was elevated so that both the overhead and bottom streams would drain to receiver tanks below. The bottom stream was routed to the 75 gal feed tank.

The adsorbents used in the waste treatment experiments are listed in Appendix A, and a summary of the 35 experiments is listed in Appendix B.

Waste Pretreatment Experiments

Before the detection of NH_4^+ in the condensate and the use of the steam stripper, activated carbon adsorption was used to reduce the organic content of the waste for long term evaluation of ion exchange materials. The effectiveness of the carbon was studied in various sized columns at waste flow rates of 0.25 to 10 gpm/ft² $\left(0.01 \text{ to } 0.4 \frac{\text{column volume}}{\text{min}}\right)$.

The activated carbon reduced the butyl phosphate concentrations to <0.1 mg/liter. Removal of hydrocarbons was not as efficient; their effluent concentrations varied from 0.5 to 4 mg/liter, with the higher values occurring at higher flow rates. Indications are that activated carbon can remove about 0.2 g of organic matter per gram of activated carbon. With about 100 mg organic per liter in the condensate, about 1000 column volumes could be treated by a bed of activated carbon. Activated carbon removal of organic matter successfully reduced the ion exchange bed hydraulic problems, but there were excessive pressure drops across the carbon beds.

In an attempt to reduce the separation necessary in any pretreatment step, a commercial membrane separator* for removing emulsified organic matter was evaluated. The separator was designed (1) to remove entrained liquid contaminants from liquid systems and (2) to separate immiscible liquids. The separator consisted of porous membranes which coalesce liquids and separate light and heavy phases for discharge. A waste simulating the emulsion characteristics of the Purex tank farm condensate was prepared from tri-n-butyl phosphate (TBP), hydrocarbon diluent, and distilled water. The concentration of the settled but cloudy mixture was about 150 mg of TBP per liter and 20 mg of hydrocarbon per liter. When the emulsion was passed through the membrane system, a very clear effluent which had about 140 ppm TBP and 8 ppm hydrocarbons was produced. Organic matter was not removed from Purex tank farm condensate after passage through a similar membrane system.

* Selas Liquid -- Liquid Separator manufactured by the Selas Corporation of America, Dresher, Pennsylvania.

The steam stripper, which was used to reduce the NH_4^+ and organic matter concentration in the bulk of the Purex tank farm condensate waste, was operated continuously for over 2500 hr without difficulty. At a feed rate of 96 ml/min to the stripper and an overhead distillate rate of 6 ml/min, the NH_4^+ concentration in the bottoms ranged from 1 to 4 mg/liter, the butyl phosphate concentration was less than 1 mg/liter and the hydrocarbon concentration varied from 1 to 4 mg/liter. Thus, the stripper removed >97% of the NH_4^+ and organic impurities. The condensed overhead stream consisted of two phases. The concentrations of radioisotopes in the organic and aqueous phases are shown in Table IV. Since Cs^{137} concentration is very low in the overheads, even though it is present in the feed in concentrations greater than any other isotope, entrainment of the aqueous feed is minor.

TABLE IV

RADIOISOTOPES PRESENT IN STEAM STRIPPER OVERHEADS

<u>Radioisotope</u>	<u>Concentration in Aqueous Phase ($\mu\text{curies/ml}$)</u>	<u>Concentration In Organic Phase ($\mu\text{curies/ml}$)</u>
I^{131}	5.9×10^{-6}	2.7×10^{-3}
Ru^{106}	5.6×10^{-6}	3.4×10^{-4}
Zr-Nb^{95}	9.4×10^{-7}	$<7.8 \times 10^{-6}$
Cs^{137}	$<1.3 \times 10^{-6}$	$<1.3 \times 10^{-5}$

After the steam stripping process to remove NH_4^+ and organic matter from the condensate waste was successfully demonstrated, use of activated carbon was continued for some time to remove residual organic matter in the stripper bottom stream. During several runs when the organic matter in the stripper bottoms averaged about 2 mg/liter, the effluent from the activated carbon bed averaged about 1 mg/liter, indicating rather limited adsorption. The pressure drop across the carbon bed began to increase after about 2000 column volumes. Up-flow of 250 F steam at atmospheric pressure through the activated carbon bed removed about 25% of the hydrocarbons and a negligible amount of butyl phosphates. It is probable that the organic material

removed by the steam treatment was present in the interstices of the bed and had been removed from the condensate by filtration rather than by adsorption. Higher steam temperatures would have to be used to remove the adsorbed organic material effectively. After the steam regeneration step, the bed was effective in removing organic matter, but the pressure drop across the bed increased at a much higher rate than before. The regeneration procedure apparently created enough fines to impair the hydraulic characteristics of the bed.

Granular anthracite coal ("Anthrafilt"-- which is often used in conventional water treatment filters) failed to prefilter the steam stripper bottom stream. The filter bed was able to remove particulate matter present in the waste, but excessive pressure drops were encountered after only a relatively small volume of waste was processed.

More recent experiments using small particle ion exchange adsorbents as a precoat material indicate that filtration and some radioisotope removal can be accomplished without serious hydraulic problems.

Radioisotope Adsorption Without Steam Stripping

The wide interest in the use of minerals for waste treatment and disposal has led to much laboratory work at Hanford on the mineral clinoptilolite. The first six experiments in this study were to determine the ability of clinoptilolite to decontaminate Purex tank farm condensate. Parameters studied include clinoptilolite particle size, feed flow rate, temperature, pretreatment of the waste to remove organic material, and pretreatment of clinoptilolite to remove associated clay.

The results of these experiments showed that clinoptilolite has an excellent ability to remove cesium from Purex tank farm condensate waste. Cesium removal efficiency was enhanced by small mineral particle size. In general, the kinetics were so poor when +20 mesh* material was used that this particle size is probably the upper limit for fixed bed adsorption applications. The lower limit was fixed by the hydraulics of the system.

* Mesh designation in this report refers to standard sieve sizes used in screen analyses as recommended by U.S. National Bureau of Standards. (The results of the work done at the University of California reported in Appendix D includes Tyler Sieve designation.)

Flow rates of less than 2 gpm/ft³ were necessary to obtain the most efficient cesium removal; however, increased temperature permits higher flow rates without impairing cesium removal efficiency.

Clinoptilolite removed some strontium from the condensate, but the decontamination factor was too low for radiostrontium concentrations less than its MPC_w. Variations in flow rate, clinoptilolite particle size, and temperature did not alter the strontium decontamination efficiency significantly. Cerium, zirconium, and ruthenium were not appreciably removed by the mineral.

Strontium, zirconium, and cerium apparently are present as nonionic forms in Purex tank farm condensate, which is normally alkaline, and thus ion exchange is not possible. Some removal of these isotopes under alkaline conditions by activated carbon is probably due to physical adsorption or filtration.

Removal of the organic impurities by activated carbon pretreatment did not improve the decontamination efficiency of clinoptilolite. The use of the activated carbon did improve the overall strontium decontamination because the carbon removed some strontium. Although organic material removed by the activated carbon minimized the hydraulic problems in the ion exchange beds, these problems were merely transferred to the carbon column. Another problem with the activated carbon was the leaching of aluminum ash present in the carbon by the alkaline solution; later, the aluminum precipitated and tended to plug downstream beds.

Hydraulic problems also were met with clinoptilolite because the mineral contained clay particles which tended to break down into fines when contacted with water. The clay had to be removed before column operation. The clinoptilolite beneficiation work is reported in Appendix C, and a study made at the University of California on crushing characteristics of clinoptilolite is included in Appendix D.

Two extended experiments (11 and 12) were performed to measure the capacity of clinoptilolite for removing cesium efficiently. The first experiment using two beds in series was not valid for measuring capacity because one of the beds became plugged about half way through the run. The plugging was caused by precipitate from the leached aluminum ash from the activated carbon pretreatment step. The second run indicated that the clinoptilolite could treat about 5800 column volumes of waste before the Cs^{137} concentration in the effluent would exceed its MPC_w value. The decontamination factor varied from 1000 at the start to 100 at the end of the runs even though about 160 mg of NH_4^+ per liter was present in the waste.

An attempt to use a calcite-phosphate replacement reaction for enhanced strontium removal during Experiment 12 was only slightly successful. Some additional strontium removal was observed, but not enough was removed to obtain an effluent Sr^{90} concentration less than its MPC_w .

Two experiments were performed to study the cesium removal efficiency of two synthetic zeolites.* Decalso removed cesium at a decontamination factor of 850 to 1000 from a waste that contained NH_4^+ . Flow rate had little effect on the cesium decontamination factor. Some strontium was removed; cerium, zirconium, and ruthenium were not appreciably removed.

Molecular Sieve 4A in the form of 1/16 in. pellets was used to treat Purex tank farm condensate. A slow flow rate was required with the relatively large adsorbent particles, but the cesium decontamination factor improved from 350 at the beginning of the run to 2000 when the run was terminated after 500 column volumes of waste were treated. Strontium was only partially removed.

Because strontium was not removed efficiently from the condensate by natural or synthetic zeolites or by the calcite-phosphate replacement reaction, it was decided to evaluate strontium removal by a conventional

* The term zeolite was first applied to a class of naturally occurring crystalline minerals (such as clinoptilolite) having cation exchange properties. Molecular sieves are synthetic counterparts of these minerals. The term was used to describe alumino-silicate materials having cation exchange properties which were used for water softening. Popular usage has extended the term to synthetic organic cation resins. The term as used in this report refers to inorganic alumino-silicate cation exchange materials (gel or crystalline).

strong acid cation resin. Ion exchange studies in the laboratory indicated that a feed pH of 2.5 would be most effective. The first experiment attempted with the acidified waste was terminated after only brief operation. The acidified waste leached iron and aluminum ash impurities from the activated carbon bed. As the waste progressed through the bed, the pH began to rise as the acid was depleted. The higher pH resulted in downstream precipitation of these elements as iron and aluminum hydroxides. The precipitate very quickly plugged the ion exchange resin bed.

A second attempt was made with a new bed of strong acid cation resin after the activated carbon bed was treated with HNO_3 to preleach the ash impurities. The test had to be stopped again because NH_4^+ in the waste was neutralized by HNO_3 when the feed was acidified; at the low pH, ionized ammonium nitrate provided a significant nonradioactive cation load on the resin bed. Breakthrough of cesium occurred before the first sample was taken. Other isotopes were removed only slightly. Until these experiments were performed, the presence of NH_4^+ in the waste was unsuspected. In the concentrations encountered, an ammonia odor should have been a clue to its presence, but the odor given off by the organic material completely masked the ammonia odor. The high pH of the condensate was believed to be due to entrained caustic soda present in the high-level waste.

After significant quantities of NH_4^+ were noted, the steam stripping process previously described was used. Thus, the later adsorption experiments were performed on steam-stripped Purex tank farm condensate. Activated carbon used in these later experiments was commercial acid-leached material.

Radioisotope Adsorption Following Steam Stripping

After the steam stripping unit process was used, further adsorption studies were made with clinoptilolite, strong acid cation resins, strong base anion resins, a weak base anion resin, and an acid-leached activated carbon. Some of the experiments involved combinations of these adsorbents either as separate beds in series or mixed in one bed. The activated carbon acted as

a prefilter-adsorber to remove residual organic matter that remained in the steam stripper bottoms. Although the activated carbon did its task adequately, its use did not appear practical for large scale application. Later experiments omitted the activated carbon prefilter-adsorption step. When used, however, the activated carbon bed often removed significant amounts of strontium, cerium, zirconium, and ruthenium. The results were not always reproducible. Small changes in pH affected adsorption efficiency for these radioisotopes, an indication that partial removal by the carbon might be due to filtration or colloid adsorption.

Strong acid cation resins used to treat steam stripped condensate were quite effective in removing cesium with decontamination factors >1000 . These resins are effective in removing all cations present. The capacity for cesium, therefore, is dependent upon the concentration of nonradioactive cation impurities. For example, when steam-stripped waste with a pH of 6 to 7 and about 0.50 meq cations/liter was passed through a bed of Amberlite IR-120 in the hydrogen form, about 6000 column volumes of waste were treated before the Cs^{137} concentration in the effluent reached its MPC_w value. In a similar test with the resin in the sodium form and the feed with a pH of 4 and about 0.27 meq cations/liter, the resin bed could treat 11,000 column volumes to the same end point. The cesium breakthrough curves for these experiments are shown in Figure 4.

The pH of the feed did not affect the cesium removal efficiency of strong acid cation resins, but the pH was significant for strontium removal. In general, when the pH of the steam stripped waste was lowered from 6 or 7 to 3 or 4, the strontium decontamination factor was increased ten-fold. The pH effect was less obvious when the hydrogen form of the resin was used, because the exchanged hydrogen ions lowered the pH of the waste. The efficiency of strontium removal by a strong acid cation resin in the hydrogen form with acidified feed is shown in Figure 5. When the sodium form of the cation resin was used, the pH effect was quite definite.

FIGURE 4
 Cs^{137} Removal from Steam Stripper Bottoms

FIGURE 5
 Sr^{90} Removal from Acidified Steam Stripper Bottoms

The efficiency of removal for cerium and zirconium was similar to that of strontium. In general, when the pH was reduced to <4 , the effluent concentrations of these radioisotopes was less than their respective MPC_w values. The decontamination factors were not high, however, because of the relatively low concentration of the isotopes in the feed. Most of the feed for these experiments was acidified with HNO_3 . In one experiment, the addition of CO_2 to the feed lowered the pH sufficiently to reduce the effluent strontium concentration. The low pH was difficult to maintain; however, because the CO_2 tended to escape while the feed was stored in the vented feed tank.

Clinoptilolite was also effective in removing cesium from steam stripped condensate. Cesium decontamination factors of >1000 were obtained, and a capacity greater than that attained with strong acid cation resins was measured. When feed containing 0.44 meq cations/liter was passed through a bed of clinoptilolite, about 15,000 column volumes of waste were treated before the Cs^{137} concentration in the effluent reached its MPC value. The breakthrough curve for this experiment is also shown in Figure 4.

With the feed at a pH of about 3, clinoptilolite initially removed strontium with a decontamination factor comparable to that measured with strong acid cation resins. Sr^{90} in the effluent reached its MPC_w value after 6000 column volumes of waste were treated. Some strontium was removed from an additional 10,000 column volumes, however. The breakthrough curve is illustrated in Figure 5.

Both types of cation exchange adsorbents removed only a small amount of ruthenium from the waste. About 10 to 20% removal was observed across a strong acid cation resin. Strong base anion resins removed about 70 to 80% of the ruthenium. With a strong acid cation resin bed in series with a strong base anion bed, over 99% ruthenium removal from about 600 column volumes of waste was accomplished. The capacity of such a system is dependent upon the concentration on anionic impurities in the waste feed. When NO_3^- and NO_2^- appear in the effluent, the efficiency of ruthenium removal decreases (Figure 6). A weak base anion resin bed following a strong acid cation resin bed also removed ruthenium, but not with the same efficiency as the strong base, strong acid series.

FIGURE 6

Ru^{106} Removal from Steam Stripper Bottoms

The steam stripper bottoms are very clear but residual organic matter is present as a filterable particulate. In one run at a flow rate of 1 gpm/ft², the initial pressure drop across an 11 in. deep strong acid cation resin bed was about 0.1 psi. The run was stopped after 6000 column volumes when the pressure drop across the bed had increased to about 4.9 psi. Most of the filterable organic material was removed in a 1 in. layer at the top of the bed (some organic matter was dispersed throughout the bed). Since the organic matter had adhesive properties which caused considerable resin particle agglomeration, mechanical means were used to break up the agglomerate, and solvent treatment was necessary to dissolve the organic matter adhering to the resin beads before regeneration.

Thin Bed Adsorption Studies

The organic material remaining in the steam stripper bottoms is not easily removed by normal backwashing and regeneration techniques; therefore, filtering is necessary to protect ion exchange beds which are to be regenerated.

The use of a precoated filter with a small particle cation exchange filter aid was considered so that both filtering and cation exchange adsorption could be handled by one unit operation. Improved kinetics would be expected with natural zeolites such as clinoptilolite in the smaller particle size, and more of the adsorptive capacity of the filter aid material would probably be used before hydraulic problems would be encountered.

Two experiments were performed with steam stripped condensate to evaluate the decontamination ability of thin precoat beds of cation exchangers. During one experiment the condensate was acidified with HNO_3 to a pH of 3.2 and was passed through 1.7 cm of 200 x 400 mesh strong acid sulfonated polystyrene cation resin at a flow rate of 5 gpm/ft^2 (10 column volumes/min). A strontium decontamination factor >1000 was maintained with >5000 column volumes of waste. The cesium decontamination factor varied from 700 at the beginning of the test to 70 when its MPC_w value was reached. The MPC_w of cesium in the effluent was reached after 2250 column volumes. Pressure drop across the bed increased from 2.1 psi at the start of the test to 3.3 psi at the end.

During the other experiment the feed was acidified to a pH of 4 and passed through 2.0 cm of 100 x 230 mesh acid-washed clinoptilolite in the sodium form at a flow rate of 5 gpm/ft^2 (10 column volumes/min). The decontamination factor for strontium and cesium ranged from 100 to 400 through 4500 column volumes. Concentrations of radiostrontium and radiocesium in the effluent did not exceed their respective MPC_w values. Pressure drop across the bed did not exceed 2 psi.

APPENDIX A

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APPENDIX A

ADSORBENTS USED IN WASTE TREATMENT PILOT PLANT TESTS

Activated Carbon, Type CAL

An activated carbon having a 12 x 40 mesh particle size produced by the high temperature steam activation of bituminous coal. Manufacturer: Pittsburgh Chemical Company, Pittsburgh, Pennsylvania.

Activated Carbon, Type SGL

Same as activated carbon Type CAL except particle size is 8 x 30 mesh.

Amberlite IR-120

A sulfonated polystyrene strong acid cation resin. Manufacturer: Rohm and Haas Company, Philadelphia, Pennsylvania.

Amberlite IR-120L

Same as Amberlite IR-120 except screened to give a slightly larger effective particle size.

Amberlite 200

A sulfonated polystyrene strong acid cation resin having a macro-reticular bead structure. Manufacturer: Rohm and Haas Company, Philadelphia, Pennsylvania.

Amberlite XE-150

A nuclear grade mixture of sulfonated polystyrene strong acid cation resin in the hydrogen form with Type I quaternary ammonium polystyrene strong base anion resin in the hydroxide form uniformly mixed in the proportion of 1 equivalent hydrogen ion to 1 equivalent hydroxide ion. Manufacturer: Rohm and Haas Company, Philadelphia, Pennsylvania.

Apatite, Canadian

A naturally occurring calcium hydroxyphosphate mineral.

Apatite, Synthetic

A synthetic calcium hydroxphosphate made by reacting calcite particles with a hot trisodium phosphate solution.

Calcite

A calcium carbonate mineral.

Clinoptilolite

A naturally occurring alumino-silicate zeolite mineral having a silica to alumina ratio of 8-10:1. The material used in this study was mined by the Baroid Division of National Lead Company, Hector, California.

Decalso

A synthetic alumino-silicate gel having 6:1 silica to alumina ratio. Manufacturer: Permutit Company, New York, New York.

Dowex 50W

A sulfonated polystyrene strong acid cation resin. Manufacturer: Dow Chemical Company, Midland, Michigan.

Illco NR-1

A nuclear grade sulfonated polystyrene strong acid cation resin in the hydrogen form marketed by the Illinois Water Treatment Company, Rockford, Illinois.

Illco NR-2

A nuclear grade Type I quaternary ammonium polystyrene strong base anion resin in the hydroxide form marketed by the Illinois Water Treatment Company, Rockford, Illinois.

Illco TM-1

A commercial grade mixture of sulfonated polystyrene strong acid cation resin in the hydrogen form with Type I quarternary ammonium polystyrene strong base anion resin in the hydroxide form uniformly mixed in the proportion of 1 equivalent hydrogen ion to 1 equivalent hydroxide ion.

It is marketed by the Illinois Water Treatment Company, Rockford, Illinois.

Molecular Sieve 4A

A synthetic alumino-silicate crystalline zeolite having a 2:1 silica to alumina ratio. Manufacturer: Line Company, Tonawanda, New York.

APPENDIX B

APPENDIX B

SUMMARY OF WASTE TREATMENT PILOT PLANT TESTS

EXPERIMENT 1

OBJECTIVES To test Waste Treatment Pilot Plant equipment reliability and develop operating procedures

CONDITIONS

Feed Distilled water at 20 to 25 C

Adsorbents

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, ²gpm/ft</u>
1	Clinoptilolite (untreated)	12 x 30	~250	1	~6

RESULTS AND CONCLUSIONS

Equipment operated continuously for 124 hr. Several equipment deficiencies were observed and corrected. Although distilled water was used as feed, pressure drop across the bed increased 50% during the run. Pressure drop increase was probably caused by degradation of clay particles associated with the clinoptilolite.

EXPERIMENT 2

OBJECTIVES To test Waste Treatment Pilot Plant equipment reliability and develop operating procedures using actual PTFC waste

CONDITIONS

Feed Purex tank farm condensate at 12 to 22 C

Adsorbents

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, ²gpm/ft</u>
1	Clinoptilolite (untreated)	12 x 30	~ 250	1	3 to 6

RESULTS AND CONCLUSIONS

Flow and temperature control was inadequate, system was further modified.

EXPERIMENT 3

OBJECTIVES To measure the decontamination efficiency of clinoptilolite at several flow rates

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Clinoptilolite (untreated)	30 x 40	~180	1	1 to 6

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor >1000 at all flows.
- (2) Strontium decontamination factor decreased from 25 to 11 as flow was increased.
- (3) Cerium, zirconium, and ruthenium decontamination factor <2 at all flows.
- (4) Clay particles associated with clinoptilolite created fines which caused hydraulic problems.
- (5) Emulsified organic in waste was being filtered out causing additional hydraulic problems.

EXPERIMENT 4A

OBJECTIVES To measure the efficiency of activated carbon for decontaminating the waste, and for removing organic impurities in the waste

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow gpm/ft ²
1	Activated Carbon (type SGL)	8 x 30	~500	1	1 to 6

RESULTS AND CONCLUSIONS

- (1) Activated carbon removed only strontium, decontamination factor decreased from 7 to 1.5 as flow rate was increased
- (2) Activated carbon removed over 98% of butyl phosphates and hydrocarbons from the waste

EXPERIMENT 4BOBJECTIVES

To measure the efficiency of a column of clinoptilolite for decontaminating after the waste had been pretreated for organic removal by passage through activated carbon.

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Two beds in series

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, ²gpm/ft</u>
1	Activated carbon (type SGL used during Run 4A)	8 x 30	~500	1	1 to 7
2	Clinoptilolite (untreated)	16 x 20	~450	1	1 to 7

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor increased from 25 to 100 as flow rate was increased.
- (2) Strontium decontamination factor across both beds decreased from 35 to 7 as flow rate was increased.
- (3) Zirconium decontamination factor across both beds decreased from 10 to 7 as flow rate was increased.
- (4) Cerium and ruthenium decontamination factor across both beds was <2 at all flow rates.
- (5) Particle size of clinoptilolite was too large for efficient cesium removal at flows studied.

EXPERIMENT 5A

OBJECTIVES To measure the efficiency of clinoptilolite having a smaller particle size than that used in Run 4B for removing cesium and strontium after the waste had been pretreated for organic removal by passage through activated carbon.

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Three beds in series

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume cm³</u>	<u>Bed Diameter in.</u>	<u>Flow, ²gpm/ft</u>
1	Activated carbon (type SGL)	8 x 30	~ 460	1	0.25 to 6
2	Activated carbon (type SGL)	8 x 30	~ 460	1	0.25 to 6
3	Clinoptilolite (untreated)	20 x 40	~ 410	1	0.25 to 6

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor across all three beds decreased from 3000 to 720 as flow rate was increased.
- (2) Strontium decontamination factor across all three beds decreased from 30 to 11 as flow rate was increased.
- (3) Cerium, zirconium and ruthenium decontamination factors not measured because of poor results in earlier runs.
- (4) Fines resulting from clay particles in clinoptilolite caused increased pressure drop across the mineral bed.

EXPERIMENT 5B

OBJECTIVES Same as Run 5A but at a higher feed temperature

CONDITIONS

Feed Purex tank farm condensate at 45 C

Adsorbents Same beds used in Run 5A.

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor across all three beds decreased 2900 to 1300 as flow rate was increased.
- (2) Strontium decontamination factor across all three beds decreased from 28 to 15 as flow rate was increased.

EXPERIMENT 5B (Contd.)

- (3) Higher feed temperature improved decontamination efficiency for cesium and strontium at higher flow rates.
- (4) Butyl phosphate appeared in the effluent from the first activated carbon column after about 1000 liters of waste were treated.

EXPERIMENT 6

OBJECTIVES

To measure the decontamination efficiency of clinoptilolite which was treated for clay removal at various flow rates after the waste had been pretreated for organic removal by passage through activated carbon.

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Activated carbon (type SGL)	8 x 30	~460	1	0.25 to 10
2	Clinoptilolite (caustic soda treated and water washed)	20 x 25	~460	1	0.25 to 10

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor decreased from 1000 to 200 as flow rate was increased.
- (2) Strontium decontamination factor across both beds decreased from 20 to 10 as flow rate was increased.
- (3) Cerium, zirconium and ruthenium decontamination factors varied from 2 to 4 across both beds at all flow rates.
- (4) Pretreatment of the clinoptilolite by immersion in hot 5% caustic soda for about 1 hr followed by a distilled water wash improved the hydraulics of the mineral column.

EXPERIMENT 7

OBJECTIVES To measure the decontamination efficiency of a synthetic apatite

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, ² gpm/ft
1	Apatite (made by reacting calcite with a hot sodium phosphate solution)	20 x 30	~440	1	1

RESULTS AND CONCLUSIONS

- (1) No radioisotope removal was observed.

EXPERIMENT 8

OBJECTIVES To measure the decontamination efficiency of a synthetic alumino-silicate gel

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, ² gpm/ft
1	Activated carbon, (type SGL)	8 x 30	500	1	1 to 10
2	Decalso	16 x 50	400	1	1 to 10

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor varied from about 850 to 1000 at the beginning of the run to about 300 after about 150 liters of waste were treated. Changes in flow rate did not appreciably effect the cesium decontamination factor.
- (2) Strontium decontamination factor across both beds varied from 15 to 30 during the run. Changes in flow rate did not appreciably effect the strontium decontamination factor.
- (3) Cerium, zirconium and ruthenium were not removed significantly.

EXPERIMENT 9

OBJECTIVES To measure the decontamination efficiency of a naturally occurring apatite mineral after passage of the waste through activated carbon for organic removal

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Activated carbon, (type SGL)	8 x 30	500	1	1 to 10
1	Canadian Apatite	10 x 40	400	1	1 to 10

RESULTS AND CONCLUSIONS

- (1) Strontium was removed with a decontamination factor of about 6 across both beds.
- (2) No other isotope removal was observed.

EXPERIMENT 10

OBJECTIVES To measure the decontamination efficiency of a synthetic crystalline zeolite after passage of the waste through activated carbon for organic removal.

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume cm ³	Bed Diameter in.	Flow, gpm/ft ²
1	Activated carbon, (type SGL)	8 x 30	500	1	2
2	Molecular sieve 4A, pellets	1/16 in.	400	1	2

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor increased from about 350 at the start of the run to about 2000 after about 200 liters of waste were treated.
- (2) Strontium decontamination factor across both beds varied from 15 to 20 during the entire run.

EXPERIMENT 11

OBJECTIVES To measure the decontamination efficiency of clinoptilolite after the waste was pretreated for removal of organic matter.

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents Three beds in series

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, ²gpm/ft²</u>
1	Activated carbon, (type SGL)	8 x 30	10,000	4	1 to 2
2	Clinoptilolite (caustic soda treated and water washed)	18 x 20	250	1	4
3	Clinoptilolite (caustic soda treated and water washed)	20 x 40	250	1	4

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor varied from 1000 at start of run to 100 when the concentration of Cs¹³⁷ in the effluent reached its MPC_w value after about 1800 liters of waste were treated.
- (2) Strontium decontamination factor across the activated carbon bed varied from 5 to 10 and across the clinoptilolite beds varied from 4 to 8 for an overall decontamination factor of 20 to 80. Sr⁹⁰ concentration in the effluent exceeded its MPC_w during most of the run.
- (3) Cerium, zirconium and ruthenium radioisotopes were only slightly removed.
- (4) The high ammonia concentration in the feed leached aluminum ash impurities from the activated carbon. Precipitation of aluminum hydroxide in the system downstream from the activated carbon bed caused plugging of the first mineral bed.

EXPERIMENT 12

OBJECTIVES To measure the decontamination efficiency and capacity of clinoptilolite for removing cesium and strontium and for measuring the efficiency with which strontium is removed using a calcite-phosphate replacement reaction

CONDITIONS

Feed Purex tank farm condensate containing about 160 mg/liter ammonia at 25 C; 200 mg/liter NaOH and 173 mg/liter Na_3PO_4 were added towards latter part of run after Cs^{137} breakthrough occurred.

Adsorbents Three beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume cm^3	Bed Diameter in.	Flow, gpm/ft^2
1	Activated carbon (type SGL)	8 x 30	10,000	4	0.5 to 1.3
2	Clinoptilolite (caustic soda heated and water washed)	20 x 40	250	1	4
3	Calcite	20 x 40	250	1	4

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor varied from 1000 at start of run to 100 when the concentration of Cs^{137} in the effluent reached its MPC_w value after about 1450 liters of waste were treated.
- (2) Strontium decontamination factor across the activated carbon bed varied from 4 to 10 and across the mineral beds varied from 5 to 8 for an overall decontamination factor of 20 to 80. When phosphate was added to the feed the overall decontamination factor increased to 100. Sr^{90} concentration in the effluent exceeded its MPC_w during the entire run.

EXPERIMENT 13

OBJECTIVES To measure the efficiency of cesium and strontium decontamination by clinoptilolite with and without activated carbon pretreatment of the waste for organic removal

CONDITIONS

Feed Purex tank farm condensate at 25 C.
Adsorbents During part A of run two beds in series. During part B one new clinoptilolite bed used.

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
<u>Part A</u>					
1	Activated carbon (type SGL)	8 x 30	10,000	4	~0.5
2	Clinoptilolite (caustic soda treated water washed)	20 x 40	250	1	1 to 6
<u>Part B</u>					
1	Clinoptilolite (caustic soda treated water washed)	20 x 40	250	1	1 to 6

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor varied from 200 to 300 in both cases.
- (2) Strontium decontamination factor across activated carbon was 15 and across clinoptilolite was 6 for an overall decontamination factor of 90. When clinoptilolite was used alone, the decontamination factor was 45.
- (3) Activated carbon pretreatment of purex tank farm condensate did not affect cesium decontamination but improved the overall strontium removal by a factor of 2.

EXPERIMENT 14OBJECTIVES

To measure the efficiency of a strong acid cation resin for decontaminating acidified waste

CONDITIONSFeed

Purex tank farm condensate at 25 C acidified to pH 3 with nitric acid

Adsorbents

Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, ² gpm/ft ²
1	Activated carbon (type SGL)	8 x 30	10,000	4	0.3
2	Amberlite IR-120 (in hydrogen form)	16 x 50	250	1	4

RESULTS AND CONCLUSIONS

Test was prematurely terminated. Acid in the feed leached iron and aluminum ash impurities from the activated carbon bed, these elements then precipitated in the effluent stream resulting in plugging of the downstream resin bed.

EXPERIMENT 15OBJECTIVES

To measure the efficiency of a strong acid cation resin for decontaminating acidified waste using an acid leached activated carbon bed for organic removal.

CONDITIONSFeed

Purex tank farm condensate at 25 C acidified to pH 2.3 with nitric acid

Adsorbents

Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, ² gpm/ft ²
1	Activated carbon (acid leached)	8 x 30	10,000	4	0.3
2	Amberlite IR-120 (in hydrogen form)	16 x 50	250	1	4

EXPERIMENT 15 (Contd.)RESULTS AND CONCLUSIONS

Radioisotope breakthrough occurred after only a few liters of waste were treated. Excessive ammonium ions and hydrogen ions in the feed were the cause of the early run termination. Prior to this run, the presence of ammonia in the waste was not suspected.

EXPERIMENT 16

OBJECTIVES To measure the efficiency and capacity of a sulfonated polystyrene cation resin in the hydrogen form for decontaminating steam stripped waste after passage through acid leached activated carbon.

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C

Adsorbents Two beds in series

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
1	Activated carbon (type CAL, acid leached)	12 x 40	10,000	4	0.3
2	Amberlite IR-120 (in hydrogen form)	16 x 50	250	1	4

RESULTS AND CONCLUSIONS

- (1) Cesium was removed by the resin bed only. Cesium decontamination factor was greater than 1000 until breakthrough. Cs^{137} in the effluent reached its MPC_w after 1500 liters of waste were treated.
- (2) Strontium decontamination factor across the activated carbon bed varied from 100 to 10 and across the resin bed remained at about 100 for an overall decontamination factor varying from 10,000 to 1000. Sr^{90} in the effluent did not exceed its MPC_w during the run treating 3000 liters of waste.
- (3) The activated carbon bed removed cerium with a decontamination factor of 50 to 1000, zirconium with a decontamination factor of 40 and ruthenium with a decontamination factor of about 20. Very little additional removal of these radioisotopes was observed across the resin bed.

EXPERIMENT 17OBJECTIVES

To measure the efficiency and capacity of a sulfonated polystyrene cation resin previously used in Run 16 and regenerated with 1500 ml of 4 N HNO₃ for decontaminating steam stripped waste after passage through acid leached activated carbon.

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C

Adsorbents Two beds in series

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
1	Activated carbon type CAL (acid leached and previously used for Run 16)	12 x 40	10,000	4	0.3
2	Amberlite IR-120 (acid regenerated bed from Run 16)	16 x 50	250	1	4

RESULTS AND CONCLUSIONS

- (1) Cesium was removed by the resin bed only. Cesium decontamination factor was 300 until breakthrough when Cs¹³⁷ in the effluent reached its MPC_w after 2200 liters of waste were treated.
- (2) Strontium decontamination factor across the activated carbon bed was about 4, and across the resin bed was about 100 for an overall decontamination factor of about 400. Sr⁹⁰ in the effluent did not exceed its MPC_w during the run treating 3700 liters of waste.
- (3) The activated carbon bed removed cerium with a decontamination factor of 10, zirconium with a decontamination factor of 10 and ruthenium with a decontamination factor of 25. Very little additional removal of these radioisotopes was observed across the resin bed.

EXPERIMENT 18

OBJECTIVES

To measure the efficiency and capacity of clinoptilolite in the hydrogen form for decontaminating steam stripped waste acidified to pH 3 and passed through an activated carbon bed.

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C acidified with nitric acid to pH 3.

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Activated carbon, type CAL (acid leached previously used for Runs 16 and 17)	12 x 40	10,000	4	0.3
2	Clinoptilolite (caustic soda treated, water rinsed, nitric acid treated, water rinsed)	16 x 30	250	1	4

RESULTS AND CONCLUSIONS

- (1) Cesium was removed by the clinoptilolite with a decontamination factor of 1000 to 10,000 during the early part of the run. Cs¹³⁷ in the effluent reached its MPC_w after 3700 liters of waste were treated. Nonradioactive ionic impurities in the feed averaged about 0.44 meq/liter during the run.
- (2) Strontium was removed by the clinoptilolite with a decontamination factor of 10,000 during the early part of the run. Sr⁹⁰ in the effluent reached its MPC_w after 1300 liters of waste were treated. Strontium removal continued for an additional 2500 liters with a decontamination factor greater than 50.
- (3) Cerium, zirconium and ruthenium decontamination factors:

	Cerium DF	Zirconium DF	Ruthenium DF
a. Across activated carbon bed	50	40	10
b. Across Clinoptilolite bed	2	10	5
c. Overall	250	400	50

EXPERIMENT 19

OBJECTIVES

To measure the efficiency and capacity of a bed of strong acid cation resin in series with a bed of clinoptilolite for decontaminating steam stripped waste at a low pH and at near neutral pH.

CONDITIONS

Feed

Steam stripped Purex tank farm condensate at 25 C acidified with nitric acid to pH 2.4 at the start of the run and nonacidified (pH 6.5) near the end of the run.

Adsorbents Three beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Activated carbon, type CAL (acid leached)	12 x 40	1000	4	0.3
2	Amberlite IR-120 (in hydrogen form)	16 x 50	250	1	4
3	Clinoptilolite (caustic soda treated, water rinsed, nitric acid treated, water rinsed)	16 x 30	250	1	4

RESULTS AND CONCLUSIONS

		Decontamination Factor				
		Cesium	Strontium	Cerium	Zirconium	Ruthenium
a.	Across activated carbon bed	1	6	3	1	3
b.	Across resin bed	1-100	100-2000	10	10	1
c.	Across Clinoptilolite bed	40-100	nil	10	2	1
d.	Overall	200-2000	600-12000	300	20	3

EXPERIMENT 19 (Contd.)

Variations in cesium and strontium decontamination factors are a result of varying the pH of the feed. Greater cesium removal was observed when the pH was near neutral, but the greatest strontium removal was observed when the pH was low (2.5). The optimum pH for removing these two isotopes is between 3.5 and 4.5.

EXPERIMENT 20

OBJECTIVES To measure the efficiency and capacity of an aluminosilicate gel cation exchange adsorbent for decontaminating steam stripped waste.

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C.

Adsorbents Two beds in series

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
1	Activated carbon, type CAL (acid leached)	12 x 40	1000	2	1
2	Decalso	16 x 30	250	1	4

RESULTS AND CONCLUSIONS

Malfunction of steam stripper resulted in termination and cancellation of run.

EXPERIMENT 21

OBJECTIVES To study the operation of the steam stripper and measure the efficiency with which ammonia is steam stripped at varying feed flow rates.

CONDITIONS

Feed Purex tank farm condensate

Adsorbents None

RESULTS AND CONCLUSIONS

Holding all other system parameters constant, the feed rate to the steam stripper was varied from 60 ml/min to 90 ml/min resulting in a change of overhead flow rate from 12 ml/min to 6 ml/min. The ammonia in the steam stripper bottom stream remained fairly constant at 2.3 mg/liter.

EXPERIMENT 22

OBJECTIVES To measure the efficiency and capacity of a strong acid cation resin in the sodium form for decontaminating steam stripped waste acidified to a pH of about 4 and passed through an activated carbon bed.

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C acidified with nitric acid to a pH of 4.3.

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Activated carbon, type CAL (acid leached)	12 x 40	1000	2	1
2	Amberlite IR-120 (in sodium form)	16 x 50	250	1	4

RESULTS AND CONCLUSIONS

		Decontamination Factor				
		Cesium	Strontium	Cerium	Zirconium	Ruthenium
(1)						
a.	Across activated carbon bed	1	1	20	10	4-10
b.	Across resin bed	1000	2000	5-10	2	10
c.	Overall	1000	2000	100-200	20	40-100
(2)	Cs ¹³⁷ in the effluent exceeded its MPC _w after 2800 liters were treated. Concentrations of all other isotopes in the effluent did not exceed their respective MPC _w values.					

EXPERIMENT 23

OBJECTIVES To measure the efficiency of a steam regenerated activated carbon bed for removing organic matter from waste.

CONDITIONS

Feed Purex tank farm condensate at 25 C

Adsorbents

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
1	Activated carbon, type CAL (acid leached previously used in Run 22)	12 x 40	1000	2	1

RESULTS AND CONCLUSIONS

The activated carbon bed used in Run 22 was regenerated by passing 250 F steam at atmospheric pressure upflow through the bed. About 25% of the hydrocarbons and a negligible amount of the butyl phosphates were removed by this technique. Probably the organic material removed by steam regeneration was primarily present in the interstices of the bed and had been removed from the condensate by filtration rather than by adsorption. Higher steam temperatures are necessary to remove adsorbed organic.

After the regeneration step the bed was effective in removing organic, but the pressure drop across the bed increased during the run at a higher rate than was previously observed. The regeneration procedure apparently created enough fines to impair the hydraulic characteristics of the bed when reused.

EXPERIMENT 24

OBJECTIVES To evaluate the use of a bed of granular anthracite coal for filtering steam stripped waste before passage through ion exchange beds

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Anthrafilt	~ 16 x 50	1500	2	1
2	Amberlite IR-120 (resin from Run 22 regenerated with caustic soda)	16 x 50	250	1	4

RESULTS AND CONCLUSIONS

The bed of granular anthracite coal was effective in filtering particulate matter from the waste, but pressure drop across the bed became excessive after only a few hundred liters of waste were treated. The run was terminated when the column pressure relief valve opened.

EXPERIMENT 25

OBJECTIVES 1. To measure the efficiency of decontamination and hydraulic characteristics of a strong acid cation resin for treating steam stripped waste without an activated carbon pretreatment step. 2. To study the acid regeneration of this cation resin

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 24 C

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Amberlite 200 (in hydrogen form)	16 x 50	500	2	1

EXPERIMENT 25 (Contd.)

RESULTS AND CONCLUSIONS

- (1) Decontamination efficiency: Cesium - 1000; strontium - 1000; cerium - 5; zirconium - 5-10; ruthenium - 1. Over 3000 liters of waste were treated with only Ru^{106} exceeding its MPC_w in the effluent.
- (2) Pressure drop across the bed at the end of the run was 20 times higher than it was at the beginning of the run. Organic matter in the waste caused the agglomeration of a 1 in. layer of resin at the top of the bed. Some organic matter was dispensed through the entire bed. Backwashing the bed with water was only partially effective in removing the organic. To obtain maximum removal of the organic, it was necessary to mechanically break up the agglomerated mat which had settled to the bottom of the column during the backwash operation; in addition a small amount of acetone was used to remove residual organic adhering to the individual particles of resin.

EXPERIMENT 26

OBJECTIVES

To measure the efficiency of decontamination and hydraulic characteristics of a regenerated strong acid cation resin for treating acidified steam stripped waste without an activated carbon pretreatment step.

CONDITIONS

Feed

Steam stripped Purex tank farm condensate waste at 24 C acidified with nitric acid to a pH that varied from 3.4 to 4.1.

Adsorbents

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm^3</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft^2</u>
1	Amberlite 200 (in hydrogen form, regenerated resin used in Run 25)	16 x 50	500	2	1

RESULTS AND CONCLUSIONS

- (1) Decontamination Efficiency:

Cesium	2000
Strontium	1000
Cerium	40
Zirconium	10
Ruthenium	1.1 (~10% removal)

EXPERIMENT 26 (Contd.)

Over 3000 liters of waste were treated with only Ru^{106} exceeding its MPC_w in the effluent.

- (2) With the feed acidified pressure drop across the bed did not appreciably increase during the run.

EXPERIMENT 27

OBJECTIVES To measure the efficiency of a two bed demineralizer followed by a mixed bed demineralizer for decontaminating acidified steam stripped waste

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 24 C acidified with nitric acid to pH 3.7.

Adsorbents Three beds in series

Bed No.	Type	Mesh Size USBS	Volume, cm^3	Diameter, in.	Flow, gpm/ft^2
1	Amberlite IR-120 (in hydrogen form)	16 x 50	500	2	1
2	Ilco NR-2 (in hydrogen form)	16 x 50	500	2	1
3	Amberlite XE-150	16 x 50	500	2	1

RESULTS AND CONCLUSIONS

Decontamination Efficiency

		Decontamination Factor				
		Cesium	Strontium	Cerium	Zirconium	Ruthenium
a.	<u>First part of run</u>					
	Across Beds 1 and 2	2500	2500	200	18	200
	Across Bed 3	2	1	3	2	1
	Overall	5000	2500	600	36	200
b.	<u>Last part of run</u>					
	Across Beds 1 and 2	2500	2500	200	30	10
	Across Bed 3	1.2	1	3	1	2
	Overall	3000	2500	600	30	20

EXPERIMENT 27 (Contd.)

Over 1500 liters of waste were treated during the run. Except for ruthenium the concentration of all radioisotopes in the effluent was less than their respective MPC_w values. Ruthenium was removed effectively by both the two beds and mixed bed systems during the early part of the run. The increase in effluent concentration in both cases coincided with the breakthrough of nitric acid. Even after the nitric acid breakthrough, however, removal of over 50% of the ruthenium was still achieved.

EXPERIMENT 28

OBJECTIVES

To measure the efficiency of a strong acid cation resin having a larger particle size for decontaminating steam stripped waste when used in the as-received sodium form.

CONDITIONS

Feed

Steam stripped Purex tank farm condensate at 25 C acidified during latter part of the run with nitric acid to pH 3.8.

Adsorbents

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
1	Amberlite IR-120L (in sodium form as-received)	16 x 40	1000	2	1

RESULTS AND CONCLUSIONS

Decontamination Efficiency:

Cesium	5500
Strontium	3000
Cerium	10
Zirconium	2
Ruthenium	1.1 (~ 10% removal)

EXPERIMENT 29

OBJECTIVES To measure the efficiency of a strong acid cation resin in the sodium form for decontaminating acidified steam stripped waste

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 24 C acidified to pH 4 with nitric acid addition

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Amberlite 120 (in sodium form)	16 x 50	1000	2	1

RESULTS AND CONCLUSIONS

Decontamination Efficiency:

Cesium	4000
Strontium	10
Cerium	3
Zirconium	2
Ruthenium	1.2 (~20% removal)

EXPERIMENT 30

OBJECTIVES To measure the efficiency of a strong base anion resin for removing ruthenium

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Illco NR-2	16 x 50	1000	2	1

RESULTS AND CONCLUSIONS

About 2000 liters of waste were treated. During the first half of the run ruthenium was removed with a decontamination factor of 7 (~85% removal) and during the last half of the run the radioisotope was removed with a decontamination factor of about 3.3 (~70% removal). The decrease in removal efficiency coincided with nitrate and nitrite saturation of the bed.

EXPERIMENT 31

OBJECTIVES To measure the decontamination efficiency and capacity of a commercial grade mixed bed ion exchange resin

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Illco TM-1	16 x 50	500	2	1

RESULTS AND CONCLUSIONS

Unexpected high concentrations of nonradioactive impurities in waste resulted in premature appearance of radioactivity in the treated effluent. The run was terminated.

EXPERIMENT 32

OBJECTIVES To measure the decontamination efficiency and capacity of a commercial grade mixed bed resin.

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C and pH 7.3. Feed contained 20 mg/liter NH₄⁺, 5 mg/liter Na, 25 mg/liter NO₃⁻ and 32 mg/liter NO₂⁻.

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Illco TM-1	16 x 50	1500	2	1

RESULTS AND CONCLUSIONS

- (1) Cesium decontamination factor was > 1000 for the first 800 liters of waste treated. Cs¹³⁷ breakthrough coincided with ammonium ion and sodium ion saturation of the bed.
- (2) Strontium decontamination factor was 35 during most of the run. Reduction of the feed pH towards the end of the run to about 4.5 increased the strontium decontamination factor to 350.
- (3) About 99% (decontamination factor of 100) of the ruthenium was removed by the bed during the early part of the run. When nitrate and nitrite ions saturated the bed, ruthenium removal efficiency decreased, but 90% (decontamination factor of 10) continued to be removed for the remainder of the run (2400 liters).

EXPERIMENT 33

OBJECTIVES To measure the ruthenium decontamination efficiency of a strong acid cation resin followed by a weak base anion resin

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C with carbon dioxide gas added to reduce the pH which varied from 4.3 to 5.8

Adsorbents Two beds in series

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Illco NR-1 (in hydrogen form)	16 x 50	250	2	1
2	Nalco WBR (in hydrogen form)	16 x 50	500	2	1

RESULTS AND CONCLUSIONS

Ruthenium decontamination factor across the cation resin bed averaged over 1.1 (~12% removal) and across the weak base anion bed varied from 3 to 15 (67 to 93% removal) with the average overall decontamination factor of 6.8 (85% removal).

EXPERIMENT 34

OBJECTIVES To measure the cesium and strontium decontamination efficiency of a thin precoat bed of small particle size strong acid cation resin

CONDITIONS

Feed Steam stripped Purex tank farm condensate at 25 C and acidified with nitric acid to pH 3.2

Adsorbents

Bed No.	Type	Mesh Size USBS	Bed Volume, cm ³	Bed Diameter, in.	Flow, gpm/ft ²
1	Dowex 50W (in hydrogen form)	200 x 400	40	2	5

EXPERIMENT 34 (Contd.)

RESULTS AND CONCLUSIONS

- (1) Cesium was removed with a decontamination factor of 700 during the early part of the run. Cs^{137} in the effluent reached its MPC_w after 2250 column volumes of waste were treated.
- (2) Strontium was removed with a decontamination factor >1000 while treating over 5000 column volumes of waste.
- (3) Pressure drop across the bed increased from 2.1 psi to 3.3 psi when the run was completed.

EXPERIMENT 35

OBJECTIVES

To measure the cesium and strontium decontamination efficiency of a thin precoat bed of small particle size clinoptilolite

CONDITIONS

Feed

Steam stripped Purex tank farm condensate at 25 C and acidified with nitric acid to pH 4

Adsorbents

<u>Bed No.</u>	<u>Type</u>	<u>Mesh Size USBS</u>	<u>Bed Volume, cm³</u>	<u>Bed Diameter, in.</u>	<u>Flow, gpm/ft²</u>
1	Clinoptilolite (acid washed, sodium regenerated and rinsed)	100 x 230	40	2	5

RESULTS AND CONCLUSIONS

- (1) Cesium and strontium decontamination factors varied from 100 to 400 while treating over 4600 column volumes of waste. Cs^{137} and Sr^{90} concentrations in the effluent did not exceed their respective MPC_w values during the entire run.
- (2) Pressure drop across the bed did not exceed 2 psi during the run.

APPENDIX C

APPENDIX C

CLINOPTILOLITE BENEFICIATION

Because the zeolite mineral clinoptilolite has properties which allow it to adsorb cesium from solutions containing much higher concentrations of other cations, research was directed at using this material for the removal of radiocesium from waste streams. The laboratory work exploring the ion-exchange properties of this mineral was done on selected samples of unweathered clinoptilolite which was crushed and screened in laboratory equipment.

All of the material used in the Waste Treatment Pilot Plant studies was obtained from deposits owned by the Baroid Division of the National Lead Company in Hector, California. Two tons of crude mine run clinoptilolite were obtained for the initial experiments. This material was crushed and screened by a Seattle, Washington, commercial firm which rented small crushing and screening equipment at a local university. The screening operation produced two fractions: one was 12 x 40 mesh (USBS), and the other was -40 mesh (USBS). This material was further screened in small batches at Hanford. Particle degradation in the wet material caused considerable trouble. A large amount of the granular mineral consisted of clay particles. When wet, the clay particles disintegrated to produce fines. These fines caused hydraulic problems when the mineral was used in fixed bed adsorption experiments.

To permit testing, further treatment of the material was necessary. The mineral was dry screened to the desired particle size range and immersed in hot caustic soda solution to peptize the clay. It was then placed in a 2 in. glass pipe column and washed (upflow) with hot distilled water. A flow rate of about 50 gpm/ft², fluidizing the bed for 8 hr was used. When allowed to settle, most of the material in the bed had the characteristic green color of Hector clinoptilolite. The lower 10 to 20% of the bed still contained brown clay particles. The clay material was discarded.

Discussions were held with personnel of the National Lead Company about the impurities present. The 2 ton batch of the mineral was mined at the exposed face of the deposit and did contain high clay and calcium carbonate impurities. An order was placed for an additional 2 tons of clinoptilolite to have a low clay content and less than 1% calcium carbonate. Except for 400 lb of crude which was sent directly to Hanford, this material was sent to the University of California for crushing and screening experiments. The results of these experiments are reported in Appendix D. The crushed and screened material was shipped to Hanford for further testing. Various samples were analyzed for carbonates and it was determined that the crushed and screened material contained an average of 3.7% calcium carbonate (determined by the acid release and measurement of CO_2) or almost four times that expected by the National Lead Company. Although fewer impurities were present in the second batch, further treatment was necessary before the material could be used in fixed bed column experiments.

Experimental work showed that a simple crushing and screening process is not adequate for producing a granular clinoptilolite material suitable for fixed bed application. A conceptual beneficiation process for preparing clinoptilolite for fixed bed use is shown in Figure C-1. The mine run clinoptilolite is fed to a jaw crusher to produce an appropriate size feed for a roll mill. The milled clinoptilolite is fed to vibrating screen separators where an appropriate particle size separation is made. The fines are discarded, the oversize material is returned to the roll mill and the intermediate product enters a Dorr classifier where the classifier rakes aid in breaking up clay particles and water removes the resultant clay and clinoptilolite fines. The clinoptilolite is then contacted with hot 5 to 10% HNO_3 solution which is followed by a 5 to 10% caustic soda wash. The acid dissolves calcium carbonate impurities, and the caustic soda peptizes and removes the last traces of clay and neutralizes any residual acid in the material. If the clinoptilolite is not going to be used with acidic solutions, the acid treatment can be omitted. The mineral leaving the hot caustic soda treatment is washed with demineralized water in a Dorr classifier to remove the fines created in the chemical treatment steps, dewatered with a vibrating screen separator, and

FIGURE C-1

Conceptual Flowsheet to Prepare Clinoptilolite
for Fixed Bed Application

packaged for storage. The best yield that can be expected for this process is 40% for a 20 x 50 mesh product.

A conceptual flowsheet for preparing clinoptilolite to be used in a precoated zeolite filter is similar to Figure C-1 with the exception that the chemical treatment to remove the fines will be less demanding. Since the product required is about 100 x 400 mesh, the yield may be > 60%.

The approximate cost of crude clinoptilolite is \$125/ton in less than carload quantities and \$80/ton in carload amounts. The price of a 2 ton batch of hand picked material was \$140/ton. If the lowest price is considered, the raw clinoptilolite cost would be \$2/ft³ (\$80/ton ÷ 40 ft³/ton). An estimated cost for crushing and screening the raw material in carload quantities is \$100/ton or \$2.50/ft³.

Table C-I compares costs of inorganic ion-exchangers. The costs for clinoptilolite were corrected to a 40% yield on the 20 x 50 mesh and a 60% yield on the 100 x 400 mesh.

TABLE C-I
APPROXIMATE COST OF INORGANIC ION-EXCHANGERS
(Excluding Shipping Costs)

	<u>\$/ft³ of Product</u>	
<u>Clinoptilolite</u>	<u>20 x 50 Mesh</u>	<u>100 x 400 Mesh</u>
Ore (Crude at \$2/ft ³)	5	3
Crush and Screen	6	4
Chemical Treatment	<u>3</u>	<u>2</u>
Total	14	10
<u>Decalso</u>	25	
<u>Synthetic Zeolites</u>	80	

For cost estimates of the ion-exchange process, \$25/ft³ of exchanger was used despite the apparent cost advantage of clinoptilolite. The higher cost was chosen because of the uncertainties in the cost estimate for clinoptilolite, since prepared clinoptilolite is not currently a commercial product.

APPENDIX D

APPENDIX DCRUSHING CHARACTERISTICS OF CLINOPTILOLITE*

J. A. Pask and D. W. Fuerstenau

The objective of this brief report is to present the results of work done in preparing 10 x 50 mesh clinoptilolite particles and also to discuss the results of a brief investigation of the crushing characteristics of clinoptilolite.

The major work concerned preparation of 10 x 20, 20 x 50, and -50 mesh fractions from lump clinoptilolite (about 3 to 6 in. pieces). Crushing and screening were carried out so as to obtain the maximum recovery of 10 x 50 mesh material.

Clinoptilolite, which was obtained from the Baroid Division of National Lead Company, was crushed in a jaw crusher to prepare the material as feed for the roll mill. After the clinoptilolite was passed through the roll mill, it was screened dry on a triple deck screen containing 10, 20, and 50 mesh screens. Three products were collected and the oversize material was recycled to the roll mill. The procedure produced the following products:

<u>Size Fraction</u>	<u>Weight, lb</u>	<u>Weight Distribution, %</u>
10 x 20 mesh	1677	47.1
20 x 50 mesh	823	23.1
Minus 50 mesh	<u>1061</u>	<u>29.8</u>
	3561	100.0

Size distribution data of each of these three products are presented in Table D-I. These size distribution data were obtained by dry sieving each of the products.

* The work reported in Appendix D was performed at the University of California under Consultant Agreement CA-330 with the General Electric Company.

TABLE D-I

SIZE DISTRIBUTION OF CRUSHED AND SCREENED CLINOPTILOLITE

Size Fraction, Tyler Mesh	Weight, %		
	10 x 20 mesh fraction	20 x 50 mesh fraction	Minus 50 mesh fraction
10 x 14	60.1	0.0	
14 x 20	35.7	0.4	
20 x 28	3.4	42.1	
28 x 35	0.2	38.4	0.0
35 x 48	0.1	18.0	8.4
48 x 65	0.1	0.6	21.5
65 x 100	0.1	0.2	21.2
100 x 150	0.1	0.1	13.5
150 x 200	0.0	0.1	15.6
200 x D	<u>0.2</u>	<u>0.1</u>	<u>19.8</u>
	100.0	100.0	100.0

To ascertain the optimum size distribution that can be obtained in crushing clinoptilolite, a series of tests was made grinding the material in a laboratory rod mill in a simulated closed-circuit with a 10 mesh sieve. Because coarse particles protect finer particles by wedging the rods apart, rod milling produces a product that possesses fine particles characteristic of the material. This wedge-action in a rod mill produces particles with a minimum amount of fines. In the rod mill experiments, 1 kg of 4 x 8 mesh clinoptilolite was ground for a 2 min period, screened on a 10 mesh sieve, and the oversize returned to the mill with enough new feed to make a 1 kg charge in the mill. This was repeated until steady state was reached. Under these conditions the circulating load was 110%. A similar series of tests was run in 4 min cycles, giving a circulating load of 23%. Since the products from the two different rod milling experiments were identical, the results of only the 4 min test will be given. The product from this type of test should yield a size distribution that is a straight line when the cumulative percent passing a certain size is plotted on log-log paper versus the

size. Since the sieve series is logarithmic (to the base $\sqrt{2}$), a semilog plot of cumulative percent finer versus size in mesh should yield the straight line (see Figure D-1).

FIGURE D-1

Cumulative Size Distributions of Clinoptilolite from Crshing Tests

For a size distribution to follow the straight line perfectly, the sieving should be done wet on a 400 mesh sieve first to wash the fine particles adhering to the coarse particles through the 400 mesh sieve. After the + 400 mesh material has been dried, it should then be sieved in the usual manner. Without the wet sieving step, deviations will exist in the - 200 mesh material. Since there appeared to be a degradation of the clinoptilolite particles when the material was screened, only dry sieving results will be reported here.

In Table D-II, cumulative size distributions are tabulated for the 4 min, closed-circuit, rod milling test; the open-circuit, roll mill, crushing test; and the composite of the crushed 3600 lb sample (this was calculated from weight distributions and the data in Table D-I).

TABLE D-II
CUMULATIVE SIZE DISTRIBUTIONS OF PRODUCTS
FROM CRUSHING TESTS

<u>Size,</u> <u>Tyler Mesh</u>	<u>4 min</u> <u>Rod Mill Cycle Test, %</u>	<u>Open-Circuit</u> <u>Roll Mill Test, %</u>	<u>3600 lb</u> <u>Composite, %</u>
8	100.0	100.0	100.0
10	99.5	88.2	99.7
14	74.6	72.6	71.4
20	55.7	54.4	54.6
28	43.6	40.0	43.3
35	35.1	30.4	34.3
48	26.2	23.6	27.6
65	20.0	17.8	21.0
100	13.8	12.5	14.6
150	10.0	8.9	10.6
200	7.0	6.8	5.9
270	0.5	3.2	1.1
400	0.2	1.5	0.3

In Figure D-1, a cumulative size distribution plot is presented. This figure indicates that all the products are quite similar; that is, the roll mill operated in closed and open circuit and the rod mill in closed circuit yield similar particles. As mentioned above, the sharp deviation of the size distribution plot below 200 mesh is probably not real but results from the dry screening procedure that was used. The equation of this line is

$$y = 100 \left(\frac{x}{1650} \right)^{0.78}$$

where x is the size in microns and y is the cumulative percent of material finer than size x.

From this plot, it can be seen that the maximum recovery of material in the 10 x 20 mesh fraction might be expected to be about 44%, about 31% in the 20 x 50 mesh fraction, and about 25% in the -50 mesh fraction. The closed-circuit roll crushing of the 3600 lb sample produced 47, 23, and 30% recovery in the respective fractions.

It is surprising that the slope of the size distribution curve for clinoptilolite is as large as 0.78. Usually materials of this nature give size distributions with excessive quantities of fines which then yields an exponent of about 0.4 instead of 0.78. This may result from the tendency of the clinoptilolite to break into two pieces rather than into fragments having a complete size distribution. For example, if a piece of quartz is struck a single blow with a hammer, the fragments will have a size distribution similar to the equation given above. If a piece of clinoptilolite is struck with a hammer, it seems to tend to break into a few coarse pieces together with a distribution of fine fragments. This would give rise to the size distribution with the greater slope. Possibly higher circulating loads in the crushing device would take advantage of this fracture mechanism and would result in the production of slightly less fine material.

APPENDIX E

APPENDIX E

PROCESS ECONOMICS

TABLE E-1

CAPITAL COST ESTIMATE FOR ION EXCHANGE

(Basis, 30,000 gal/day;
Feed, Condensate from Stored Purex Waste)

<u>Equipment and Construction Costs</u>	<u>Dollars</u>
(1) Feed Pumps; 2, 3 HP at \$100/HP	600
(2) Tanks; 2, 10,000 gal carbon steel at \$2/gal	40,000
(3) Stripper Feed Pumps; 2, 2 HP at \$100/HP	400
(4) Stripper Reboiler; carbon steel shell, stainless steel tube bundle, 50 ft ² , 1,400,000 Btu/hr.	4,000
(5) Stripper Tower; 10 ft of 12 in. schedule 40 pipe	2,500
(6) Feed Preheater; carbon steel shell, stainless steel tube bundle, 18 ft ² , 750,000 Btu/hr	2,000
(7) Condenser; carbon steel shell, stainless steel tube bundle, 37 ft ² , 500,000 Btu/hr	3,000
(8) Decanter	250
(9) Bottoms Cooler; carbon steel shell, stainless steel tube bundle, 50 ft ² , 800,000 Btu/hr	4,000
(10) Bottoms Pump; 1, 2 HP at \$100/HP	200
(11) Precoat Filter; stainless steel, 10 ft ² filtering area	8,000
(12) Anion Column; 28 ft ³	3,000
(13) Initial Anion Charge; 14 ft ³ \$80/ft ³	1,120
(14) Cation Column; 12 ft ³	1,200
(15) Initial Cation Charge; 6 ft ³ at \$25/ft ³	150
(16) Initial Filter Precoat; 1.7 ft ³ at \$25/ft ³	40
(17) HNO ₃ Addition System; tank and pump	3,550
(18) NaOH add system, tank and pump	2,100
(19) Precoat Makeup System	2,000
(20) Instruments; 22 points at \$300/point	6,600
<u>TOTAL</u>	86,000
Equipment Costs	86,000
Installation; 15% equipment costs	13,000
Contractors Markup; 20% equipment costs	17,000
Bid Price	116,000
Piping; 30% bid price	35,000
Building; 40 x 40 x 20 ft at \$2/ft ³	80,000
Total Equipment and Installation Costs	231,000
Design; 15% total equipment and installation costs	35,000
Contingency; 40% total equipment and installation costs	93,000
<u>TOTAL ESTIMATED COST</u>	~360,000
<u>Estimated Operating Costs</u>	<u>\$/day</u>
(1) Building Amortization	1.10
(2) Depreciation; 20% of major equipment per year	46.08
(3) Maintenance; 5% of major equipment per year	11.52
(4) Labor; 24 man-hr/day at \$7.50/man-hr (includes direct and indirect)	90.00
(5) Steam at \$1.00/1000 lb	51.50
(6) Water at \$90/10 ⁶ gal	4.33
(7) Filter Precoat; 0.24 ft ³ /day at \$25/ft ³	5.70
(8) Cation Resin; 0.2 ft ³ /day at \$25/ft ³	5.00
(9) Anion Resin; 2% replacement/wk at \$80 ft ³	3.20
(10) Chemicals, Utilities and Burial	6.00
<u>TOTAL</u>	~225.00
Estimated Cost = \$225/30,000 gal = \$7.50/1000 gal	

TABLE E-II

CAPITAL COST ESTIMATE FOR CONDENSATE EVAPORATION

(Basis, 30,000 gal/day;
Feed, Condensate from Stored Purex Waste)

<u>Equipment and Construction Costs</u>	<u>Dollars</u>
(1) Pumps; 1, 2HP at \$100/HP	200
(2) Tanks; 2, 10,000 gal, carbon steel at \$2/gal	40,000
(3) Feed Pumps; 2, 2HP at \$100/HP	400
(4) Concentrator; 2, carbon steel shell, stainless steel tube bundle, 220 ft ² , 6,000,000 Btu/hr	50,000
(5) Condensers; 2, carbon steel shell, stainless steel tube bundle, 200 ft ² , 6,000,000 Btu/hr	20,000
(6) Condensate Tank; 2, 2000 gal, carbon steel at \$2/gal	8,000
(7) Bottoms Tank; 1, 250 gal stainless steel at \$15/gal	3,750
(8) Condensate Pumps; 3, 2HP at \$100/HP	600
(9) Instruments; 18 points at \$300/point	5,400
<u>TOTAL</u>	128,350
Installation; 15% equipment costs	19,000
Contractors Markup; 20% equipment cost	26,000
Bid Price	173,350
Piping; 30% bid price	52,000
Building; 40 x 40 x 20 ft at \$2/ft ³	80,000
<u>TOTAL EQUIPMENT AND INSTALLATION COST</u>	305,350
Design; 15% total equipment and installation costs	45,650
Contingencies; 40%	122,000
<u>TOTAL ESTIMATED COST</u>	~473,000
<u>Estimated Operating Costs</u>	<u>\$/day</u>
(1) Building Amortization; 5%/yr	1.10
(2) Depreciation; 20% of major equipment per year	70.32
(3) Maintenance; 5% of major equipment per year	24.70
(4) Labor; 12 man-hr/day at \$7.50/man-hr (includes direct and indirect)	90.00
(5) Steam; 12,000 lb/hr at \$1.00/1000 lb (1 $\frac{1}{4}$ lb steam required for 1 lb evaporation)	288.00
(6) Water; 400,000 gal/day at \$90/10 ⁶ gal	36.00
(7) Utilities	1.00
<u>TOTAL</u>	~511.00
Estimated Cost = \$511/30,000 gal	
= \$17/1000 gal	

TABLE E-III
COST COMPARISON*
LOW-LEVEL WASTE TREATMENT PROCESSES

<u>Estimates</u>		<u>\$/1000 gal</u>
Ion-Exchange Process	30,000 gal/day	7.50
Reevaporation (HAPO)	30,000 gal/day	17.00
Chemical Flocculation (HAPO)	15,000 gal/day	9.00
<u>Actual Costs** (a)</u>		
(1) <u>Evaporation</u>		
BNL	1,000 gal/day	103.00
KAPL	3,000 gal/day	65.00
Bettis	4,600 gal/day	38.00
(2) <u>Chemical Flocculation</u>		
Rocky Flats	4,300 gal/day	9.00
LASL	38,000 gal/day	12.00

* All costs normalized to 20 year building and equipment amortization.

** Actual costs were not corrected for increases from 1955 to present.

(a) A. B. Joseph. Radioactive Waste Disposal Practices in the Atomic Energy Industry--A Survey of Costs, NYO-7830, December 31, 1955.

FIGURE E-1
Evaporation Costs for Condensate Treatment