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THE RECOVERY AND PURIFICATION OF MULTI-KILOCURIEQUANTITIES OF FISSION PRODUCT STRONTIUM BY CATION EXCHANGE

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THE RECOVERY AND PURIFICATION OF MULTI-KILOCURIE
QUANTITIES OF FISSION PRODUCT STRONTIUM BY CATION EXCHANGE

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Fission Product Chemistry
 Chemical Research
 HANFORD LABORATORIES OPERATION

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HANFORD ATOMIC PRODUCTS OPERATION
 RICHLAND, WASHINGTON

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I. INTRODUCTION

In mid-summer 1960, the Hanford Atomic Products Operation was asked by the AEC to undertake the recovery and purification of slightly over a megacurie of strontium-90 from the Hanford fission product wastes. This heat-generating fission product was designated to be the power source for five thermoelectric power units developed by the Martin Company for the AEC's Division of Reactor Development¹. The five units were: a small 5 electrical watt Weather Bureau unit, two 10 watt and two 60 watt units for the Navy and Coast Guard. The 10 electrical watt units required 40,700 curies each of strontium-90 at the time of loading, and the 60 watt units required 245,000 curies, for a total of about 650,000 curies. To allow for processing losses during pelletization and packaging a decision was made that Hanford should recover and purify slightly over a megacurie of strontium-90. A very tight time schedule was imposed. The Weather Bureau unit was scheduled to depart for the Arctic not later than July 10, 1961.

Fission Product Chemistry Operation was given the twofold responsibility of (a) devising the necessary precipitation-type flowsheet by which a strontium crude concentrate could be isolated from the Purex 1WW waste, and (b) developing a final purification ion-exchange process and then, using Purex strontium crude as feed solution, producing 60,000 curies of highly purified fission-product strontium-90. Chemical Development Operation was given the task of developing a solvent extraction flowsheet and processing the major part of the Purex strontium concentrate in an idle solvent extraction pilot plant known as the Hot Semiworks. The two approaches were taken to give an added margin of success and to meet the tight time schedule for the Weather Bureau unit. This document reports the work performed by the Fission Product Chemistry Operation.

Strontium-90 was chosen as a heat source because of its relatively long half-life (28 years), adequate specific power (0.47 thermal watts per gram of total strontium), its high fission yield and its relative freedom from troublesome associated gamma activity. In order to take advantage of these desirable properties, specifications requiring rigorous purification from gamma-emitting fission products and heat-diluting inert contaminants were imposed. The specifications required less than 0.0015 curies of hard gamma (>2 Mev) and less than 0.15 curies of soft gamma (.02 to 1.0 Mev) per gram of strontium. Less than 25 percent total non-strontium impurities could be tolerated. Because of unit design complications, specifications imposed strict requirements on the maximum strontium-89/strontium-90 ratios at the time of loading. Although strontium-89 is a beta emitter, it has a 51-day half life and the wattage far exceeds that of strontium-90 in short-cooled material.

A strong interest in strontium-90 had been anticipated and the necessary background laboratory work for the Purex flowsheet was nearing completion. This flowsheet² provided for the removal of fission-product rare earths and strontium from the Purex 1WW waste by an "acid-side" double sulfate precipitation with a lead carrier. The sulfate precipitate was converted to the carbonate by a basic sodium carbonate metathesis and the carbonate precipitate dissolved in nitric acid. Strontium was then separated from lead and the rare earths by a low-acid oxalate precipitation. Because of operational and new equipment difficulties, the flowsheet was not successfully used until late November, 1960. Thus the composition of the Purex strontium crude was not known until early December. The approximate composition was as follows:

Sr = 0.32 g/l (25.6 curies/l)

Ca = 0.1 g/l

Ba = 0.05 g/l

Fe = 8.0 g/l

Pb = 5.6 g/l

Na = 10.0 g/l

Zr-Nb-95 = 70 curies/l

Ce-Pr-144 = 750 curies/l

At the time the strontium purification program was initiated, A-Cell of the 325A Facility contained a six-column ion exchange processing unit. The unit had been designed for testing a process for the separation of promethium-147 from the adjacent rare earths and three successful "cold" runs had been completed in the equipment. Because of the availability of in-cell equipment and a preliminary flowsheet, a decision was made to use ion exchange for the final strontium purification to be done in the 325A Facility. Consequently, the five-month period beginning in August, 1960 was devoted to "cold" flowsheet demonstration runs, in-cell equipment changes, and associated laboratory work leading to the final flowsheet. The research conducted during this period was severely handicapped because the composition of the feed solution could only be guessed and was not known until December, just prior to the first full-scale "hot" run.

II. SUMMARY AND CONCLUSIONS

In seven production runs, 75,000 curies of strontium-90 were isolated and purified in the ion-exchange equipment of Hanford Laboratories High Level Cells². The production goal (60,000 curies of purified strontium-90) and the time schedule were met or exceeded and the product exceeded the customer's purity requirements. The hot-cell-purified strontium made possible the completion on time of the Weather Bureau power source and the Martin 10 watt SNAP VII-A and VII-C units. The

1

isotopic purity of the strontium product was 56 percent strontium-90. The chemical purity was greater than 95 percent strontium. The zirconium-niobium-95 contamination of the product was less than 5×10^{-5} curies per curie of strontium-90, and the cerium-praseodymium-144 contamination was less than 1×10^{-5} curies per curie of strontium-90.

The final run yielded 16,500 curies of strontium-90 in an eight liter product solution. The product was > 98 percent strontium and contained less than one curie of zirconium-niobium-95 or cerium-praseodymium-144. Eleven days of continuous operation were required to complete the run.

III. DISCUSSION

A modified version of the rare earth ethylenediaminetetraacetate separation process developed at the Ames Laboratory^{4,5} was used for the three "cold" rare earth separation hot-cell runs and served as a starting point for the strontium work. In a general simplified case, cations A, B, C, and D, absorbed in a relatively homogeneous band on a column of cation resin, can be separated into individual bands of each ion when the cations are eluted onto and down an elution column of cation exchange resin onto which cations of another species, X, have been absorbed, if certain conditions are satisfied. These conditions are:

- a) The eluting agent must form soluble complexes with the ions A, B, C, D and X over a reasonably broad pH range.
- b) The magnitude of the stability constant defining each complex must be significantly different for each ion.
- c) The complex formed between the eluting agent and ion X must be stronger, i.e., have a greater stability constant, than the complexes formed between the eluting agent and the ions to be separated.

On continued elution, the bands of the individual separated ions move down the column, each band directly above the preceding one with no vacant or dead space between the bands. Each band contains only one kind of ion, with binary

mixing occurring only in the relatively narrow zone at the leading or trailing edge of the band where it is in contact with the preceding or succeeding band. Physical separation of ions A, B, C, and D is accomplished by collecting the effluent solution into fractions as the bands are eluted off the bottom of the final column or by valve manipulations which isolate the bands and divert one or more onto other ion exchange columns.

Theoretical equations have been derived⁶ which describe this type of separation and which can be used to predict the distance which an absorbed band containing two elements must be eluted, under equilibrium conditions, before the band subdivides into two sub-bands, each containing only one kind of ions. These equations are similar to those used to describe the action of a solvent extraction column in total reflux. The number of band displacements required to effect a separation of the bulk mixture of absorbed ions into pure components is given by equation (1).

$$\sqrt{\epsilon} = \frac{1 + \epsilon N_0}{\epsilon} \quad (1)$$

where N_0 = mole fraction of the first component in the binary mixture.

$$\epsilon = K_{\text{sep}} - 1 = \frac{K'_A}{K'_B} - 1 \quad (2)$$

and

K_{sep} = the separation coefficient,

K'_A = stability constant of the complex formed between species A and the eluting agent,

K'_B = stability constant of the complex formed between species B and the eluting agent.

The values of $\log K'$ for the calcium, strontium and barium ethylenediamine-tetraacetate complexes are given in the literature⁷ as 10.7, 8.63 and 7.76 respectively. From these values, and assuming equal-molar binary mixtures, separation coefficients can be calculated for the separation of calcium from

strontium and for the separation of strontium from barium.

$$K_{\text{sep}}(\text{Ca}, \text{Sr}) = \frac{K'_{\text{Ca}}}{K'_{\text{Sr}}} = 118$$

and

$$K_{\text{sep}}(\text{Sr}, \text{Ba}) = \frac{K'_{\text{Sr}}}{K'_{\text{Ba}}} = 7.4$$

The number of band displacements which a homogeneous band of two elements must be eluted can be calculated from these values with equation (1). They are:

$$\gamma(\text{Ca}, \text{Sr}) = 0.51 \text{ band length}$$

$$\gamma(\text{Sr}, \text{Ba}) = 0.66 \text{ band length}$$

These values of K_{sep} and γ can be compared with neodymium-praseodymium values of

$$K_{\text{sep}}(\text{Nd}, \text{Pr}) = \frac{K'_{\text{Nd}}}{K'_{\text{Pr}}} = 2.0$$

$$\text{and } \gamma(\text{Nd}, \text{Pr}) = 1.5 \text{ band lengths}$$

or with europium-gadolinium values of

$$K_{\text{sep}}(\text{Gd}, \text{Eu}) = \frac{K'_{\text{Gd}}}{K'_{\text{Eu}}} = 1.02$$

and

$$\gamma(\text{Gd}, \text{Eu}) = 50 \text{ band lengths}$$

Since gross quantities of the rare earths have been separated from each other about as predicted by equation (1), the separation of strontium from calcium or barium would be expected to be relatively easy. Ion "X" serves as a barrier and forces the reabsorption and consequent "banding" of the other ions behind it⁶.

A. HOT CELL ION-EXCHANGE EQUIPMENT

A-Cell of the 325A Building High Level Facility is a stainless-steel-lined cubicle 15' wide, 8' deep and 11' high (from the floor up to the General Mills' track). The cell is equipped with three large lead glass viewing windows, two sets of Model 8 manipulators, a through-the-wall periscope and a sufficient number of through-the-wall services. The six-column ion-exchange unit shown in the piping schematic of Figure 1 was installed in the cell. The columns were mounted on two racks toward the rear of the cell and the stainless steel tanks 1, 1A, 3, 4 and 7 were placed under the windows at the front of the cell. A heavy 4' x 6' stainless steel tray was placed over tanks 1A, 3 and 7 in a position immediately in front of one of the two operating windows. Tanks 5 and 6 (calibrated 5 gal. Pyrex glass carboys used to collect, sample and measure the volume of effluent solution from the columns) were placed on one corner of the stainless steel tray. The columns were so placed that by using all three windows they could be viewed over their entire length. All valves not operated by air pistons were placed to be operated by the Model 8 manipulators. The air-operated valves are 1/4" Durimet 20 Fig. F118S screwed-end type-F plug valves. All other valves are Hoke stainless steel needle valves or Dahl stainless steel demi-diaphragm valves. The last two kinds of valves were modified with extended handles, so they could be easily operated by the Model 8 manipulators. All solution lines within the cell are stainless steel tubing (type 304, 304L or 347) and the fittings are stainless steel Crawford Swagelok.

One piece of equipment not shown on Figure 1, but which proved to be invaluable to the success of this project, was a 4-position lead-shielded sample holder placed at the front of the cell in such a position that slits in the sample holder lined up with two through-the-front-face step ports. Thus, with scintillation crystals in the two ports, connected to a 400-channel pulse height analyzer,

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it is possible to determine the gamma spectrum of analytical samples without removing them from the cell⁸.

Resin is slurried into the columns from the front face of the cell through the resin addition line (wall 115) by applying a 10- to 15-lb air pressure to a six-gal. polyethylene bottle containing a slurry of resin in water. The resin addition line extends into the resin and terminates at a point near the bottom of the bottle. Resin is removed from the columns by pumping water upflow through the columns and backwashing the resin out the top of the columns, then diverting the resin through the resin line (wall 257) to a shielded resin disposal cask. No difficulty has been encountered in adding resin to the columns. A water backwash is sufficient to remove resin from 1-inch and 2-inch diameter columns but not sufficient to remove all of the resin from 4-inch diameter columns. The remaining resin is removed from the 4-inch diameter columns by floating it out on a dense solution of aluminum nitrate.

The vacuum-vent-pressure system for each column is used to adjust the liquid level height in the column, for column degassing, and for a variety of other uses during column operation.

The pump shown in Figure 1 is a remote-head, stainless-steel, double-diaphragm, Lapp Pulsafeeder Model CPS2. The reagent head with one diaphragm is located in the cell and a second diaphragm is mounted near the pump, external to the cell. A one-inch diameter line, hydraulically connecting the two diaphragms (not shown in Figure 1), contains water.

Raw feed solution is transferred to feed tanks 1 or 1A from B-Cell or directly from a shielded transfer cask by vacuum transfer. After feed adjustments, the prepared feed is pumped through one or more of columns 1, 2 or 3 in series and to tanks 5 or 6, where it is measured and sampled. The feed-effluent solution is then transferred by vacuum to waste tanks 3 or 4. The column piping is such that the columns may be used singly or with two or more of them in series.

On the initial installation, columns 1, 2 and 3 were 2-inch diameter Pyrex glass pipe 6 feet long with stainless steel end fittings. These columns were jacketed with 3-inch diameter Pyrex glass pipe through which thermostated water circulated for temperature control. Columns 4, 5 and 6 were 1-inch diameter Pyrex glass pipe, 6 feet long, jacketed with 2-inch diameter Pyrex pipe for temperature control. This system of six columns was used successfully for the three "cold" rare earth runs and for the first three "cold" strontium runs. The columns were then removed and replaced by larger columns when it became evident that a production effort would be made. On revision, columns 1, 2 and 3 are 4-inch diameter Pyrex glass pipe 9 feet long, jacketed with 6-inch diameter Pyrex glass pipe. Columns 4, 5 and 6 are 2-inch diameter Pyrex glass pipe 9 feet long jacketed with a 3-inch diameter Pyrex glass pipe. The end fittings are of stainless steel.

B. PRELIMINARY "COLD" INVESTIGATIONS

The first cold strontium run (run 8-1) attempted in the hot cell ion-exchange equipment was initiated on August 11, 1960. Column number 1 was filled to a depth of 5 feet with Dowex 50W, X-12 (50-100 mesh) resin in the ammonium cycle. Columns 2, 3, 4, 5, and 6 were filled to the same depth with the same resin, then converted to the mixed copper-hydrogen cycle by regenerating with an excess of a solution 0.5M in CuSO_4 and 0.5M in H_2SO_4 . All columns were water washed.

The absorption column, column No. 1, was loaded to saturation from a feed solution containing iron, lead, calcium, strontium and barium in a molar ratio of 1:2:5:5:5 respectively. The feed was prepared by dissolving the nitrates of these metals in water. The feed solution pH was about 0.5. Following the absorption cycle, the resin bed in Column 1 was washed with water, connected in series with column No. 2 and the system eluted with a 0.03M solution of ethylenediaminetetraacetic acid (hereafter designated as EDTA) buffered to pH 8.4 with ammonium hydroxide. The two resin columns were heated to 50°C by using the water

jackets. The EDTA solution was degassed by passing it through a line heated to 90°C and at the same time pulling a partial vacuum above the solution. A white precipitate became visible 30 minutes after the start of elution and continually increased. The run was terminated after 7 hours of elution. The precipitation occurred toward the top of column No. 1. The precipitate dissolved in dilute ammonium hydroxide, so it was assumed that the precipitate was the insoluble acid-form of the chelating agent, formed as a result of contact of the chelating agent with some hydrogen ions absorbed during the absorption cycle. Previous beaker experiments had indicated that the metal ions, mixed with solutions of EDTA at pH 8.5, at concentrations fourfold higher than in the ion exchange run, are soluble both at 25°C and at 85°C.

The resin columns for run 8-2 were conditioned in the same manner as for the previous run. Column No. 1 was loaded to saturation from a feed solution containing equal-molar amounts of lead, calcium, strontium, and barium. The nitrate salts of these metals were dissolved in water and the pH adjusted to 4.5 to ensure that no hydrogen ions were loaded with the feed. Once again columns 1 and 2 were connected in series and eluted with a 0.03M solution of EDTA buffered to pH 8.4 with ammonium hydroxide. The column temperature was 50°C and the degasser was set at 90°C. As with the previous run, the precipitate formed very soon and the run was terminated after 5 hours.

The evidence from runs 8-1 and 8-2 seemed to indicate that the solubility determinations made in beakers were in some way not valid for ion-exchange columns. It was therefore erroneously concluded that the precipitation trouble was due to the use of an eluting agent that was too concentrated.

In run 8-3, the resin columns were preconditioned exactly the same as for the two previous runs. The run 8-3 feed solution had a pH of 2.3 and was prepared by dissolving the nitrate salts of iron, lead, calcium, strontium and barium in

water in the molar ratio of 1:2:5:5:5 respectively and adjusting the pH with ammonium hydroxide. Following the absorption and water-wash cycles, columns 1 and 2 were connected together in series and eluted with a 0.015M solution of EDTA buffered to pH 8.82 with ammonium hydroxide. The column temperature was 60°C and the degasser was set to operate at 90°C. The elution proceeded satisfactorily for 5 hours and then a precipitate started to form. The precipitate formation increased and the run was terminated after an additional 3 hours of operation.

An investigation revealed that the temperature control of the degasser was not at all times effective and had at times during the three runs operated above 95°C. On boiling for only 2 minutes in a beaker, the pH of the EDTA solution decreased to 7.3. The run was therefore started up again with the degasser operating at 76°C. The precipitate dissolved after 50-60 hours of elution and no trouble was encountered during the remainder of the run. The precipitation must have resulted from the formation of one or a mixture of the following: H_4Ch , $MHCh^-$, or MH_2Ch where M is one of the divalent metal ions involved and Ch represents the tetraacetate anion. It is known that, for many metals, the species $MHCh^-$ and MH_2Ch are more insoluble than the corresponding ammonium salts, MNH_4Ch^- or $M(NH_4)_2Ch$.

The absorbed band of run 8-3 was eluted through all five of the elution columns. After elimination of the precipitation problem, the copper-hydrogen trailing edge was very sharp. The average elution flow rate was 3.5 ml/min, cm^2 .

The iron remained on column 1 and would not elute with EDTA at the pH used. The non-elution of iron was not in any manner detrimental to the progress of the run (since it could be readily eluted with dilute acid after completion of the run). The elution band was collected in fractions as it eluted off the bottom of column 6 and the content of each was determined. The resulting elution curve is shown in Figure 2. In order to save time, the run was terminated before much of the barium

ION-EXCHANGE EQUIPMENT

3 2 IN. COLUMNS, 6 FT. LONG
-AND-
3 1 IN. COLUMNS, 6 FT. LONG

ELUANT SOLUTION

EDTA 0.015 M
pH 8.82

FEED SOLUTION MOLAR RATIO

Fe - 1
Pb - 2
Ca - 5
Sr - 5
Ba - 5

OPERATING CONDITIONS

AVERAGE FLOW RATE = 3.5 ml/min, cm^2
ELUTION TEMPERATURE = 50°C

BAND WAS ELUTED THROUGH ALL SIX COLUMNS WITH EDTA.
THE IRON REMAINED ON THE ABSORPTION COLUMN AND DID NOT ELUTE.

FIGURE 2

Strontium Elution Curve

had been eluted. The separation is excellent and the flat-top bands indicate that near-equilibrium conditions were in effect during the run.

The in-cell equipment changes described in Section I were made following the completion of run 8-3. Early in November 1960 it became known that the strontium crude, produced by the Chemical Processing Department, would not have the anticipated composition. The iron and lead would be considerably higher than strontium, calcium about the same as strontium, and barium much less than strontium. In addition to these impurities, the feed would contain a prohibitively high concentration of cerium-praseodymium-144. It was believed that the sodium concentration would be a modest 1-2 grams per liter. The high iron, lead, and rare earth content of the feed solution forced a revision in the ion-exchange flow sheet. The absorption by the resin of all of the cations in the feed solution could no longer be tolerated. The high concentration of iron and lead would saturate the resin and result in a very low strontium production per run. In addition, the large amount of intensely radioactive cerium-144, if absorbed on the resin, would rapidly destroy the resin. Fortunately, the undesirable impurities, iron, lead and rare earths, form much more stable complexes with most chelating agents than does strontium. It should, therefore, be possible to add sufficient complexing agent to the feed to render the iron, lead and rare earths unabsorbable without much effect on the strontium. This plan was tested in run 9-1.

In preparation for run 9-1, column 1 (now a 4-inch diameter column) was filled to a depth of approximately eight feet with Dowex 50W, X-12 (50-100 mesh) resin in the ammonium cycle. Column 1 was to be the absorption column. Columns 2 and 3 (also 4-inch diameter columns) as well as columns 4, 5 and 6 (2-inch diameter columns) were filled to a depth of eight feet with the same resin, and the resin was then converted to the copper-hydrogen cycle with a conditioning solution: $0.5M\ H_2SO_4$, $0.5M\ CuSO_4$. The columns were all water washed. A feed solution was prepared by dissolving metal nitrate salts in water. The feed volume was 257 liters and the composition as follows:

	<u>Mole Ratio</u>	<u>Grams/Liter</u>
Sr	1.0	0.80
Ca	0.8	0.31
Pb	1.7	3.23
Fe	32.7	16.68
Ba	0.5	0.63
EDTA	34.4	91.75

Concentrated ammonium hydroxide was added to adjust the feed solution pH to 4.5. The feed solution was pumped through the column at an average rate of about 4 ml/min, cm². The feed-effluent solution analysis showed that the iron and lead had passed through the column without absorption and that all of the strontium had absorbed. Following a water wash of column 1, columns 1 and 2 were connected in series and the elution initiated with 0.015M EDTA, buffered to pH 8.80 with ammonium hydroxide. The elution flow rate was approximately 3 ml/min, cm². It was decided that the applicable separation coefficients are sufficiently large and the reaction kinetics sufficiently fast at 30°C that elevated temperature operation was not needed to obtain good separation at the intended elution flow rate. Therefore, the columns were operated at ambient cell temperature (30 to 35°C) and the degasser was not used. The elution band was moved down all of the columns, new columns being added to the system as needed. As the band eluted off column 6, the effluent solution was collected in fractions and the composition of each was determined. A small amount of lead appeared in the first few effluent samples, indicating that not quite enough EDTA had been added to the feed solution. The separation of strontium from calcium and barium was equally as good as the separation obtained in run 8-2.

The results of run 9-1 confirmed the feasibility of preventing the absorption of iron, lead and the rare earths by the addition of a complexing agent to the feed

solution and also indicated that ambient-temperature operation is satisfactory. They also pointed out the great difficulty of accomplishing this action when depending on the addition of a complexing agent exactly stoichiometric with the sum of iron, lead and rare earths. Errors in determining the exact solution volume in the in-cell feed tanks, errors associated with the analytical determinations of these impurities in highly radio-active solutions (high dilutions required) and the high molar ratio of impurities to strontium all combine to make the stoichiometric matching of the complexing agent with the impurities an impossible task.

A process wherein an excess of complexing agent is added and the elements complexed controlled by the pH of the solution seemed to be an answer to the problem. Such a process is possible when, (a) the complexing agent is a weak acid, (b) the free acid form of the complexing agent is reasonably soluble, and (c) the strontium complex is significantly weaker than the complexes formed between the complexing agent and iron, lead and the rare earths. The complexing agent N-hydroxyethylethylenediamine-triacetic acid, hereafter designated HEDTA, meets these three qualifications.

Feed solutions of the following composition were prepared and then adjusted to the desired pH with ammonium hydroxide:

Sr	0.0045 <u>M</u>
Ca	0.0038 <u>M</u>
Pb	0.0078 <u>M</u>
Fe	0.15 <u>M</u>
Na	0.58 <u>M</u>
HEDTA	0.18 <u>M</u>
Ce	0.00005 <u>M</u>

Cerium and strontium tracers were added and the solutions were passed through 30 cm long columns of Dowex 50W, X-12 (50-100 mesh) resin at 5 ml/min, cm². Standard breakthrough curves were determined in each case for both cerium and strontium. The results

of these experiments are given in Table I and indicate that high strontium loadings with negligible absorption of cerium can be achieved in the neighborhood of pH 4.

TABLE I

EFFECT OF pH ON ABSORPTION OF CERIUM
AND STRONTIUM FROM HEDTA COMPLEXED SOLUTION

(See text for feed composition)

<u>pH</u>	<u>Cerium Results</u>	<u>Strontium Results</u>
2	All cerium absorbed	Resin capacity = 12 g Sr/l at 7% breakthrough
3	30% of cerium absorbed	Resin capacity - 10 g Sr/l at 1% breakthrough
3.5	4% of cerium absorbed	Resin capacity - 12 g Sr/l at 6% breakthrough
4.0	<4% of cerium absorbed	Resin capacity - 12 g Sr/l at 10% breakthrough
5.0	<1% of cerium absorbed	Resin capacity = 10-12 g Sr/l at 10% breakthrough

One final in-cell "cold" run was made to examine the feed pretreatment with HEDTA on a full-scale feed makeup. The 8-foot resin bed in column 1 was conditioned and converted to the ammonium cycle. The resin was the same as that used for run 9-1. A 513 liter feed solution of composition

Sr	=	0.70 g/l
Ca	=	0.15 g/l
Pb	=	1.60 g/l
Fe	=	10.0 g/l
Na	=	1.30 g/l
Ce	=	0.10 g/l
HEDTA	=	65 g/l

was prepared by dissolving the metal nitrate salts in water. The solution was transferred to the in-cell feed make-up tanks and measured volumes of partially neutralized

HEDTA solution were added. A final feed pH adjustment to 3.90 was accomplished by the addition of a small volume of concentrated ammonium hydroxide. The prepared feed was pumped through column 1 at a flow rate of approximately 10 ml/min, cm². The column effluent was collected in 18 liter fractions and analytical samples withdrawn from each. No strontium could be detected in the feed effluent solution. The column capacity thus appeared to be at least 360 grams of strontium from the synthetic feed used. This run concluded the cold testing. Full-level operation with Purex-produced strontium crude began on December 25, 1960.

C. SUMMARY OF THE FULL-LEVEL PRODUCTION RUNS AND THE RESULTS OBTAINED

Since the operational procedure was similar for each of the runs, a brief description will be given prior to a more detailed description of each run. The strontium crude was transported from the Purex Chemical Separations Plant to the 325A Building in 200-gallon shielded casks. Once in the 325A Building, a loaded cask was placed in a special enclosed loading station and connected via 1/2" polyethylene tubing to a shielded 1/2" stainless steel line leading directly to one or more in-cell storage tanks. Fresh resin was used for each run. The resin placed in the elution columns was completely saturated with barrier ion "X" by passing a nitrate solution of ion "X" through the columns, followed with a water flush.

A stock solution of HEDTA was prepared by dissolving the organic acid in water and ammonium hydroxide followed by a final volume and pH adjustment to give a solution 2.3M HEDTA at pH 4.0. Because of the large volume of feed solution required per run and the relatively small size of feed tanks 1 and 1A, it was necessary to make up more than one batch per run. Raw feed solution was measured into either tank 1 or 1A, a given volume of HEDTA was added, and then ammonium hydroxide was added to give a pH of 3.9 to 4.2. The usual practice was to add 40% excess HEDTA beyond that required to complex the iron and lead (in order to assure that an excess was indeed present and that all the cerium would be complexed).

The prepared feed was then pumped through the feed absorption columns (column 1, 2 or 3) until an appreciable strontium breakthrough occurred. At that point the feed cycle was terminated and the columns washed free of excess feed solution with a wash solution, 0.1M HEDTA adjusted to pH 4.0 with ammonium hydroxide. The wash caused little or no movement of strontium.

Following the washing cycle, the absorption columns were connected in series with column 4 and the system eluted with a 0.030M solution of EDTA adjusted to pH 8.40 to 8.82 with ammonium hydroxide. All operations were at ambient cell temperature (30 to 35C). As the elution progressed, the strontium banded between the calcium and the barium. The position of the strontium could be determined visually by turning off all cell lights and observing the radiation glow produced by the strontium (Figure 3). Column 5 was valved into the system when needed.

By the time the strontium band moved to the bottom of column 5, the strontium band was relatively free of calcium, barium, or any other extraneous impurity. Elution was continued with the column effluent solution going to waste until all of the calcium was removed from column 5 and the strontium appeared in the effluent. At this point column 6 was valved into the system and the strontium band eluted onto the resin in column 6. When the strontium was entirely on column 6, a valve manipulation isolated column 6 from column 5, thus separating the strontium from the trailing barium band. The barrier ion "X" on column 6 served to confine the strontium on column 6. Thus, at the end of the elution cycle the purified strontium was absorbed in a compact band on the top portion of column 6 with the barrier ion occupying the resin bed below the strontium band.

During the several runs, copper, yttrium, and neodymium were used as the barrier ion on column 6. Each forms a much more stable complex with HEDTA than does strontium and could therefore be removed from column 6 by pumping a 0.1M solution of HEDTA

(adjusted to pH 4.0 with ammonium hydroxide) through the column without significant movement of the strontium band. A wash of water removed the complexing agent from the column and the purified strontium was then removed from the column by one of several desorbing agents. A 0.10M solution of HEDTA adjusted to pH 9.0 with ammonium hydroxide quickly and completely removed the strontium at an average concentration of 9 grams per liter. A 5M solution of ammonium nitrate will effectively remove the strontium at an average concentration in excess of 40 grams per liter. Four to six molar nitric acid is also a very effective eluting agent.

Run No. 1

Dowex 50W, X-12 (50-80 mesh) resin was placed in each column. The resin in columns 1 and 2 (used as absorption columns) was in the ammonium cycle. Columns 3, 4, 5 and 6 were used as elution columns and contained resin in a mixed hydrogen-copper cycle. The feed-cycle flow rate averaged 10 ml/min, cm². The absorption and elution cycles were performed at ambient cell temperature. Following the removal of copper from column 6 and a subsequent water flush, the strontium was removed with 6M HNO₃. The results of the run are given in Table II.

The greatest difficulties encountered in the run were the presence of excessive solids in the Purex crude feed and the unexpected reduction of some of the copper to metal on the resin. This reduction is accomplished by the intense radioactive field in the presence of EDTA. No reduction takes place in the absence of EDTA. These results were duplicated in cobalt source experiments. In addition, an unexpectedly low resin-absorption capacity resulted from the very high monovalent ion concentration in the feed solution (ammonium ion from neutralization of excess acid in the Purex feed). Several thousand curies of strontium-90 passed through the absorption columns and to waste because the analytical results on the feed effluent samples were slow coming from the analytical laboratory.

FISSION-PRODUCT STRONTIUM RECOVERY AND PURIFICATION

		<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>	<u>Run 4</u>	<u>Run 5</u>		<u>Run 6</u>		<u>Run 7</u>
						A	B	A	B	
<u>FEED SOLUTION</u>										
<u>Purex Crude</u>										
Volume	(liters)	673	250	435	617	537	345	558	211	762
Sr-90	(curies)	15,000	9,250	11,300	10,200	7,700	8,100	14,800	5,200	19,800
Zr-Nb-95	(curies)	4.0×10^4	1.7×10^4	4.3×10^5	1.4×10^4	6,800	5,700	6,300	3,500	8,700
Ce-Pr-144	(curies)	4.6×10^5	1.8×10^5	2.7×10^5	2.1×10^5	1.7×10^5	1.5×10^5	2.5×10^5	9.1×10^4	3.4×10^5
Fe	(gms/l)	8.0	8.1	9.0	4.47	4.5	7.8	10.9	7.8	7.8
Pb	"	4.5	5.7	4.0	3.9	3.9	2.7	2.6	2.7	2.6
Na	"	7.8	7.8	9.1	5.0	5.0	29.0	32	29	29
HNO ₃	"	0.6	0.6	0.6	56	60	62	63	62	63
<u>Prepared Feed</u>										
Volume	(liters)	763	287	506	710	1150	1,442	2,460	929	3,398
Dilution Factor		1.14	1.15	1.17	1.15	2.14	4.24	4.4	4.4	4.46
<u>Rework of Waste from Previous Runs</u>										
Volume	(liters)	_____	_____	_____	816	30	_____	_____	_____	_____
Sr ⁹⁰	(curies)	_____	_____	_____	3,800	1,500	_____	_____	_____	_____
<u>PRODUCT</u>										
Sr-90 in product	(curies)	5,100	8,350	9,500	12,400	13,600	_____	11,200	_____	16,500
Sr-90 in rework	(curies)	1,600	200	500	1,500	_____	_____	_____	_____	_____
<u>Product Purity</u>										
Sr	gms.	62	102	97	156	166	_____	137	_____	201
Ca	gms.	2	2	2	3.8	3.2	_____	< 1	_____	< 1
Ba	gms.	3	2	2	1.8	1.8	_____	< 1	_____	< 1
Ce-Pr-144	curies	< 1	< 1	< 1	0.04	0.07	_____	3.4	_____	< 1
Zr-Nb-95	curies	< 0.05	< 0.05	< 0.05	0.03	0.01	_____	0.47	_____	< 1

Run No. 2

Columns 2 and 3 were filled with ammonium cycle Dowex 50W, X-12 (50-80 mesh) resin and were used as absorption columns. Columns 4, 5 and 6 were filled with the same resin. The resin in columns 4 and 5 was then converted to the calcium cycle by passage of a calcium nitrate solution through them and the resin in column 6 was saturated with copper and hydrogen from a solution $0.5M$ $CuSO_4$, $0.5M$ H_2SO_4 . The use of calcium as the barrier ion on columns 4 and 5 served to restrain the strontium while permitting cerium as well as the strontium-90 daughter, yttrium-90, to pass through and on to waste, and avoided the partial column plugging caused by reduction of cupric ion to metallic copper. The strontium was removed from column 6 with $3M$ HNO_3 . Flow rates and temperature were similar to run 1. The results are shown in Table II. A considerable quantity of cerium-144 was inadvertently loaded onto column 2 from one batch of prepared feed because of an error in the HEDTA addition. The resin darkened and exhibited a higher pressure drop in a matter of 2 to 3 hours. The cerium was selectively removed by washing the resin with $0.1M$ HEDTA buffered to pH 4.0 with ammonium hydroxide and loading then continued.

The photograph shown in Figure 3 was taken through the lead glass window while the strontium band was on column 6. The photograph was taken shortly after the initiation of the copper-removal HEDTA wash. The faint glowing zone below the strontium band contains the yttrium-90 daughter. It is removed from the strontium zone by the HEDTA wash.

Run No. 3

At the start of run 3, columns 2 and 3 were filled with ammonium cycle Dowex 50W, X-12 (50-80 mesh) resin and were used as absorption columns. Columns 4 and 5 contained the same resin but in the calcium cycle and column 6 contained the same resin, however, in the copper-hydrogen cycle.

The first portion of the prepared feed contained an unusually large amount of solids and column 2 plugged after a very short feed cycle. The feed was then diverted

FIGURE 3

Absorbed Band of Purified Fission Product Strontium

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to column 3 and column 1 was filled with Dowex 50W, X-12 (20-50 mesh) resin in the ammonium cycle. After column 3 was loaded to a high breakthrough and the resin was water washed, the feed was diverted to column 1. The larger mesh resin in column 1 permitted a higher feed flow rate and allowed most of the solids to pass through. Following the complete loading of column 1, the resin was water washed and an attempt was made to load more feed through column 2 with only limited success. The loading cycle was therefore discontinued and the elution cycle initiated. The solution flow rates and temperature were approximately the same as in the first two runs.

Following the removal of the copper barrier from column 6 with an HEDTA wash, the strontium was removed with 0.1M HEDTA adjusted to pH 8.8 with ammonium hydroxide. The eluted strontium was collected in 4-liter pyrex glass jugs and is shown in the sequence of photographs in Figure 4. The strontium-90 concentration in the eluate was approximately 8 grams per liter. The results of the run are given in Table II. Acidification of the strontium-HEDTA solution to pH 2.0 permitted direct absorption of the strontium in the Decalso shipping cask and gave higher Decalso-loading capacities than can be obtained in the presence of competing sodium, ammonium, or hydrogen ions⁹. Consequently HEDTA elution was favored - when the shipping cask was available for loading. At other times it was necessary to elute with ammonium nitrate or nitric acid, since the HEDTA underwent radiolysis (with production of tarry products) on prolonged storage.

Run No. 4

Columns 1, 2 and 3 were filled with Dowex 50W, X-12 (20-50 mesh) resin in the ammonium cycle and were used as absorption columns. Columns 4 and 5 were filled with Dowex 50W, X-12 (50-80 mesh) resin in the calcium cycle. Column 6 contained Dowex 50W, X-12 (50-80 mesh) resin in the yttrium cycle. Except for occasional loading of some cerium due to insufficient HEDTA addition to the feed, the feed absorption and elution cycles were quite uneventful. The feed solution flow rate, elution flow rate and temperature were the same as the other runs. The Purex crude feed for this run

FIGURE 4

Removal of Strontium from Column 6

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and all subsequent runs was clarified by centrifugation at Purex and was relatively free of solids. Use of the coarser resin and centrifuged feed effectively eliminated difficulty with column plugging.

Prior to charging of freshly prepared feed to the absorption columns, 816 liters of rework material from the previous runs were passed through the absorption columns. The results of run 4 are given in Table II.

The strontium was removed from column 6 by elution with a 5M ammonium nitrate solution adjusted to pH 1.0 with nitric acid. All of the strontium was eluted in eight liters with over 11,000 curies being eluted in only four liters.

Run No. 5

Once again columns 1, 2 and 3 were filled with ammonium-cycle Dowex 50W, X-12 (20-50 mesh) resin, columns 4 and 5 with Dowex 50W, X-12 (50-80 mesh) resin in the calcium cycle and column 6 with yttrium-cycle Dowex 50W, X-12 (50-80 mesh) resin. In an attempt to increase the resin capacity for strontium, the feed solution was diluted with water by a factor of 2.14 during the preparation step. This does not change the ratio of divalent to monovalent ion concentrations in the feed but the dilution does increase divalent ion distribution factor relative to that of the monovalent ion. A second type of raw feed in which the sodium concentration was very high was used as part (column B, Table II) of the run 5 feed. This material was diluted by a factor of 4.24 during the feed preparation step.

The flow rates, temperature and elution sequences were the same as run 4. The results are given in Table II. The higher yield of strontium from a much less desirable feed indicates the success of the feed dilution step.

Run No. 6

Column and resin conditions at the start of run 6 were the same as those for run 5 except that neodymium was used as the barrier ion on column 6 in place of yttrium. Two different feed solutions were used in the feed preparation step (columns

A and B, Table II), but the dilution factor of 4.4 was kept constant throughout the loading steps. In an attempt to increase the yield of strontium per run, the three absorption columns were loaded twice. Feed solution was passed through columns 1, 2 and then 3 - each column was added to the series when needed - until a 25% breakthrough from column 3 was experienced. The columns were then washed free of feed solution with 0.1M HEDTA at pH 4.0 and then water. The three columns were then connected together in series with column 4 and the strontium eluted from columns 1, 2 and 3 onto 4 with 0.03M EDTA (adjusted to pH 8.4 with ammonium hydroxide). Following a water wash of column 4 to remove the EDTA, columns 1, 2 and 3 were then reloaded. The reloaded columns were then washed first with the HEDTA solution and then water. Columns 1, 2, 3 and 4 were then connected together in series and the elution commenced again.

The strontium band on column 6 was more than 200 cm. long. The strontium was removed from column 6 with 5M ammonium nitrate at pH 1.0. Unfortunately, a valve leak in the product removal line permitted a loss of approximately one third of the strontium so the product yield was less than run 5. The results are shown in Table II.

Run No. 7

This run was a duplicate of run 6 except a rerouting of the product prevented any losses via the defective valve. Nearly 15,000 curies of strontium-90 were collected in one 4-liter product fraction, and all of the strontium was collected in eight liters. The results are given in Table II.

D. RESIN RADIOLYSIS

At no time during the seven runs was the resin damaged sufficiently to cause an adverse effect on the run. The resin was discarded after each run and was not excessively "hot". No quantitative determination of residual resin activity was made. Following the completion of run 7, the resin in columns 1 and 4 was loaded to breakthrough with copper from a copper sulfate solution. The capacity of the resin was found to be not altered significantly, indicating little damage to cross linkage. The resin in column 1 had received a dosage of approximately 2×10^8 R.

E. PROCESS APPLICATIONS

During the absorption cycle of the first strontium production run, the capacity of the resin in the absorption column was limited to approximately 3 grams of strontium per liter of resin. The low strontium capacity was a direct result of the high mole ratio of monovalent ions (sodium and ammonium) to strontium ions. The ratio in the prepared feed solution was 500. In both run 1 and run 2 nitric acid was added to the low acid Purex crude in unsuccessful attempts to dissolve the precipitate. This acid caused an increase in the final ammonium ion concentration when neutralized by ammonium hydroxide during the feed pH adjustment step.

Laboratory runs using synthetic feed solutions indicate the existence of a direct relationship between feed dilution and strontium absorption on the resin. Without dilution, feed containing 0.4 grams Sr per liter and an ammonium to strontium mole ratio of 167 gave a loading of 12 grams Sr per liter of resin. In a second experiment, a feed containing 0.4 grams Sr per liter and an ammonium to strontium ratio of 364 gave a loading of 2.8 grams Sr per liter of resin. A twofold dilution of the feed used in the second experiment yielded a loading of 6.4 grams Sr per liter of resin and a fourfold dilution gave a loading of 11.7 grams Sr per liter of resin. The results of the four runs are summarized in Table III.

These results indicate that a reduction of the monovalent ion concentration in the feed, combined with a dilution of the feed during the final feed preparation step would yield a substantial increase in the resin absorption capacity of future production runs. Since the resin capacity for strontium during the feed cycle is the limiting factor on the amount of pure strontium produced per run, the strontium production per run would be substantially increased.

TABLE III

THE EFFECT OF FEED DILUTION ON THE STRONTIUM
ABSORPTION CAPACITY OF DOWEX 50W, X-12 RESIN

		<u>Run 1</u>	<u>Run 2</u>	<u>Run 3</u>	<u>Run 4</u>
Sr	(g/l)	0.4	0.4	0.2	0.1
Ca	(g/l)	0.15	0.15	0.077	0.038
Pb	(g/l)	1.62	1.62	0.81	0.40
Fe	(g/l)	8.34	8.34	4.2	2.1
NH ₄	(g/l)	13.7	30	15	7.5
HEDTA	(g/l)	50	50	25	12.5
Dilution Factor		1	1	2	4
Feed pH		4.0	4.0		
Resin Capacity at 10% Breakthrough (grams Sr/l of resin)		12.0	2.8	6.4	11.7

In future Purex production runs, if the carbonate concentration step is used, the strontium crude feed clarified by centrifugation, and advantage taken of the feed dilution technique, it would be possible to load 12 grams of strontium per liter of resin. This would allow the recovery and purification of 58,000 curies of strontium-90 in a single loading cycle run in the High Level Cell ion exchange equipment. If a double feed cycle technique like that in production run 7 were used, the production of pure strontium would be increased to a little over 100,000 curies per run.

In a new production facility designed for this process, a three column system would be satisfactory. One 8-inch diameter absorption column containing a 10-foot resin bed would contain 98.6 liters of resin and thus would have a one cycle absorption capacity of 95,000 curies of strontium-90. Two 4-inch diameter elution columns of the same length would be adequate to yield a product purity comparable to that of production run 7.

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V. BIBLIOGRAPHY

1. Keenan, James J., Strontium-90 Power Project, Final Summary Report, MND-Sr-1676, March, 1960.
2. Bray, L. A., and Van Tuyl, H. H., Fission Product Strontium Recovery from Purex Chemical Processing Plant Waste Solutions, HW-69534, May 15, 1961 (Unclassified).
3. Wheelwright, E.J., Bray, L.A., Roberts, F.P., and Moore, R.L., Report of Invention - An Integrated Process for the Recovery and Purification of Multi-Kilocurie Quantities of Strontium from a Purex Crude Strontium Concentrate, HW-70336, July 11, 1961.
4. Spedding, F.H., Powell, J.E., Wheelwright, E.J., J. Am. Chem. Soc., 76 2557 (1954).
5. Spedding, F.H. and Powell, J.E., Transactions of the Metallurgical Society of AIME, 215 No. 3, 457 (1959).
6. Powell, J.E., and Spedding, F.H., Basic Principles Involved in the Macro-Separation of Adjacent Rare Earths from Each Other by Means of Ion Exchange, ICS-617, October 26, 1955 (Unclassified).
7. Schwarzenback, G. and Ackermann, H., Helv. Chim. Acta, 30, 1798 (1947).
8. Upson, U.L. and Roberts, F.P., An In-Cell Gamma Analyzer, HW-70833, September 1, 1961 (Unclassified).
9. Smith, F.M., A Laboratory Study to Define Conditions for Loading Strontium-90 on Decalso for HAPO-1A Cask Shipment, HW-71574, November 30, 1961 (Unclassified).