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TITLE

B-PLANT FISSION PRODUCT FLOWSHEETS
PART I

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B-PLANT FISSION PRODUCT FLOWSHEETS - PART I

By

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Advance Process Development
Research and Engineering Operation
Chemical Processing Department

January 12, 1961

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

Work performed under Contract #AT(45-1)1350
between the Atomic Energy Commission and the General Electric Company.

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B-PLANT FISSION PRODUCT FLOWSHEETS-PART II. INTRODUCTION

B-Plant is currently being evaluated for use as an integrated fission product plant operating in conjunction with the Purex Plant and a waste calcination system (Ref. 1). If the forecasted demands for fission products should increase to rates exceeding present capabilities and if private enterprise continues to remain outside the recovery field, present budget plans are to develop the use of B-Plant in three phases. In Phase I, the B-Plant canyon would be activated and provisions made for preparing and storing fission product concentrates (Ref. 2). In Phase II, additional equipment would be installed to provide a single-line demonstration system for purifying and packaging fission products. In Phase III, the plant would be converted to a double-line production system for recovering, segregating and storing, purifying and packaging fission products.

Scope design of the Phase I activities planned for B-Plant is underway (Ref. 2). As part of prescoping studies, the integrated process and service cell needs in B-Plant have been estimated for all phases of the fission product program and for the waste calcination program. Equipment and architectural revisions required to update the canyon for safe operation at the standards employed in the present separations plants have been reviewed. In addition, areas of possible interference between operation and construction activities have been evaluated to permit efficient phasing of project actions (Ref. 3).

Alternative program concepts, such as extending use of the Hot Semiworks on an interim basis, are being evaluated separately. Such concepts would have the potential advantage of delaying B-Plant project action until construction needs can be consolidated and optimum technology defined. The choice of alternative program directions and specific plans for program phasing and timing will represent a balance among: 1) anticipated level and timing of market demands, 2) relative capabilities and reliability of alternative processing systems, 3) hazards control needs and possible construction interferences, and 4) availability of construction funds.

The purpose of this document is to present the technical bases for B-Plant project scoping studies, including:

1. Design flowsheets for the preparation and storage of fission product concentrates in the scope design of Phase I activities, and
2. Conceptual flowsheets for the purification of stored concentrates in the engineering studies of Phase II activities.

Design flowsheets for Phase II purification operations will be issued in a second document (Part II) when scope design is initiated.

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II. SUMMARY

Design flowsheets are presented in Figures 1 through 6 for the recovery of strontium, cerium and promethium and the separation of strontium and promethium for storage. The flowsheets are based largely on the precipitation processes currently employed in the Purex Plant for strontium recovery, with additional process steps defined to permit segregation of cerium and promethium. The Purex FTW (Formaldehyde Treated Waste) raffinate stream is processed through a lead-carrier sulfate precipitation step and a carbonate-hydroxide metathesis step to recover a concentrate mix of strontium, cerium and rare earths. Strontium is separated from the cerium and rare earths by an oxalate precipitation to produce a supernatant solution suitable for boil-down and storage. Cerium is separated from the remaining rare earths by a peroxyacetate precipitation for either shipment as a product or disposal as a waste, leaving a promethium-bearing supernatant solution suitable for boil-down and storage.

An integrated series of process steps is presented as a basis for design to provide for adequate flexibility in equipment routings and capabilities for multi-product operations. Principal emphasis during Phase I operation, however, will probably be on the recovery of strontium-90 concentrates because of greater anticipated needs. The process flowsheets are intended to permit throughput capabilities approaching the output from the Purex plant for : (1) strontium, assuming integrated operation of the Purex head-end precipitation-centrifugation equipment and one precipitation-centrifugation section in B-Plant, and also for (2) strontium plus promethium, assuming integrated operation of Purex equipment and two precipitation-centrifugation sections in B-Plant. Similarly, cooling needs for storage of fission products concentrates are defined, assuming storage of both strontium and promethium at rates approaching the output from Purex.

Conceptual flowsheets are presented in Figures 7 through 13 for alternative cerium separation procedures and for purifying strontium prior to packaging and loadout operations. The strontium purification flowsheets are based on the chromatographic ion-exchange process currently being employed in the Hanford Laboratories' High-Level Cells as part of the strontium-90 program. The ion-exchange techniques supplemented by precipitation processes was chosen for engineering studies vice solvent extraction because of its potentially direct application to the separation of promethium and its relatively advanced state of development. Final choice of purification methods for B-Plant scope design, however, should await the results of forthcoming demonstration runs in the strontium-90 program at the High-Level Radiochemistry Facility and the Hot Semiworks.

III. PROCESS AND DESIGN BASES

A principal goal of the B-Plant process and design studies is to provide long-range flexibility in B Plant for the various possible products and possible processing functions, balancing anticipated needs with costs. Potential products of interest include strontium-90, cesium-137, cerium-144, promethium-147, and technetium-99. Possible product forms desired by the customer include segregated concentrates, intermediate pure products, and

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packaged pure products. Processing functions to achieve such products include recovery, segregation and storage, and purification and packaging. Thus, providing low cost capabilities to meet these various needs in a timely fashion at potential demand rates involves integrated use of the Purex and B-Plant equipment with flexible process flowsheets based on precipitation techniques to the maximum extent practicable.

Current plans are to perform the initial recovery operations in Purex Plant head-end centrifugation equipment, transfer the concentrate of mixed fission products to B-Plant via underground lines and the 244-CR Vault tanks, and then process the concentrate in existing B-Plant equipment for storage and later purification in the B-Plant canyon. Waste solutions will be routed to the existing non-boiling waste tanks associated with B-Plant or to the Purex self-boiling waste tanks, depending on availability of cross-country lines and the timing of waste calcination plans.

Phase I activities in B-Plant are intended to permit the preparation and storage of megacurie amounts of strontium and promethium concentrates. A 12- to 18-month aging period is required for strontium and promethium fission products to provide products having acceptably low concentrations of undesirably short-lived isotopes. Strontium-90, for example, with its 28-year half-life is accompanied by large amounts of strontium-89 with a 51-day half-life. Aging 18 months after reactor discharge, however, reduces the activity level of the strontium-89 to less than 10 percent of the strontium-90. Similarly, the pure beta emitter, promethium-147, is contaminated by the gamma emitter, promethium-148, which limits the utility of the material. Appropriately aged cesium-137 is also in demand (for teletherapy sources) but at low enough levels that the existing inventory in the Purex waste tank supernatants should suffice for a number of years. Cerium and technetium (which is also available in the waste tank supernatants) do not materially benefit from aging. The storage system in B-Plant is thus planned to be capable of maintaining a 15-month inventory of strontium and promethium concentrates at throughput rates corresponding to the output from the Purex process.

Integrated multi-product flowsheets are presented as the design basis for recovering and preparing strontium and promethium concentrates and separating cerium. It is anticipated, however, that principal emphasis during Phase I operation will be on the recovery and storage of strontium-90 due to more immediate market needs. The multi-product flowsheet is intended to provide for adequate flexibility, equipment routings and capabilities for long-range operation, and to permit interim recovery of promethium or cerium. Throughput capabilities for strontium alone should approach the output from the Purex Plant, assuming integrated operation of the Purex head-end equipment and one precipitation-centrifugation equipment section in B-Plant. Campaign operations of such a system will permit recovery and storage of limited amounts of promethium to develop markets or to meet specific needs on a custom basis. Throughput capabilities for strontium plus promethium should also approach the output from the Purex Plant, assuming integrated operation of the Purex equipment and two precipitation-centrifugation

sections in B-Plant. Throughput rates for cerium will correspond to those for promethium although loadout facilities are not currently planned for Phase I projects.

Phase II activities will provide a single-line demonstration system for purifying and packaging fission products. Present plans are to install an ion exchange system for purifying strontium (or limited amounts of promethium) and a manipulator cell for bulk packaging of purified solids. An integrated precipitation - ion exchange flowsheet is presented for the purification of strontium at rates approaching the output from the Purex Plant. The conceptual flowsheets serve to indicate the process and service needs for Phase II activities. A chromatographic - ion exchange process was chosen for preliminary engineering studies because of its potentially direct application to the separation of promethium and its relatively advanced state of development. Solvent extraction, however, may well prove to have greater capability and flexibility for an integrated fission product system. The strontium-90 program in the Hanford Laboratories' High-Level Cells and Hot Semiworks will provide the opportunity for thoroughly evaluating the relative merits of the two processing techniques.

IV. DESIGN FLOWSHEETS AND EQUIPMENT NEEDS

The objective of the design flowsheets shown in Figures 1 through 6 is to recover 70 to 80 percent of the strontium and rare earths from the Purex Plant raffinate in a concentrated form suitable for storage and aging. The process involves the following six steps:

1. Lead-carrier sulfate precipitation and carbonate metathesis, Figure 1.
2. Lead removal by iron-carrier precipitation, Figure 2.
3. Strontium-cerium separation by oxalate precipitation, Figure 3.
4. Cerium - rare earth separation by peroxyacetate precipitation, Figure 4.
5. Preparation of a strontium - rare earth concentrate by evaporation, Figure 5.
6. Waste disposal, Figure 6.

The first step (lead-carrier sulfate precipitation and carbonate metathesis) separates the strontium, cerium and rare earths from most of the other components of the Purex raffinate stream. Steps 2 and 3 prepare feed suitable for peroxyacetate separation of cerium. Step 2 removes the lead, and Step 3 separates iron and strontium from the cerium - rare earth fraction. Step 4 (cerium - rare earth separation by peroxyacetate precipitation) is not feasible unless the feed is relatively free of lead, iron and other metallic

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impurities. If strontium is the only desired product, the recovery scheme is simplified through elimination of Step 4 and possibly Step 2. Step 5 is merely a boil-down operation in which the rare earths and strontium are shown as again being combined. There are obvious advantages in subsequent processing steps for storing these two fractions separately, and the actual storage arrangement will depend on relative product demands and purification needs. Figure 6 summarizes the volumes and composition of the generated fission product wastes.

Lead-Carrier Sulfate Precipitation Process

The lead-carrier sulfate precipitation process^(5,6) has been demonstrated in full-scale Purex Fission Product Test Plant runs. The bulk of the cerium and rare earths are precipitated from Purex raffinate as a sodium - rare earth double sulfate salt by adjusting the pH to approximately 2. Lead is added to carry strontium with the precipitate, and tartaric acid is added to complex iron and prevent its precipitation. After centrifugation, the cake is washed with dilute sodium sulfate and treated with a mixture of sodium carbonate and sodium hydroxide. The metathesis process removes the sulfate by converting the rare earths and strontium sulfates to the highly insoluble carbonates and solubilizing the bulk of the lead as the plumbite. The remaining crude carbonate product consisting of rare earths, cerium, and strontium is dissolved in dilute nitric acid for further treatment.

The flowsheet is not sensitive to normal fluctuations in Purex raffinate composition, and the pH adjustment is not critical. However, too low a pH reduces recovery efficiencies and too high a pH will precipitate voluminous quantities of iron. Excessive batch size must also be avoided since the intense decay heat of cerium can cause partial calcination of the cake, making the cake very difficult to dissolve or remove from the centrifuge.

Lead Removal Process

The next process step is the removal of lead to prevent its interference in the peroxyacetate process planned for separating cerium from the rare earths. The lead removal process consists of precipitating the rare earths, strontium, and iron with a mixture of sodium carbonate and sodium hydroxide. The rare earths and iron act as carriers for the strontium, and the caustic solubilizes the bulk of the lead not removed in the previous carbonate-hydroxide treatment of the sulfate cake. The carbonate product is centrifuged, washed, and dissolved in dilute nitric acid. A slight modification of this process has been successfully tested in the Purex Fission Product Test Plant. It was used to

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concentrate and separate strontium from sodium and other monovalent ions. The B-Plant flowsheet differs only in that higher concentrations of sodium hydroxide are used to remove lead. The process is not expected to be sensitive to normal variations in feed composition with the exception that excessive quantities of iron may require more than one centrifuge cleanout per run.

Strontium - Cerium Separation Process

Iron interferes with the peroxyacetate process planned for separating cerium from the rare earths. The most assured process currently known for avoiding iron interference is to separate the iron and strontium by precipitating the cerium and rare earths with oxalic acid in a slightly acidic solution. The precipitate is separated by centrifugation, washed, slurried from the centrifuge with water, and made slightly acidic with nitric acid. The strontium supernatant solution is concentrated and placed in storage.

The oxalate precipitation process is used in the Purex Fission Product Test Plant for interim strontium recovery. The parameters affecting separation efficiencies have not as yet been fully defined; some Purex runs have had satisfactory yields, while other runs have had low yields. Sulfate is believed to be a major interference. The lead removal step should minimize the amount of sulfate reaching the strontium-cerium separation step.

Cerium - Rare Earth Separation

The peroxyacetate process is used for separating cerium from the rare earths (ref. 7). In this process hydrogen peroxide is used to oxidize cerium in an acetate-buffered solution. Cerium is then precipitated as a peroxyacetate. The solution is centrifuged and the supernatant solution containing the rare earths is concentrated and placed in storage. The cerium peroxyacetate precipitate is dissolved in warm nitric acid. The cerium at this point is relatively pure and is suitable for cask loading or packaging should the market demand develop.

This process has been tested in the plant and shows promise. The process is sensitive to pH adjustment and metallic impurities. If the metallic impurities can be kept below 0.008 M, the buffer solution should control the pH in the desired range. The process does not volatilize ruthenium which makes it more attractive than other processes employing strong oxidants in highly acidic solutions.

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Alternative Flowsheets

Alternative conceptual flowsheets are presented in Figures 7, 8, and 9. They are based on old Bismuth Phosphate data that indicate good separation of cerium from rare earths and strontium is achieved in the by-product strike (Ref. 8). The flowsheets are not recommended for scope design but represent probable future trends in flowsheet development where only one step would be required in the Purex Plant and one step in B-Plant to prepare a strontium - rare earth product suitable for concentration and storage. Flexibility should be provided in plant equipment layouts to permit taking advantage of such flowsheets at a future date.

The path of neptunium in the fission product flowsheets is currently being investigated. If it is found that significant amounts of neptunium are present in or could be routed to the B-Plant feed, precipitation flowsheets based on the BiPO_4 process could be employed to recover a crude neptunium product for recycle to Purex or Redox (Ref. 4). With another additional precipitation step, plutonium could also be recovered and recycled to the primary plants. Some reduction in strontium and rare earth capabilities would result if additional precipitation steps were made to recover neptunium and/or plutonium. Also, equipment to load out a neptunium and/or a plutonium product would have to be provided on a separate project.

Equipment Needs

The flowsheets are based on batch precipitation processes to permit maximum use of existing equipment. Based on operating experience with Purex Plant centrifuges, the existing B-Plant centrifuges can be adapted for fission product processing since they are of similar design and equipped with temperature elements and radiation recorders for monitoring the accumulation of fission products. Cooling coils and temperature recorders are required in all process vessels for dissipating and controlling the decay heat from the fission products. The expected heat generation rates of the various process solutions are included in the flowsheets. The estimated heat removal requirements for the storage vessels are listed in Table 1. The estimates are based on a strontium - promethium concentrate entering storage with a "cooling" time of approximately 100 days. This estimate is conservative since lag storage in the 244-CR Vault could increase the age to approximately 130 days.

The existing B-Plant samplers are not adequate for the radiation levels anticipated. Process vessels should have samplers to allow taking samples of a size consistent with Purex Analytical Laboratory requirements. Many of the precipitation steps require pH adjustment, and provision for taking rapid pH measurements should also be provided.

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No special equipment will be needed initially at B Plant for disposal of wastes. If the wastes are transferred to the Purex Tank Farm, the self-boiling solutions will have adequate capacity to concentrate B-Plant fission product wastes in addition to normal amounts of Purex Plant waste as indicated in the flowsheets. If the wastes are transferred to the B, BX, BY tank farms, the limited space available in the tanks coupled with waste management plans for segregated handling of carbonate wastes dictate accelerating the in-tank concentration program or installing a waste concentrator in B-Plant Phase II operations.

V. CONCEPTUAL FLOWSHEETS AND EQUIPMENT NEEDS

Following 12 to 15 months of storage for decay of the short-lived fission products, the strontium - rare earth concentrate will receive additional processing to produce a highly-purified packaged product. Conceptual flowsheets for a single-line strontium plant are presented in Figures 10 through 13. The conceptual scheme consists of the following steps:

1. Iron removal by sulfate precipitation, Figure 10.
2. Strontium and promethium separation by oxalate precipitation, Figure 11.
3. Strontium purification and concentration by ion exchange, Figure 12.
4. Waste Disposal, Figure 13.

The flowsheets are based on a chromatographic ion exchange process for final purification of the strontium (Ref. 9). The ion exchange process requires a feed relative free of inert cations since they compete directly with strontium for resin sites. Inert cations in the feed thus reduce column capacity available for strontium production. A semi-refined feed is a necessity if reasonable sized columns are to have a production capability equal to the Purex Plant.

Two precipitation steps are planned for preparing a semi-refined feed to ion exchange: A sulfate precipitation step to remove iron and sodium, followed by an oxalate strike to remove rare earths and lead. The two process steps are similar to those described earlier except the fission product concentrations have been increased by evaporation. If the strontium and rare earths are stored separately, only the sulfate precipitation step is required at this point.

The ion exchange process consists of loading the strontium onto one or more columns containing cation resin in the ammonium form. After loading, the column is eluted with buffered ethylene-diamine-tetra acetic acid solution (EDTA) down two or more columns in the copper-hydrogen form.

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(Recent data from hot cell runs indicate the columns should be in the calcium - hydrogen form to prevent copper contamination of the product.) The elution process separates and concentrates the various cations into individual bands. The strontium band is segregated during elution from the last column and reloaded on a column with resin in the copper-hydrogen form. The column is then washed and eluted with dilute nitric acid. The resulting product is very pure, has a concentration of around 40 grams per liter, and is suitable feed for the packaging cell.

The ion exchange process can also be used to purify promethium. However, the purification of promethium from chemically similar rare earths requires more separation capacity than strontium purification. By recycling and reloading promethium fractions on the same columns several times, limited amounts of promethium could be purified. Promethium can be separated from the EDTA complex by precipitation with oxalic acid, or reloading on an ion exchange column in the copper-hydrogen form in a manner similar to the strontium concentration step described previously.

Two major limitations of the chromatographic elution ion exchange process are the sharp reduction in capability caused by the presence of contaminants in the feed and the short life of the resin (approximately 60 hours) when fully loaded with radioactive fission products. Both of these limitations are indirectly associated with the low solubility of EDTA since the rate of elution determines both the capacity and the duration of exposure to the fission products. A promising substitute for EDTA is alpha-hydroxy-isobutyrate. It has a solubility several-fold greater than EDTA. This means that several-fold more moles of elutriant per unit time can be passed through the column at comparable flow rates. The net result is shorter time cycles or greater processing capability and less exposure to radiation. Although alpha-hydroxy-isobutyrate costs more than EDTA, higher operating costs may be justified through higher production capabilities without increased capital expenditure for new equipment.

Equipment Needs

The conceptual flowsheets for the purification of strontium employ existing B-Plant centrifugation equipment to prepare a feed suitable for ion exchange processing. The attention to heat dissipation, temperature monitoring, and radiation monitoring required in the concentrate preparation and storage sections of the plant will also be required in the purification sections.

The ion exchange system will require a new process equipment system consisting of four columns 5 to 10 feet in height and 8 to 12 inches in diameter. Each column will require flow, temperature and differential pressure instrumentation, a resin charge-discharge system, effluent samples, and a vent system for de-gassing the column. Radiation recorders to monitor the effluent lines will also be required.

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The ion exchange resin will be converted to the ammonium form before each loading cycle; in addition, varying amounts of ammonia may be required for pH adjustment of the ion exchange feed. Future flow-sheets may use ammonium for special precipitations or pH adjustment. Therefore, the B-Plant vent system should be scoped to permit a possible installation of an ammonia scrubber when the purification equipment is installed. The ammonia scrubber should be located upstream of the planned vessel vent heater and filter.

If promethium market demand increases sharply, additional ion exchange columns will be required to provide the necessary capability. Approximately three times as many ion exchange columns are needed for promethium as for strontium when the processing capability matches that of the Purex Plant. Successful demonstration of alpha-hydroxyisobutyrate as a substitute for EDTA may improve promethium capability in the strontium columns.

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TABLE I

ESTIMATES OF THE MAXIMUM HEAT GENERATION RATES
IN THE FISSION PRODUCT CONCENTRATE STORAGE VESSELS

<u>Vessel</u> <u>Use</u> <u>Sequence</u>	<u>Equipment Piece</u> <u>Number</u>	<u>Heat Generation Rate</u> <u>BTU/hr.</u>	<u>Approximate Mean Age of</u> <u>Concentrate in Days</u>
1	3-TK-11-1	1,500,000	140
2	3-TK-11-2	630,000	230
3	4-TK-11-3	360,000	310
4	4-TK-11-4	250,000	400
5	4-TK-8	190,000	480
6	4-TK-7	160,000	570

Assumptions:

1. The fission product concentrate will be separated from Purex raffinate while processing uranium irradiated to 600 MWD/T and 7.5 MW/T. The concentrate entering storage will represent uranium "cooled" 100 days.
2. The fission product concentrate will contain the following percentages of the fission products present in the Purex raffinate:

SR	-	70 - 80%
TRE	-	70 - 80%
Ce	-	10%
Y	-	10 - 20%
Ru	-	----
Zr	-	6 - 1%

3. The tanks will be operated in series.
4. Maximum accumulation in a single tank will be 4500 gallons of concentrate.
5. Maximum rate of concentrate accumulation will be 53 gal./day.

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TABLE IINOTES FOR FIGURES 1 THROUGH 13

1. Fission product concentrations and heat generation rates are based on uranium irradiated to 600 MWD/T at 7.5 MW/T and "cooled" 100 days.
2. Unless otherwise indicated, the amount of material processed per run is one batch. One batch represents Purex raffinate from 37 tons of uranium production.
3. All fission product percentages are based on Purex raffinate as being 100 percent.
4. All volumes are given in gallons.
5. All temperatures are ambient unless otherwise noted.
6. The units for flow in Figure 12 are ml/min. cm^2 ; all other flows are indicated as gallons per minute (gpm) on batch additions or batch transfers.
7. The trivalent rare earths are abbreviated on the flowsheets with the letters "TRE".
8. All process water is "soft" water.

Notes: See Table II

FIGURE 1
Purex Design Flowsheet Fission Product Recovery

Notes: See Table II

FIGURE 2
B-Plant Design Flowsheet Lead Removal

FIGURE 3

B-Plant Design Flowsheet Strontium and Cerium Separation

Notes: See Table II

FIGURE 4

B-Plant Design Flowsheet Cerium Separation

Notes: See Table II

FIGURE 5

B-Plant Design Flowsheet Strontium and Rare Earth Solution Concentration

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Notes: See Table II

FIGURE 6
B-Plant Design Flowsheet Waste Disposal

Notes: See Table II

FIGURE 7

B-Plant Conceptual Flowsheet Alternate Cerium Separation

Notes: See Table II

FIGURE 8

B-Plant Conceptual Flowsheet Alternate Strontium-Rare Earth Solution Concentration

Notes: See Table II

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FIGURE 9

B-Plant Conceptual Flowsheet Alternate Waste Disposal

Notes: See Table II

FIGURE 10
B-Plant Conceptual Flowsheet Iron Removal

Notes: See Table II

FIGURE 11
B-Plant Conceptual Flowsheet Strontium and Promethium Separation

FIGURE 12

B-Plant Conceptual Flowsheet Strontium Purification and Concentration

Notes: See Table II

FIGURE 13

B-Plant Conceptual Flowsheet Waste Disposal - Storage and Purification

DATE

FILMED

9/16/94

END