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FISSION PRODUCT PRODUCTION PLANT
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FISSION PRODUCT PRODUCTION PLANT

ENGINEERING STUDY BASIS - CHEMICAL FLOWSHEET

By

R. L. Moore, G. L. Richardson
W. H. Swift, H. H. Van Tuyl
HANFORD LABORATORIES

March 13, 1963

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FISSION PRODUCT PRODUCTION PLANT

ENGINEERING STUDY BASIS - CHEMICAL FLOWSHEET

INTRODUCTION

At the request of the U. S. Atomic Energy Commission, the General Electric Company has undertaken an engineering study preliminary to the design and construction of a fission product heat source production plant at Hanford. Assumptions basic to the engineering study are as follows:

1. Overall compatibility with Hanford Waste Management processes and schedule.
2. Maximum utilization of existing facilities and technology.
3. Production capacity to match Hanford Waste Management Operation.
4. Waste Management products to be used as feeds.
5. Design to allow for operation by private industry.
6. Encapsulated products to include:
 - A. Sr-90 as strontium titanate or oxide
 - B. Cs-137 as cesium glass
 - C. Ce-144 as cerium oxide
 - D. Pm-147 as promethium oxide
7. Encapsulated products to be produced in various sizes and shapes.

This document presents proposed feed materials, product specifications, and the chemical flowsheet basis for the engineering study. A preliminary waste extraction chemical flow-sheet is included to illustrate the integration of the proposed production plant with the Hanford Waste Management Program.

SUMMARY

Engineering study basis chemical flowsheets are presented for the purification, conversion, and packaging of the fission product radionuclides, strontium-90, cesium-137, cerium-144, and promethium-147. Also presented is a preliminary chemical flowsheet illustrating integration of the fission product plant with the Hanford Waste Management Program.

Feed materials to the fission product plant include the quantities of fission products as shown on page 2:

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FISSION PRODUCT PRODUCTION PLANT
FEED MATERIALS - 1966 DATA

<u>Source</u>	<u>Megacuries/Year</u>			
	<u>Sr-90</u>	<u>Cs-137</u>	<u>Ce-144</u>	<u>Pm-147</u>
Current Purex (1966)	13.7	14.8	335	56

Although the annual production capacity of the fission product plant has an unclassified stated basis of 10, 10, 100, and 30 megacuries of strontium, cesium, cerium, and promethium, respectively, capacity is planned to match the Hanford Waste Management Program waste processing rate including work off of stored alkaline wastes. Thus, process equipment will be sized for net product rates of 15, 25, 100, and 50 megacuries per year of strontium, cesium, cerium, and promethium respectively.

Tentative product specifications for chemical and radiochemical purity, product form, and encapsulated source sizes are given. Properties (theoretical) of the encapsulated source materials are shown below:

PROPERTIES OF ISOTOPES

	<u>Sr-90</u>	<u>Cs-137</u>	<u>Ce-144</u>	<u>Pm-147</u>	
Types of Decay	Beta	Beta-Gamma	Beta-Gamma	Beta	
Half-life, Years	28	30	0.78	2.5	
Purity, Weight Percent	50	35	18	95	
Chemical Form	SrTiO ₃	SrO	Glass	CeO ₂	Pm ₂ O ₃
Isotope in Compound, Weight Percent	24	43	16	15	82
Density of Compound, g/cc	5.1	4.7	3.2	6.4	6.6
Power Density of Compound, Watts/cc	1.16	1.92	0.215	25	1.87

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The chemical flowsheet for the fission product plant includes the following general areas:

1. Carbonate precipitation separation of strontium and cesium from the waste extraction process LBP stream.
2. Solvent extraction purification of strontium and cesium by di-2-ethylhexylphosphoric acid (D2EHPA) and 4-sec-butyl-2 (α -methylbenzyl) phenol (BAMBP) respectively.
3. Pyrochemical separation of cerium and promethium from the waste extraction process LCP stream, or alternatively, the higher capital and operating cost (but best proven technology) D2EHPA solvent extraction process. It is recommended that the engineering study include evaluation of the relative incentive for developing and applying the pyrochemical process.
4. Promethium purification is by chromatographic ion exchange.
5. Conversion and encapsulation of the ceramic heat sources (SrTiO_3 , SrO , CeO_2 , and Pm_2O_3) is achieved by the high-rate densification (Dynapak) process, achieving compact, flexible, and generic (multiproduct) capability.
6. Conversion of cesium to borate glass is achieved by acid oxalate precipitation and blending with glass formers prior to melting and casting. High-rate-densification techniques are also potentially applicable.
7. A separate conversion step for cerium is not required if purification by the pyrochemical process is employed. If solvent extraction cerium purification is used, conversion via oxalate precipitation and calcination is necessary.
8. Promethium conversion is achieved by oxalate precipitation and calcination.

Product Specifications

The specifications of the products from the fission product plant are primarily dependent on the requirements of the final user. Strontium, cerium, and promethium are to be used as heat sources for space, terrestrial, and marine applications, while cesium may also be used in irradiation processing such as inhibition of sprouting and rot in potatoes. The chemical form and package shape may be different for different users. For example, low solubility may be desired for some applications, while high power density may be more important in other applications. However, to simplify plant design and minimize unit costs it is assumed that a single chemical form will be sufficient for each of the four products, and that all packages will be right cylinders enclosed in stainless steel (or alloys with comparable fabrication metallurgy) with a choice of several cylinder diameters. The chemical forms assumed for study purposes are strontium titanate or oxide, cesium borate glass, cerium dioxide, and promethium sesquioxide. By consideration of these product forms, sufficient flexibility should be attained in the design to permit production of alternative materials at a later date and as the need arises.

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Maximum power density with a particular chemical form of product requires that impurities be kept to a minimum. In order to meet the expected needs of the most exacting probable customers (SNAP program), a maximum of five percent inert impurities is established. In addition to the impurities allowed in this specification, stable isotopes of the same element will of course be present along with the desired isotopes for all elements of interest except promethium. Since separation of isotopes of the same element is not economically possible, the product will be considered to consist of the desired isotopes along with all other isotopes of the same element which are present in Purex wastes. Although high purity is required for many applications, others may be satisfied with lower quality material. In most cases a product of lower purity may be obtained by shortening or eliminating certain purification steps. However, capability will be provided for producing all of the products at specification purity. An additional restriction because of the requirement for maximum power density is that the cerium product must be processed with minimum cooling time. As the Ce-144 decays, the ratio of active to inert cerium decreases, thus decreasing the power density obtainable.

Another requirement is that the power density vary as little as possible with time. This requires that the heat generation rate from short-lived nuclides be a small fraction of the total. This is accomplished only by age in the case of isotopes of the same element. For strontium a minimum age of about 15 months is required for adequate decay of the Sr-89. Nuclides of other elements should contribute less than two percent of the total power at all times during the useful life of the source (usually taken as one half-life). An exception to this latter requirement is the permissible admixture of cesium and strontium in heat sources since the half-lives, heat generation rates, and shielding requirements for large sources are about the same. This is limited by the compatibility of the two elements in a single source compound. In spite of the permissibility of this admixture, it is not expected to occur since the separation of strontium from cesium is not difficult.

In addition to the above requirements, the radiochemical purity must be adequate to minimize shielding requirements. In general, the dose rate from a source should not be increased by more than ten percent because of impurity radionuclides. As the source age and size vary, the degree of decontamination required will also vary slightly. The maximum shielding requirement is assumed to be the shielding required to reduce the radiation from a 1000 watt source to a dose rate of 10 mr/hr at the surface of the shield. The minimum shielding requirement is assumed to be the shielding required to reduce the radiation from a 10 watt source to a dose rate of 10 mr/hr at one meter from the surface of the shield. For impurities like cerium which have a high energy gamma, the maximum impurity radiation from the shielded source relative to the radiation from the source nuclide occurs with the maximum shielding case. For certain other nuclides, such as zirconium in strontium, the maximum

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impurity radiation from the shielded source relative to the radiation from the source nuclide occurs with the minimum shielding case. Thus, a higher zirconium content would be more acceptable in a large strontium source than in a small one. A higher cerium content would be acceptable in a small strontium source than in a large one. If some, but not all, of the product specifications are not met, it may be possible to use the material in sources of certain sizes, but not in other sources. If two or more nuclides have the same limiting decontamination factors required, the amount listed is the maximum amount of a single nuclide in the absence of others. Higher decontamination factors are required if two or more are present at the same time. This information is summarized in Table I. The decontamination factors shown represent the overall requirement including that achieved in the waste extraction process.

In the case of cesium sources, an important consideration is the absence of other gamma radiation so that dose rates may be calculated with greater accuracy. Purification from other elements is not difficult but Cs-134 content is important. Several years decay are required for the cesium to be of adequate quality for teletherapy purposes. Therefore, cesium should be isolated according to age. This is especially important when processing cesium which is recovered from stored wastes which are several years old.

TABLE I

PRODUCT PURIFICATION REQUIREMENTS

<u>Source</u>	<u>Impurity</u>	<u>Required Decontamination Factor</u>	<u>Limitation</u>
Strontium	Yttrium	50	(1)
	Zirconium	2×10^3	(2)
	Ruthenium	3×10^3	(3)
	Cesium	50	(4)
	Cerium	3×10^4	(3)
	Promethium	10	(1)
Cesium (1 Year)	Yttrium	100	(1)
	Strontium	200	(3)
	Zirconium	1×10^3	(3)
	Ruthenium	4×10^3	(3)
	Cerium	4×10^5	(3)
	Promethium	20	(1)
Cerium (6 Months)	Yttrium	20	(1)
	Strontium	20	(1)
	Zirconium	100	(4)
	Ruthenium	15	(1)
	Cesium	20	(4)
	Promethium	3	(4)

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Table I Continued

<u>Source</u>	<u>Impurity</u>	<u>Required Decontamination Factor</u>	<u>Limitation</u>
Promethium (6 Months)	Yttrium	2×10^3	(1)
	Strontium	1×10^3	(1)
	Zirconium	1×10^4	(1)
	Ruthenium	2×10^4	(3)
	Cesium	300	(3)
	Cerium	3×10^5	(3)
Promethium (3 Years)	Yttrium	60	(4)
	Strontium	6×10^5	(3)
	Zirconium	6×10^4	(3)
	Ruthenium	1×10^7	(3)
	Cesium	5×10^6	(2)
	Cerium	7×10^7	(3)

- (1) Excessive heat leveling requirements
 (2) Small source (10 watt) shielding limitation
 (3) Large source (1000 watt) shielding limitation
 (4) Power density (inert impurities)

Table II summarizes source size, power and quantity relationships for fission products from processing 7600 tons uranium per year. Sizes do not include capsule walls.

TABLE II

HEAT SOURCE SIZING

Size of Cylinder for Different Source Strengths

	Diameter, Inches (also equal to height)				
Source ⁽¹⁾	<u>10 Watts</u>	<u>100 Watts</u>	<u>1000 Watts</u>		
Cs	1.4	3.0	6.5		
Sr	0.9	1.9	4.1		
Pm	0.8	1.7	3.6		
Ce	0.3	0.7	1.4		
	<u>Power from Fixed Size Pellets - Watts</u>				
Source ⁽¹⁾	<u>2"</u>	<u>3"</u>	<u>4"</u>		
Cs	29	99	240		
Sr	120	400	940		
Pm	180	590	1,400		
Ce	2,600	8,900	21,000		
	<u>Pellets per Year⁽²⁾</u>				
Source ⁽¹⁾	<u>2"</u>	<u>3"</u>	<u>4"</u>	<u>5"</u>	<u>6"</u>
Cs	2,500	730	310	160	91
Sr	770	230	96	49	28
Pm (1 Year)	110	32	13		
Ce	890	260	110		

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Table II Continued

Notes:

(1)	Cs glass density	3.0	1 Year
	SrTiO ₃	4.1	1 Year
	Pm ₂ O ₃	5.8	Any age
	CeO ₂	5.8	6 Months

(2) Based on fission products from 7,600 tons uranium per year.

WASTE MANAGEMENT PROGRAM INTERACTIONS

The Hanford Waste Management Program is currently outlined for processing (starting in 1966) of (1) current Purex denitrated high activity waste, (2) 241-A and AX tank farm supernates, and (3) 241-A and AX tank farm sludges. The CSREX⁽¹⁾ process will be employed for the extraction of strontium, cesium, and cerium from current Purex wastes, and strontium from treated alkaline waste sludges. Inorganic zeolite ion exchange will be used for removal of cesium from alkaline waste supernates. Alternate backup processes being considered are D2EHPA extraction of strontium, cesium zinc ferrocyanide precipitation or solvent extraction (BAMBP) removal of cesium from alkaline waste supernates and inorganic ion exchange removal of cesium from Purex acidic wastes.

Inasmuch as the CSREX process is regarded as the mainline waste extraction process and since the alternate backup processes produce comparable products, the CSREX waste extraction option is discussed in detail below.

The Purex Plant feed to the waste extraction process will be denitrated LWV derived from a flowsheet employing uranium(IV) as the plutonium reducing agent. A detailed estimation of the composition of the denitrated waste is presented in Table III, together with the composition of LAF (B-Plant solvent extraction feed) prepared from the denitrated waste.

TABLE III

ESTIMATED COMPOSITION OF B-PLANT INPUT STREAMS

Basis: Feed streams are Purex waste derived from processing one ton of uranium, irradiated to 600 MWD/T at 10 MW/T and cooled for 120 days.

	<u>Denitrated LWV</u>		<u>Waste Extraction LAF</u>	
	<u>Gram-Moles</u>	<u>Total Watts</u>	<u>M</u>	<u>Watts/L</u>
H	150	-	(pH 4.5)	-
Na	90	-	1.4	-

(1) Bray, L. A., Solvent Extraction Process for Recovery of Strontium, Cerium, Rare Earths, and Cesium from Radioactive Waste Solutions (CSREX Process), HW-75537, November 5, 1962

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Table III Continued

	Denitrated LW		Waste Extraction LAF	
	Gram-Moles	Total Watts	M	Watts/L
Al	15	-	0.040	-
Ca	0.25	-	0.00066	-
Cr	6	-	0.016	-
Fe	30	-	0.079	-
Ni	3	-	0.0079	-
Rb	0.09	-	0.00024	-
Sr	0.25	147	0.00066	0.38
Y	0.13	210	0.00034	0.55
Zr	0.83	890	0.00020	0.23
Mo	0.62	-	0.0016	-
Tc	0.17	-	0.00045	-
Ru	0.33	143	0.00087	0.38
Cs	0.37	10.8	0.0010	0.029
Ba	0.18	15.3	0.00005	0.040
Ce	0.42	394	0.0011	1.04
Pm	0.052	4.1	0.00014	0.011
Total RE & Y	1.37	-	0.0036	-
U	0.9	-	0.0024	-
SiO ₂	3	-	0.008	-
F ⁻	0.16	-	0.0004	-
SO ₄ ⁼	45	-	0.12	-
PO ₄ ⁼	1.5	-	0.01	-
NO ₃ ⁻	316	-	0.83	-
Tartrate	0	-	0.033	-
Citrate	0	-	0.16	-

	Gram-Moles	Kilocuries	Total Watts	M x 10 ⁵	Curies/L	Watts/L
Sr-Y-90	0.14	1.83	12.2	37	4.8	0.032
Y-91	0.026	58.1	2.0	7	153	0.55
ZrNb-95	0.065	182	890	2	48	0.23
RuRh-106	0.018	6.32	61.7	5	17	0.16
CsBa-137	0.17	1.98	9.5	45	5.2	0.025
CePr-144	0.096	44.1	355	25	116	0.94
Pm-147	0.052	7.42	3.1	14	20	0.008

The energy and material balances presented in the attached flowsheets are based on processing the waste from one ton of uranium, irradiated to 600 MWD/T at 10 MW/T and cooled 120 days. Volumes corresponding to the flows can be read directly on all flowsheets as gal/ton U processed. For capacity estimations, it is assumed that 7,600 tons of uranium are processed per year.

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CSREX PROCESS

The proposed flowsheet for waste extraction as presented in Figure 1, employs the CSREX process recently invented by L. A. Bray and presently under intensive development in the chemical laboratories. The fission products of interest are accumulated in two product fractions in B-Plant. The LBP stream will contain cesium and strontium; the LCP stream will contain cerium and promethium. The approximate compositions of these streams are given in Table IV.

TABLE IV

ESTIMATED COMPOSITIONS OF WASTE EXTRACTION PRODUCT STREAMS

Basis: Feed Streams are Purex waste derived from processing one ton of uranium, irradiated to 600 MWD/T at 10 MW/T.

	Concentrated LBP, 1 Year Cooled			Concentrated LCP, 120 Days Cooled		
	M	Watts/L	Curies/L	M	Watts/L	Curies/L
HNO ₃	0.5	-	-	1.0	-	-
Na	4.0	-	-	-	-	-
Ca	0.13	-	-	0.001	-	-
Sr	0.12	8.6	-	0.0001	0.008	-
Rb	~0.02	-	-	-	-	-
Cs	0.19	5.4	-	0.0002	0.006	-
Y	-	-	-	0.0003	0.55	-
Ce	0.004	2.0	-	0.020	19	-
Pm	-	-	-	0.0021	0.17	-
TRE & Y	-	-	-	0.058	-	-
Sr Y-90	-	6.2	930	-	0.006	1
Y-91	-	-	-	-	0.55	150
CsBa-137	-	4.9	1035	-	0.005	1
CePr-144	-	2.0	260	-	17	2140
Pm-147	-	0.01	30	-	0.17	300

The composition of the strontium-cesium fraction is based on one-year cooling from reactor discharge; the cerium-promethium fraction is cooled 120 days from reactor discharge.

The solvent, a mixture of di-2-ethylhexylphosphoric acid (D2EHPA) and 4-sec-butyl-2(α methylbenzyl) phenol (BAMBP), has a synergistic effect on cesium, permitting co-extraction of cesium together with strontium and the rare earths. The feed to this process contains a combination of complexing agents (tartaric and citric acids are currently specified) which effectively prevent the extraction of LWV components other than cesium, strontium, calcium, rare earths, and to a limited extent, sodium. The extraction of these ions is pH dependent, optimum extraction taking

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place at pH 4 to 4.5. It is possible to selectively scrub most of the extracted sodium from the solvent by use of a buffer solution which maintains a pH between 2.4 and 4. The strontium, cesium, and calcium can be effectively stripped together in the 1B column by lowering the pH from 1.5 to 2. The pH must be lowered still further in the 1C column (to 0.5 or less) to strip the bulk of the rare earths, including cerium and promethium. Yttrium, however, will remain in the solvent and must be removed by solvent treatment.

The following are highlights of some of the special features of the CSREX flowsheet application.

1. The denitrated LWV entering B-Plant will require clarification to remove precipitates of silica, zirconium, barium, and "cruds" originating in Purex processing. A small amount of strontium will be associated with these solids and must be recovered by leaching. The optimum leaching process has not yet been demonstrated but presumably would be a nitric acid rinse in the centrifuge.
2. Due to interactions with the complexing agent in the feed, chromium ion reduces the extraction rate of cerium and other rare earths. The bulk of the development work has been done with citric acid, which is acceptable as long as quantitative rare earth extraction is not necessary. To assure cerium and promethium recovery, it may be necessary to use other, more expensive, complexing agents to permit the degree of rare earth extraction indicated. The mixture of tartaric and citric acids currently specified may be a suitable solution. Another alternative would be to operate the column at about 50C with increased BAMBP in the solvent to compensate for the decrease in the cesium distribution ratio with increased temperature.
3. Being pH dependent, the process requires positive pH control. The LAF complexing agent itself provides some buffer capacity, the remainder being provided by pre-neutralizing the solvent with an amount of caustic approximately equivalent to the amount of sodium that would normally extract from the LAF.
4. The buffer capacity of formic acid is near optimum for pH control during the sodium scrubbing step. Hydroxyacetic acid and citric acid are acceptable substitutes. The acid must be pre-neutralized to at least pH 2.4 to prevent strontium reflux. The caustic added to the scrub will limit the sodium in the product to a minimum of about 0.01M.
5. The pH in the 1B column must be maintained in a fairly narrow range - between 1.3 and 2.3 for optimum strontium and cesium stripping. Higher pH values give incomplete stripping while lower pH values will tend to allow cerium to strip. Dilute nitric acid can be used only if sodium is adequately removed in the 1A column. (The process is essentially an ion exchange

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process - hydrogen ion must transfer to the solvent to strip sodium from the solvent). Although calcium will follow the strontium, some of it can be diverted to the cerium product by operation of the LB column at elevated temperatures.

6. Spent solvent will contain yttrium and small amounts of iron, aluminum, and zirconium which are extracted essentially irreversibly. Suitable washing processes are being developed. These might be as simple as a dilute oxalic acid wash or as complex as a carbonate wash containing a complexing agent and followed by an acid wash to reconstitute the LAX.
7. The solvent extraction products are too dilute for practical storage. It is visualized that the LBP (Sr-Cs product) will be concentrated over 300-fold, limited by the sodium nitrate concentration. The LCP (rare earth product) can be concentrated at least 8-fold. Some of the nitric acid will be boiled off (equilibrium HNO_3 concentration in the LBP and LCP are roughly 1.7 and 5M, respectively). The remainder will be killed by formaldehyde, sugar, or other suitable reagents. The concentrated LBP will be stored for decay of strontium-89 and cesium-134. The LCP should be processed through to final form immediately for maximum specific activity of the cerium-144.

The processing of stored alkaline supernates in B-Plant will produce additional cesium suitable for heat or radiation source application. At the present time use of an inorganic ion exchange is planned for isolation of cesium from this source. This product will require additional purification from sodium prior to conversion to the solid product. Although strontium is scheduled for recovery from the alkaline waste sludge, its quality is not expected to meet customer specifications for source material due to dilution with natural strontium-88.

As outlined above, the CSREX waste extraction flowsheet requires some modification to prepare suitable feeds for the proposed fission product packaging plant. The major addition foreseen at this time would be the use of a more expensive complexing agent in the LAF to guarantee adequate rare earth extraction. For Waste Management purposes, the concentrated LBP stream (strontium and cesium) would be lag stored for about one year prior to packaging on a synthetic inorganic ion exchange medium for long-term storage. The LCP would be stored as a solution for 3-5 years and then blended with low heat generating wastes for in-tank solidification.

BACKUP PROCESSES

Although laboratory and pilot plant studies of the CSREX process have been highly promising, it may prove necessary to use other processes to produce the crude fission product fractions. Of these backup processes, a D2EHFA process for strontium-rare earth extraction should produce strontium and rare earth crudes of similar composition

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to the CSREX LBP and LCP streams (with cesium absent and 70 to 90 percent of the calcium diverted to the LCP). The LA column feed or raffinate would be processed by ion exchange techniques to extract cesium. The cesium after elution with ammonium carbonate and evaporative concentration would be suitable for subsequent purification and packaging with the processes outlined for coupling to the CSREX process. A second cesium isolation option employs BAMBP extraction from alkaline waste supernates. Again, coupling to fission product purification is readily achieved.

FISSION PRODUCT PRODUCTION CHEMICAL FLOWSHEETS

The flowsheets for purification and conversion of the fission product crudes are presented in Figures II through VI.

Strontium-Cesium Separation

For convenience in storage and processing, the strontium will be separated from the cesium in the concentrated LBP by carbonate precipitation. The process (Figure II) is similar to that currently employed in the Strontium Semiworks. The feed to the precipitator is adjusted to pH 1 by the addition of sodium hydroxide. A concentrated solution of sodium bicarbonate is then added and digested at 80 C for about one hour to precipitate strontium carbonate. After digestion, the pH is raised to 10 (ca 0.05M NaOH) with sodium hydroxide to lower strontium solubility losses. The precipitate can conveniently be washed free of cesium and sodium in the precipitator tank by slurring it several times with water, removing the water each time through a filter-end dip leg. The strontium is then dissolved in a minimum volume of nitric acid. Strontium and cesium losses in the precipitation-washing step should be less than 1 percent.

Additional research and development may show that the above precipitation separation step will not be necessary and that the cesium-strontium mixed crude can be processed directly through the strontium solvent extraction process. In this instance, the cesium bearing raffinate would contain organics potentially degradable to undesirable products. This raffinate, after appropriate pH adjustment, would be a suitable feed for the cesium purification process. In either case, the extraction processes are sufficiently selective to prevent cross-contamination.

Strontium Purification

The strontium purification process (Figure III) uses a modified version of the Strontium Semiworks D2EHPA flowsheet. DTPA (diethylenetriamine penta-acetic acid) is added to the feed to complex cerium, and acetic acid is added to supply some buffer capacity. The solvent is also pre-neutralized for buffer control. The chosen solvent concentration limits the maximum total concentration of calcium plus strontium in the LAP to about 0.075M. Buffered formic acid or hydroxyacetic acid is used to scrub sodium from the extract phase.

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The strontium is selectively stripped from the solvent in the 2B column with 2M citric acid. This, at the indicated flow rate and pH, should provide a calcium decontamination factor of at least 10. The concentration of citric acid would be adjusted as necessary to maintain the product pH at about 2 to 2.3.

The citric acid is continuously destroyed by feeding the 2BP product into a boiling concentrator together with a 12 w/o hydrogen peroxide solution. Nitric acid is added periodically to prevent strontium carbonate precipitation. Approximately 5 moles of peroxide and 0.4 moles of nitric acid are used per mole citrate destroyed. During the citrate-kill, the 2BP can be concentrated two or three fold. This process is based on the current Strontium Semiworks procedure, modified for continuous rather than semi-continuous operation. Under semi-continuous operation, an additional day of peroxide addition (after the 2BP flow is stopped) is required to kill residual citrate. It is not known how low the citrate concentration can be maintained in the concentrated product under continuous operating conditions. Thus provision should be made to digest the product for 24 hours.

The spent solvent must be washed to remove calcium and residual cerium. A simple batch wash with 1M HNO_3 may be adequate most of the time, but periodically oxalic acid addition may be required to prevent buildup of unstripped cerium. A simple batch wash with a mixture of 0.5M HNO_3 - 0.5M oxalic acid would serve as an all-purpose wash. The flow of the wash stream would be about 20 percent of the solvent flow.

Pulse columns are the recommended contactors for the 2A-2B columns. These have demonstrated capacities in systems of this type of 600 to 800 gal/(hr)(ft²). Assuming a three month campaign per year per product, the required column diameters would be 2 to 3 inches, compared with 4 inches used in the Strontium Semiworks columns. Tentatively, cartridge heights are set 15 feet in the 2A column, including at least 9 feet for the extraction section, and 10 feet in the 2B. Shorter columns may prove feasible at lower flow rates. These columns can also be used on a campaign basis as the 3A and 3B columns as outlined below for cesium purification. The capacities and efficiencies are comparable in both systems. Provision of two 4-inch columns would provide for processing of current Purex waste extracts and aged alkaline waste cesium and strontium extracts.

Cesium Purification

The cesium purification process is based on BAMBP extraction from an alkaline solution. Without the synergistic effect of D2EHPA, the pH must be about 13 for optimum cesium extraction. The feed as shown in Figure IV contains an oxalate recycle stream from the final cesium oxalate precipitation stream. The cesium and oxalate are sufficiently dilute to avoid solubility problems at this point.

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Pilot plant experience has shown that about 0.05M sodium will extract with the cesium. A boric acid scrub, chosen to provide buffer capacity in the pH 8 to 10 range, should remove over 80 percent of this sodium. Very dilute NaOH has been also used for this purpose at ORNL with satisfactory results.

The cesium will be stripped from the solvent with nearly saturated oxalic acid solution. The solubility of cesium acid oxalate is highly temperature dependent; therefore it may be necessary to heat the column during this step to about 40 or 50 C to prevent cesium precipitation. This method of stripping is preferred over other potential methods because of the simplicity of the subsequent cesium precipitation step. Simply cooling the 3BP to 15 - 20 C will precipitate over 80 percent of the cesium as $\text{CsH}_3(\text{C}_2\text{O}_4)_2 \cdot 5\text{H}_2\text{O}$, leaving 2 to 5 g/l cesium in solution to be recycled to the 3AF. It will be necessary to provide the 3B column with a steam or hot water jacket to provide the necessary temperature control.

Other processes using either the same equipment or similar-sized equipment can be substituted for the BAMBP process if for some reason it should prove impractical. Extraction of cesium with 0.02M dipicrylamine in nitrobenzene is practical, could use the same equipment, and has been well demonstrated in pilot plant pulse column runs(2).

Cerium Purification

Two alternate cerium purification flowsheets are presented in Figure V. The solvent extraction flowsheet is based on technology currently in use at ORNL(3). The main features of this flowsheet are as follows:

1. Potassium permanganate is added to oxidize cerium to the tetravalent state. As side reactions occur, it is necessary to add and maintain an excess of permanganate ion.
2. The initial 10 minute batch contact is made with 1M D2EHPA which is then diluted to 0.5M with fresh diluent for an additional 10 minute contact. This two step process accelerates the rate of extraction and minimizes solvent degradation. If the extraction is performed with 0.5M D2EHPA only, a 30 minute contact is required.
3. Manganese dioxide slurry formed during the oxidation step is dissolved by addition of citric acid to permit phase disengagement and to recover absorbed trivalent rare earths. Contact time following citric acid addition is 10 minutes.
4. After decanting the aqueous promethium-containing fraction, the solvent is scrubbed (10 minute contact time) with 10M HNO_3 to remove yttrium and residual rare earths.

(2) Richardson, G. L., Solvent Extraction of Cesium by Dipicrylamine III Semiworks Demonstration, HW-75447, March 1, 1963.

(3) Butler, T. A., and E. E. Ketchen, Ind. Eng. Chem. 53, 651 (1961)

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5. The solvent is further diluted with fresh diluent (to facilitate cerium stripping) and the cerium-stripping reagent, a mixture of nitric acid and hydrogen peroxide, is added. The hydrogen peroxide serves to reduce unstrippable tetravalent cerium to the strippable trivalent state. The highly degraded solvent is sent to waste and the cerium product to the oxalate precipitation-calcination conversion process.

An alternate method for oxidizing cerium in the solvent extraction process is currently being investigated at Oak Ridge and Hanford. This process involves electrolytically oxidizing a portion of the cerium continuously and cycling organic through the aqueous repeatedly to obtain adequate cerium removal. About five passes with organic appear to be adequate. However, development has not progressed sufficiently to permit a definitive flow-sheet to be presented. If the solvent extraction process is selected, the facility should permit modification to accommodate this alternate.

An alternate cerium separation method has been investigated at Ames⁽⁴⁾. This involves the addition of an inert salt to serve as a flux, conversion of the feed to a molten salt, and heating to oxidize the cerium to cerium dioxide. This process minimizes equipment size, and the effects of radiation and decay heat should be much less than with the solvent extraction process. Either sodium or magnesium nitrates were found suitable as fluxes, but magnesium appears better for the present application since the feed solution may be evaporated to a molten salt without formation of a solid phase and conversion to cerium dioxide is accomplished more rapidly. The temperature should be controlled to within 10 or 20 C since lower temperatures require longer times for cerium oxidation while higher temperatures may result in precipitation of other rare earths or part of the magnesium.

The major disadvantage of the pyrochemical process appears to be the problem of separating the precipitated cerium from the molten salts. The thickness of precipitate must be kept small to avoid excessive heating. Batch or continuous filtration processes may be used with the molten salts, or water may be added and the separations made from an aqueous system. The precipitate as formed is probably finely divided, but filtration has been demonstrated. Following a water wash of the precipitate to reduce contamination of the cerium product with inert materials, the cerium product is immediately ready for encapsulation while the product from solvent extraction must be converted to the oxide before encapsulation.

Promethium Purification

Prior to promethium processing by ion exchange it is essential that most of the cerium and yttrium be removed to minimize radiation damage to the resin. The yttrium is of no concern to this plant since it is removed in the CSREX process. Therefore, the feed to this process is the rare earth fraction after cerium removal. The feed is loaded onto Dowex 50-X12 resin, which has a capacity of about 2.0 equivalents per liter of resin.

(4) Johnson, R. W and E. H. Olson, Separation of Cerium From Other Rare Earths by Ignition of the Nitrates, ISC-1069, September, 1958

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The resin as received is in the hydrogen form and must be converted to the ammonium form for use in the loading column and to the aluminum or stable yttrium form for use in the elution columns. Conversion is accomplished by passing a solution containing the desired ion (for example, ammonium, aluminum, or yttrium nitrates) through the column until the hydrogen has been displaced to the desired extent. This may be done either before placing the resin in the columns or after the resin is in place.

Loading is accomplished at ambient temperature in about 48 hours or with a maximum flow rate of about 10 ml/min/cm² (150 gal/hr/ft²), whichever is slower. Since the proposed feeds are relatively concentrated, the 48 hour minimum loading time is assumed. The manganese or magnesium introduced in the cerium separation process also load onto the column. Either the column must be large enough to accommodate this extra material, or a simple separation step must be devised and included prior to ion exchange loading. The second column must have a capacity approximately equal to the quantity of rare earths loaded onto the first column. Since about 75 percent of the rare earths are eliminated at the end of the second column (the front 5 percent and the back 70 percent), the third column may have a capacity of about one-fourth of this amount. The fourth column must have the same capacity as the third since no rare earths are removed after the third column. The last two columns (No. 5 and 6) may have capacities of about half of the third and fourth columns since half of the remaining rare earths are removed at the end of the fourth column. (In practice some removal of rare earths will occur after the third and fifth columns, but this is not shown on the flow-sheet since the amount is small.)

All of the elution columns are operated at a temperature of 50 to 60 °C with a flow rate of 3 ml/min/cm² (44 gal/hr/ft²). The front edge of the band will thus move at a rate of 0.133 ft. per hour, or 7.5 hours per foot. Removal of the barrier ion (aluminum or yttrium) from a 12 foot column will require 90 hours. Elution of the rare earths from the column will require an additional 90 hours since the quantity of rare earths on a column is approximately equal to the quantity of barrier ion initially present. Each column will therefore be in service for about 7.5 days. Additional time is required in the fourth column since much of the rare earths band is eluted to a smaller column, requiring a lower flow rate of eluting solution. A decreased time cycle may be obtained by increasing the diameter of the last two columns, but the band length on the product (sixth) column will be shorter. The promethium band length on the final column should be at least four feet to minimize mixing at the front and back of the band, and not over eight feet to minimize radiolysis of the resin. (Use of longer final bands might be possible, but has not yet been demonstrated.)

Because of damage from the promethium radiation, the resin in the two final columns (No. 5 and 6) will be used a single time and then discarded, at least if the band length is on the order of eight feet. Use of the resin in the intermediate columns (No. 3 and 4) for about two cycles should be possible, and even more cycles may be permissible

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in the larger columns (No. 1 and 2) since the dose from promethium per unit volume of resin is smaller. However, in the first two columns the dose from cerium and yttrium may be limiting on resin life, rather than the promethium radiation, resulting in single cycle utilization.

Three different arrangements of column sizes with 12 ft. resin bed length will demonstrate the allowed limits in the equipment. Final columns of four inch diameter with an eight foot promethium band, or six inch diameter with either a four or eight foot band appear reasonable. This corresponds to batch sizes of 300, 300, and 600 tons of uranium respectively. In either the first or second case, the intermediate columns (No. 3 and 4) would be about eight inches in diameter, and the second column would be 14 inches in diameter. The loading column (No. 1) would be 14 inches if the feed contains no extraneous ions, 20 inches for the manganese-containing feed, or 24 inches for the magnesium-containing feed. An individual column would be in use for a maximum of 10 days per batch with the four inch columns, or seven days per batch for the six inch columns. Allowing one day for turn-around, batches could be started every 11 or 8 days respectively. In the third case (that is, a six inch column (No. 6) with an eight foot promethium band), the intermediate columns (No. 3 and 4) would be 10 inches and the second column would be 20 inches. The loading column (No. 1) would be 20 inches for pure feed, 26 inches for the manganese-containing feed, or 32 inches for the magnesium-containing feed. Each individual column would be in use for a maximum of 10 days per batch, permitting batches to be started every 11 days. Processing rates would be 27, 37, or 55 tons per day. With batches of 600 tons of uranium, the cycle could be reduced to nine days (including one day for turn-around) if the final columns were increased to eight-inch diameter, thus increasing the processing rate to 67 tons per day, but decreasing the band length on the final column to about 4.5 feet. In any of these cases, about 25 days are required from the beginning of a loading cycle until the product is removed from the final column.

The process as currently planned will produce most of the promethium at well over 99 percent purity, but the promethium at the band edge will contain up to five percent impurities. Back-cycling of the impure promethium fractions from the final column will permit production of all promethium at very high purity. Alternatively, promethium may be removed at 95 percent purity from an earlier column, but in this case the impure promethium fractions at the band edge will have to be recycled. Increased throughput may be achieved by increasing the elution flow rate, but this may result in reduced purity.

The location of promethium in the process is followed by gamma monitoring, using the radiation from Pm-148. Samplers and continuous gamma monitors should be provided at the bottom of each column to facilitate process control. Provision must also be made for removing the gas formed by radiolysis in the columns. Provision for back-cycling material from the elution columns to the second column (first elution column) or to the loading column during elution to the second column would be helpful in minimizing promethium losses and possibly in increasing throughputs.

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SOLVENT TREATMENT AND WASTE HANDLING

The various flowsheets and the discussion above indicate special solvent treatment and waste rework facilities will be required. The only solvent treatment contemplated at this time would be one or more successive batch washes with various aqueous reagents. Adequate provision for washing dictates the use of at least four vessels in each solvent-handling chain: a receiver tank, two wash tanks equipped to give a fine decant between aqueous and organic phases, and a solvent feed tank. Normally one of the wash tanks would be used as an alternate feed tank. All tanks should be equipped with agitators to permit their use as batch wash tanks. Aqueous wash solutions would be added and removed batchwise. The spent wash may occasionally require rework to recover product values.

Generally, rework of aqueous wastes may be done by backcycling through the solvent extraction columns. Occasionally such wastes may require concentration to reduce the volume or digestion to remove some undesired impurity. Strontium Semiworks experience has shown that at least one and preferably two general purpose waste concentrators are highly desirable. These concentrators must have adequate vapor space and oversized scrubbing or disengaging towers to handle heavily foaming solutions such as might be derived from the various organic materials in the processes.

Source Form Preparation and Encapsulation

The accompanying flowsheets (Figures III through VI) provide for conversion of the purified fission product solutions to the source forms listed previously. These forms are strontium titanate, strontium oxide, cesium borate glass, cerium dioxide, and promethium sesqui-oxide. Though these product forms may become obsolescent, the recommended conversion and encapsulation techniques are sufficiently generic as to permit adaptation to meet changing product needs.

Conversion of the purified fission product to the source form is in all cases achieved by precipitation of the final product or of an intermediate compound. Thus, strontium carbonate is precipitated for subsequent calcination to the oxide or for blending with titanium dioxide for calcination to strontium titanate. Cesium is precipitated as the acid oxalate and subsequently blended with glass forming agents for preparation of cesium borate glass. Cerium, if prepared via the nitrate pyrochemical process is already in the precipitated oxide form. However, if cerium is isolated via the solvent extraction route, oxalate precipitation followed by calcination to the oxide is required. Promethium oxide is prepared via calcination of the oxalate salt.

At the present time, proven technology exists only for the small scale preparation of small strontium titanate pellets and much "hand work" is required. Technology for the preparation of cesium borate glass is in a very early stage of development and technology for preparation of strontium oxide, cerium oxide, and promethium oxide sources is largely non-existent.

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High-rate-densification (Dynapak) has been selected as the densification and initial encapsulation technique. This process overcomes the disadvantages inherent in scale up and adapting of the existing strontium titanate ceramic technique in that the Dynapak process is largely independent of source form, inert atmosphere handling is not necessary for strontium oxide preparation, large sources can be prepared, radioactive material handling is minimized, accurate diametral sizing of pellets is achieved, and cell volume requirements are potentially minimized.

As currently visualized, the densification process will consist of loading the primary compaction capsule with the product form or an intermediate as follows:

<u>Source Form</u>	<u>Capsule Loading Intermediate</u>
SrTiO ₃	SrCO ₃ and TiO ₂
SrO	SrCO ₃
Cs Glass	Cs oxalate, boron oxide, silica
CeO ₂	Ce oxalate or CeO ₂
Pm ₂ O ₃	Pm oxalate

Capsule loading may be conducted dry or, hopefully, by direct filtration. If dry loading is employed, preliminary cold pressing may be necessary. The primary capsule is then capped with provision for outgassing and thermally soaked under moderate vacuum prior to sealing of the outgassing tube. The sealed capsule is loaded into the Dynapak die and compacted under impact pressures of 100,000 to 400,000 psi. Cleaning, second encapsulation, second cleaning, leak detection, and calorimetric measurement follow.

CHEMICAL COSTS

It is not in the scope of this report to present a detailed analysis of the economics of the proposed flowsheet or its alternates; however a quick survey of the chemical costs has been made and is given in Table V.

TABLE V

CHEMICAL COST ANALYSIS

	<u>\$/Ton U</u> <u>Processed</u>	<u>\$/Watt of</u> <u>Recovered</u> <u>Isotopes(a)</u>	<u>\$/Kilocurie(a)</u>	<u>Isotopes</u> <u>Processed</u>
<u>B-Plant (CSREX</u> <u>Process)</u>				
Solvent Extrac- tion(b)	19.64	-	-	Sr-90 Cs-137 Ce-144 Pm-147
Acid-Kill	0.84	-	-	
Solvent Washing	<u>0.91</u>	-	-	
Total	21.39	0.072	0.495	

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Table V Continued

	<u>\$/Ton U Processed</u>	<u>\$/Watt of Recovered Isotopes (a)</u>	<u>\$/Kilocurie(a)</u>	<u>Isotopes Processed</u>
<u>FPPP (Purification) and Precipitation)</u>				
Strontium-Cesium Separation	0.012	0.0006	0.003	Sr + Cs
Strontium Purifi- cation				
Solvent Extrac- tion(b)	1.80	-	-	
Precipitation	0.043	-	-	
Solvent Washing	<u>0.037</u>	<u>-</u>	<u>-</u>	
Total	1.88	0.165	1.10	Sr-90
Cesium Purifica- tion				
Solvent Extrac- tion(b)	0.119	-	-	
Precipitation	<u>0.041</u>	<u>-</u>	<u>-</u>	
Total	0.160	0.018	0.084	Cs-137
Cerium Purifica- tion				
Solvent Extrac- tion (Alt. I)	5.93	0.0215	0.173	Ce-144
Pyrochemical (Alt. II)	0.37	0.0013	0.011	
Promethium Puri- fication(c)	4.78	2.12	0.898	Pm-147
Total Cost, FPPP(d)	7.20	2.30	0.167	All
Total Cost, FPPP B-Plant(d)	28.60	2.37	0.662	All

(a) Assumes the recovery of 95 percent of the Sr-90 and Cs-137 (aged one year), 90 percent of the Ce-144 and 75 percent of the Pm-147 (aged 180 days)

(b) Assumes 0.5 percent of the solvent loss per pass (BAMBP at \$3/pound)

(c) Assumes resin discarded after each pass

(d) Assumes pyrochemical process

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These costs as far as possible are based on prices published in Chemical and Engineering News, May 7, 1962. Prices not available from this source are based on Hanford Essential Materials Costs or vendor quotations. The following are some of the observations pertinent to the cost analysis.

1. The organic complexing and buffering reagents, such as citric acid and oxalic acids, are major costs in most of the various flowsheets.
2. Solvent costs, except in the cerium solvent extraction purification process, are relatively low at the assumed 0.5 percent loss rate. For example, the solvent cost is only about 20 percent of the total chemical cost in both the CSREX and the cesium purification flowsheets.
3. In the promethium purification flowsheet, the resin represents about 80 percent of the chemical cost, based on what may be an unrealistic assumption that the resin is discarded after each loading cycle.
4. The cerium pyrochemical flowsheet cost is only 6 percent of the solvent extraction flowsheet cost. This difference may be enough to encourage development of this unique process. (Note that the costs of cerium watts and kilocuries as packaged are based on 180-day cooled material).

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FIGURE I
Extraction Process
Treatment Program
Waste Processing
Sheet No.2

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FIGURE II

Fission Product Production Plant
Preparation of Strontium and Cesium Crudes
Study Flowsheet No.2

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FIGURE V

Fission Product Production Plant
Cerium Purification and Packaging
Study Flowsheet No.2

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Production Plant
Cerium Purification and Packaging
Study Flowsheet No.2

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FIGURE VI

Fission Product Production Plant
Promethium Purification and Packaging
Study Flowsheet No. 2

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