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NP^{237} AND PU^{238} SEPARATION AT THE SAVANNAH RIVER PLANT

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A program to manufacture Pu-238, an alpha emitter well suited for use in power generators in space vehicles, was assigned to the Savannah River Plant (SRP) in 1959. Pu-238 is produced by neutron irradiation of Np-237 which has previously been separated from irradiated uranium. Ion exchange and solvent extraction processes were developed at the Savannah River Laboratory for the initial recovery of Np-237 from irradiated natural and enriched uranium, and for the separation and purification of neptunium and plutonium from irradiated neptunium targets. The radioactivity of the solutions associated with the neptunium and plutonium required that new plant equipment be installed in the heavily shielded Separations processing areas.

Initial Neptunium Recovery - Solvent Extraction

RECOVERY FROM PUREX

The fundamental SRP Purex process (figure 1) required only one modification to insure that essentially all neptunium in the feed appears in high level waste (1AW) from which it is recovered by ion exchange. Nitrite was added to the next to last stage of the 1A mixer-settler extraction section to maintain the neptunium in the +5 valence state and thus minimize neptunium reflux and consequent risk of loss to streams other than the 1AW; the nitrite added is equivalent to 0.007M in the solvent phase. High uranium saturation of solvent at the feed stage minimizes neptunium loss from the 1A mixer-settler.

The 1AW, the 1DW (second uranium cycle waste which contains most of the neptunium that escaped the 1A mixer-settler), and the wastes from the neptunium purification frame are combined and concentrated. This waste concentrate contains most of the neptunium present in the Purex plant feed.

RECOVERY FROM ENRICHED URANIUM

The flowsheet used to recover Np-237 from the enriched uranium process (figure 2) maintains neptunium in the +4 valence state throughout two cycles of solvent extraction by addition of ferrous sulfamate to 1AF, 1AS, 1BX, 2AF, and 2AS streams. Neptunium is extracted along with enriched uranium in the 1A mixer-settler and partitioned from uranium in the 1B mixer-settler. Further decontamination is obtained in the second cycle using the normal Purex flowsheet for the second plutonium cycle, except for additions of ferrous sulfamate.

The original flowsheet for the enriched uranium process was modified to isolate most of the neptunium in a new 1BP stream and to reduce the loss to the 1AW as follows.

◊ More of the neptunium was maintained in the +4 valence state in the 1A mixer-settler by adjusting the 1AF with ferrous sulfamate in addition to the ferrous sulfamate already in the 1AS.

◊ The TBP strength of the solvent was increased from 2.5% to 3.5%.

◊ The 1B mixer-settler was placed in service to partition the neptunium into a uranium-free 1BP stream.

◊ Minor changes were made in flow ratios, primarily in the 1B and 2A mixer-settlers.

Ion Exchange Processing

PROCESS OUTLINE

The process sequence for recovery and purification of Np-237 from Purex waste concentrate is shown in figure 3. The strongly acidic concentrate is cooled and the neptunium valence is adjusted to the absorbable +4 state with ferrous sulfamate and stabilized with hydrazine. The adjusted concentrate is fed to an agitated anion column (primary recovery column or PRC). Np-237 is absorbed on the resin along with Pu-239, which is present primarily from the 1AW, even though the plutonium valence in solution is predominantly +3 because of the presence of Fe^{+2} . The plutonium is absorbed effectively because the strong affinity of the resin for the Pu^{+4} nitrate complex causes a continuous shift of Pu^{+3} to

Pu^{+4} to satisfy the solution equilibrium. After the feed step, the column is washed with clean 8M nitric acid (not recovered acid) and neptunium and plutonium are eluted with 0.035M nitric acid. The eluate is transferred to the hold tank for neptunium purification.

Recovered neptunium is passed through two anion columns and one cation column. Pu-239 is separated from neptunium on the first anion column and is transferred to the second plutonium cycle of the main Purex process.

The irradiated neptunium processing is outlined in figures 4 and 5. Neptunium and plutonium are dissolved, processed together through the first anion column, and partitioned on the second anion column. The plutonium receives one additional decontamination cycle.

GENERAL DESIGN

New equipment installed in the existing Separations canyon facilities[1] consisted of four ion exchange columns for neptunium recovery from natural uranium and five columns and a dissolver for neptunium-plutonium separation from irradiated neptunium targets. The size of the ion exchange columns and their associated tankage, small by comparison with normal canyon equipment, permitted development of a concept in which a number of columns and tanks were mounted in a frame that could be installed as a unit in one module.[2] Three frames were installed in the canyons, two for neptunium-plutonium recovery and separation from the irradiated neptunium targets and one for purification of neptunium recovered from Purex. A single ion exchange column and its supporting tanks were installed by more conventional methods, but all in one module, for initial recovery of neptunium from Purex waste. Another single-column system was installed to recover neptunium and plutonium from irradiated target processing wastes.

This type of installation is much more economical than a conventional installation in which each vessel is placed in one canyon module (a canyon space approximately 10 feet square by 17 feet high with a standard arrangement of pipe nozzles to supply services). Only a very few pieces of equipment mounted in a frame can be removed separately from the whole frame but critical items susceptible to failure were made individually remotable. These included ion exchange columns, air lifts, steam jet syphons, and any mechanical agitators.

Placing several equipment pieces in a frame in a single module is economical of canyon space but places a heavy burden on services available for that module. Pipe and electrical connections were made to the frame (by conventional canyon jumpers) rather than to the individual pieces of equipment. Services were piped to equipment pieces as part of the permanent frame structure. This efficiently utilized existing services but additional services were required, primarily for pneumatic liquid level and specific gravity instrumentation for the several vessels in each frame. Bundles of up to six small, stainless steel tubes (1/2" OD) were drawn through the 3" pipes embedded in the canyon shielding walls. Practical demonstration of this technique has great utility in adapting existing facilities to alternate processes. No significant problems have been encountered with the installation of these lines or their use; no lines have been removed from service.

PROCESS CONTROL

Space and service piping limitations restrict moderately the ability to monitor each individual step of the process at any given time. For example, only four tanks in the first target processing frame can be sampled at one time although each may be sampled by moving one or more canyon jumpers; normally RC-2 feed tank is not sampled. Despite the absence of routine analytical observation of every process step, combinations of liquid level and specific gravity instruments and samples of all waste have provided excellent knowledge of performance as follows:

◊ Feeds to columns RC-1, RC-3, RC-5, 2K, and 5K are analyzed.

◊ Feed adjustments to 8M nitric acid are confirmed by specific gravity measurement.

◊ Waste streams from each column are analyzed.

◊ Decontamination for various columns is measured by analyzing product solutions from columns RC-2, RC-4, RC-5, PRC, 2K, and 7K.

◊ Partition efficiency is measured by analyzing both products from columns RC-2 and 2K.

PROCESS PERFORMANCE

Primary Recovery from Purex Waste. Waste concentrate to be fed to the agitated anion exchange column is adjusted in the bottoms run tank of the continuous 1AW evaporator. The continual introduction of thermally hot, unadjusted concentrate and the radiation of the concentrate require more ferrous sulfamate and hydrazine to achieve and hold neptunium +4 valence than any other column; up to 0.15M of each is used. Concentrate temperature is held to 35°C, or less, to minimize consumption of reductants.

Nitrate strength of the waste, as concentrated, must be lower than is desired for efficient column operation to avoid volatilization of ruthenium in the evaporator. Nitrate strength of the concentrate is increased approximately 1.3M to at least 7.8M by the addition of 13M nitric acid.

A single stage agitated anion exchange bed recovers 70-75% of the neptunium fed to it. Recovery has been improved to more than 90% by placing three agitated beds in series. Pu-239 recovery typically is slightly higher than neptunium recovery. Agitation is essential to passing the waste concentrate through the bed. The entire feed step and the first two bed volumes of decontamination wash are fed to an agitated bed; the remainder of the wash and the elution are fed to a settled bed.

Decontamination from fission products is very good and the over-all DF averages about 5000. Thorium in the eluate is below the limit of routine analytical detection; the slight uranium content is readily removed by subsequent anion exchange cycles. The low thorium content of feed to the neptunium purification frame indicates that the final cation exchange column in the frame could be replaced by anion exchange; fission product decontamination achieved on the final column is very desirable but this could be achieved with an anion column.

Dissolution of Neptunium Targets. The frame dissolver is charged with irradiated neptunium target slugs[3] (aluminum-jacketed compacts of neptunium oxide[4] and aluminum) and nitric acid. Dissolution is catalyzed by successive additions of mercuric nitrate and potassium fluoride. Fluoride is added only after enough aluminum has dissolved to insure that the fluoride will be complexed. The dissolver solution contains small quantities of solids, primarily silica. These solids would plug a settled resin bed, so the solution is filtered through a 5-micron stainless steel filter during the transfer to the feed tank for the first anion column. Irradiated slugs dissolve slowly with no significant surge of off-gases. A negative pressure of approximately one inch of water is adequate to prevent dissolver pressurization and spread of contamination. Dissolver off-gases are discarded to the environment after they have been water scrubbed and filtered through fiberglass and sand filters. No significant alpha contamination has been observed in the water scrubber.

Dissolving times of 20 and 48 hours per cycle have resulted in 60% and 85% dissolution, of the total charge, respectively, with catalyst concentrations of 0.005M mercuric nitrate and 0.02M potassium fluoride. The heel in the dissolver thus approaches a constant value for constant dissolving conditions. In two dissolver cleanouts, the ratio of neptunium to plutonium in the heel has been the same as in freshly charged slugs. Since there is no selective dissolution, the only incentive for heel cleanout is to account for neptunium and plutonium in the heel.

Anion Column Operation in the Frames. All of these ion exchange columns are of the agitated bed design[5] that permits remote resin replacement. Unlike the column used for initial recovery of neptunium from Purex waste, the frame columns are operated as settled beds except during resin regeneration or replacement. Feeds are adjusted to a range of 7 to 8M total nitrate with nitric acid (neptunium target dissolver solution includes other nitrates, primarily aluminum nitrate). Ferrous sulfamate is added to reduce neptunium to the readily absorbed +4 valence state. Hydrazine is also added to the adjusted dissolver solution which is then heated to 55°C for 30 minutes to destroy ferrous ion and oxidize plutonium to the +4 state; the brief period of heating does not oxidize the neptunium.

Each resin column is prepared by washing downflow with 8M nitric acid that contains 0.1M potassium fluoride; the concentrated acid replaces the dilute acid from the column and the fluoride improves removal of fission products. The column is agitated during part of this regeneration step to redistribute resin within the bed and insure uniform resin exposure, especially to alpha bombardment from Pu-238. Fluoride is flushed from the column with 8M nitric acid.

Adjusted feed solution is passed downflow through the settled resin bed and the anionic complexes of plutonium and neptunium are selectively absorbed. Most fission products and other cations pass through. More complete decontamination from fission products is obtained by washing with 8M nitric acid; on some columns this wash contains 0.005M potassium fluoride. If neptunium and plutonium are to be co-eluted or if only one is present, the column is eluted next with 0.35M nitric acid; if neptunium and plutonium are to be separated, a plutonium partition wash of 4.0 to 6.0M nitric acid, 0.05M ferrous sulfamate, and 0.05M hydrazine is used to remove plutonium before neptunium is eluted.

Anion Exchange Resins. "Dowex 1" and "Permutit SK" anion exchange resins are used. "Dowex 1" resin provides higher decontamination from fission products but "Permutit SK" resin is more stable under alpha radiation. For these reasons, "Dowex 1" resin has been specified for columns used principally for decontamination or for neptunium alone, whereas the "Permutit SK" resin has been used in the Pu-238 columns.

The "Dowex 1" resin has been used in various particle sizes and with different cross linkages. Originally, "Dowex 1-X4" (4% nominal cross linkage) was used in the 40-60 mesh size; later, special resins of lower cross linkage (down to 2%) were shown to provide better absorption and elution performance, so that 30-50 mesh resin may be used with consequent flow increases.

The flow that can be achieved through any settled resin bed depends on the size of resin particles and, more importantly, on the amount of gas collected within the bed. The effect of gas is most pronounced in columns processing Pu-238. Evidently alpha-induced radiolysis of nitric acid to nitrous acid is the principal source of gas; the reaction of nitrous acid with residual sulfamate releases nitrogen which collects in the resin bed and restricts flow. Flow is maintained to the column throughout each run to prevent the settled but unrestricted resin bed from being disturbed by collected gas. Efforts to eliminate the flow restriction due to gas have been relatively unsuccessful. Extended heat kills have increased neptunium losses, while the use of less ferrous sulfamate has decreased gassing only slightly. Sodium nitrite was added to the feed to a plutonium column to destroy sulfamic acid; flow improved but plutonium losses were high near the end of the cycle.

During operation, resin is degraded gradually by alpha bombardment. Slight to moderate damage has little effect on performance (losses tend to increase) presumably because only 10-30% of the initial exchange capacity of the resins (1.2 - 1.6 mol equivalents per liter of packed resin) is normally used. Very high exposure can result in physical changes such as significant particle softening and clinker formation. Resin exposure is measured in Pu-238 grams absorbed times the hours of exposure; most plant experience has been limited to 180 Pu-238 g-hr/liter for "Dowex 1" resin and 720 Pu-238 g-hr/liter for "Permutit SK." Higher exposures are being explored. Brief experience with exposures as high as 600 Pu-238 g-hr/liter for "Dowex 1" and 1200 Pu-238 g-hr/liter for "Permutit SK" has been satisfactory so far as resin removal from the columns is concerned but further process evaluation is required.

These anion resins shrink and swell with changing acidity during the processing cycle. The volume change varies with cross linkage. The amount of resin charged to a column is kept constant by measuring the charge volume as the settled resin volume in a solution of 1.2M nitric acid. Resin is charged by slurrying it as a near-suspension in a solution of aluminum nitrate or sodium nitrate at a specific gravity of 1.19, or in a solution of nitric acid at a specific gravity of 1.27. Occasionally some size classification and consequent flow restriction occurs during resin charging because of the particular combination of flow and column agitation. This is best corrected by agitating without flow and pulsing the air pressure on the column outlet weirs to clear the resin retention screen. Resin is removed from the columns by pressurizing both outlet weirs and overflowing several bed volumes of solution.

Anion Column Absorption Performance. If neptunium and plutonium valences are correctly adjusted and maintained in the feed, losses from anion columns depend on feed and wash volumes, actinide and nitrate concentrations, feed rate, and total load on a column. The valence adjustment described earlier has been uniformly satisfactory except that it was modified to include the addition of hydrazine to RC-1 when feed of short cooling time (50-100 days) was processed. Neptunium and plutonium losses from RC-1 increased sixfold and twofold, respectively, as the cooling time of the feed was decreased below 100 days. Evidently the increase in the products of gamma radiolysis interfered with valence adjustment so that undesirable neptunium and plutonium oxidation occurred. Another benefit from hydrazine was a twofold decrease in volatilization of iodine from the system so that only approximately 0.002 curie is now released per curie charged to the frame dissolver.

Losses during the feed step have averaged less than 0.1% for all columns except RC-1 which has had typical losses of ~0.3%. Larger losses occur to decontamination wash waste and to reconditioning wash waste. Reconditioning loss is a result of incomplete elution when actinides that have not been eluted are washed from the column by 0.1M fluoride in the reconditioning solution. The table below shows the pattern of loss among waste streams.

Typical Process Losses

Column	Total Waste Loss, % of feed	Portion of Loss, % of total		
		Reconditioning	Feed	Decontamination
RC-1				
Np	1.4	15	25	60
Pu	2.8	40	10	50
RC-2				
Np	0.7	13	10	77
Pu	0.6	15	2	83
RC-3				
Pu	0.4	80	11	9
RC-4				
Pu	1.1	57	3	40
RC-5				
Np	0.5	84	8	8

All wastes are now combined and fed through another anion exchange column called the frame waste recovery (FWR) column. Recoveries greater than 97% are achieved with the FWR system for neptunium and plutonium so that over-all losses are very low. Eluate from the FWR column is transferred to column RC-1 in Frame I-H. Prior to installation of the FWR system, reconditioning washes were recycled within the frames to recover the significant loss contained in the small volume.

The effect of decontamination wash volume on losses is summarized below for RC-1 performance with a normal actinide loading. Decontamination was ample at the lowest volume of wash shown and was not significantly greater at the larger volumes.

Wash, bed volumes	Losses, wt % of feed	
	Np	Pu
30	18	6
20	10	3
10	1	1

The rate of loading actinides on a column has an effect on losses that is at least partly independent of the effect of feed flow rate. After the FWR system was in use and recycle within the frame was discontinued, the volume of feed decreased and concentration of actinides in the feed increased. Loading actinides on the resin in a column 35% faster than normal but at constant volume rate resulted in an 80% increase in the loss, an indication that resin absorption and diffusion rates are limiting.

Anion Column Decontamination Performance. Decontamination was so good that the final two columns of the original irradiated target processing system were removed from service. The main decontamination is achieved during the feed step because the fission products either are not absorbed on the resin or are only lightly absorbed. The subsequent decontamination wash displaces the final volume of feed from the column and washes off weakly absorbed fission products. Decontamination increases significantly as the wash volume is increased up to about 10 bed volumes. Decontamination of neptunium and plutonium on RC-2 increased fivefold and twentyfold, respectively, when wash volume was increased from two to five bed volumes. Decontamination of neptunium on RC-5 increased by a factor of 2 to 3 when wash volume was increased from five to ten bed volumes.

Typical decontamination factors for the frame columns are summarized in the following table.

Decontamination Factors

	Zr-Nb	Ru	Gross γ	Gross γ Forecast*
RC-1 and RC-2				
Np	8,000	25,000	9000	9000
Pu	12,000	3,000	6000	5000
RC-3 and RC-4				
Pu	-	-	300	100
RC-6				
Np	11	12	13	20
PRC				
Np	-	-	5000	1000
2K				
Np	-	-	500	400
5K-7K				
Np	-	-	50	100

* From design data.

The limiting fission product activity for neptunium from irradiated target processing is Zr-Nb. The gross gamma activity will increase because of the growth of protactinium-233, a neptunium daughter which has a 27-day half life. Protactinium activity is not a problem normally because only a short storage time is allowed between process steps. Decontamination of neptunium purified after recovery from natural uranium and of the plutonium product from irradiated target processing is limited by ruthenium. The poorer ruthenium decontamination of Purex neptunium may be associated with the nature of feed to the PRC. The poorer ruthenium decontamination of plutonium from the irradiated targets is attributed to selective removal of ruthenium during plutonium partiion as a result of hydrazine in the partition solution. This is confirmed by high ruthenium decontamination of neptunium product from the partition column and by a fivefold increase in ruthenium decontamination when RC-1 feed was adjusted with hydrazine (ruthenium retention on the RC-1 column was apparently reduced with consequent improvement in ruthenium decontamination).

Good decontamination from uranium is routinely achieved with the anion exchange columns. Uranium may be introduced as a contaminant by the initial neptunium recoveries from irradiated natural and enriched uranium processes and must be removed prior to target slug fabrication to avoid problems in the reactors. Decontamination from uranium improves with wash volume as shown in the following table.

Decontamination from Uranium

Decontamination Wash, bed volumes	Uranium DF
2	15
4	40
5	70

Production of a very pure plutonium product requires essentially complete removal of any feed and its contained ferric ions from the column prior to elution. RC-4 column was washed with four bed volumes of decontamination wash to remove the ferric ions whether or not fission product decontamination was necessary. Only clean, new acid (ie, not acid recovered from waste evaporation) and deionized water are used for the wash make-up. Even with these precautions, a minimum plutonium concentration of 2 grams/liter is needed to provide a ratio of plutonium to contaminants that meets purity specifications routinely.

Partition Performance. A high degree of separation is required between plutonium and neptunium to avoid the loss of either actinide as a contaminant of the other and to provide products of high purity. The separation requirement and the large number of factors affecting separation make partitioning the most demanding service in the irradiated target process. The efficiency of the partition step depends on cross linkage and size of resin particles, on the acidity of the partition solution, on the ratio of ferrous to ferric ions in the partition solution, on the flow rate of the partition solution, and on the relative and total amounts of neptunium and plutonium loaded on the column.[6]

Plutonium removal by a given volume of partition solution increases as cross linkage of the resin decreases, as shown in figure 6 (plutonium removal efficiency in plant columns is generally a little poorer than shown by these laboratory data, a difference attributed to gassing in the plant). Neptunium contamination of plutonium product is decreased with resins of lower cross linkage, both because a smaller volume of partition solution can be used and because the resin retains neptunium more effectively. The improved performance is attributed to faster absorption and elution characteristics of the resin, presumably because of more rapid diffusion in resin of lower cross linkage.

The efficiency of plutonium removal from any given resin is dependent on the acidity and flow rate of the partition solution. As acidity is decreased, plutonium and neptunium are removed from the resin more readily and reductions in acid concentration are limited to values that cause only a small leakage of neptunium to the partition solution. With resin that does not provide satisfactory partition (too much plutonium must be left on the resin unless neptunium leakage is accepted), a recycle cut of decreased acidity can be used to complete the plutonium removal. A moderate neptunium recycle is not of concern and plant experience with this technique showed that it was highly effective in partitioning with mediocre resin. Typical data are summarized as follows.

Split Acid Partition

Type of Partition	Cut	Cut Size, bed volumes	Cut Acidity, M HNO_3	% Np in Pu	% Pu in Np	% Pu Loss to Np Elution
Standard	Product	10	5.7	0.6	1.2	4.4
	Recycle	5	5.7			
Split Acid	Product	10	5.7	0.6	0.2	0.6
	Recycle	5	4.0			

A lower flow rate of partition solution also favors plutonium removal. Lower flows are not conducive to high capacity but a reasonable over-all cycle can be maintained by feeding the first half of the solution at a relatively high rate and the remainder at a slower than average rate. Only half as much plutonium is left on the resin by the split rate technique as would be left with a constant flow at the average rate. Evidently the flow rate is of principal importance after most of the plutonium has been removed. Figure 7 shows the effect of partition flow on partition effectiveness.

Anion Column Elution Performance. Neptunium and plutonium are eluted from the anion exchange columns with 0.35M nitric acid. The acidity provides efficient elution without risk of formation of plutonium polymer or other hydrolysis products. Both elements are approximately 98% eluted by 4 to 5 bed volumes of 0.35M acid. The low acidity is especially important for plutonium removal; at an acidity of 0.8M, only 30-50% of the plutonium but more than 90% of the neptunium is eluted.

When a concentrated product is desired, 80% to 95% of the column loading can be eluted in a product cut of two bed volumes; the remainder of the elution is recycled. The product cut size has been investigated in the FWR system with the following results.

Effect of Elution Cut Size

Elution Cut, bed volumes	% of Np Eluted	% of Pu Eluted
1.0	70	55
1.5	74	70
2.0	83	95

Process Capacity. Initial flowsheet and equipment designs were based on laboratory experience and provided the ability to recycle high waste losses or off-standard products. Installation of the frame waste recovery system eliminated much of the need to await analytical data and permitted some alteration of the flowsheet so that waste losses were not truly minimized. Plant experience has permitted smaller decontamination wash and reconditioning wash volumes generally. These changes substantially increased capacity above the initial design basis despite greater than anticipated flow restrictions due to gassing. Present capacity is more than 400% above the initial design capacity; further increases may require some moderate changes in equipment and should permit the capacity to be raised at least another factor or two. This favorable experience and the equipment reliability have demonstrated that it is practical to install and operate small equipment in large radiochemical separations plants.

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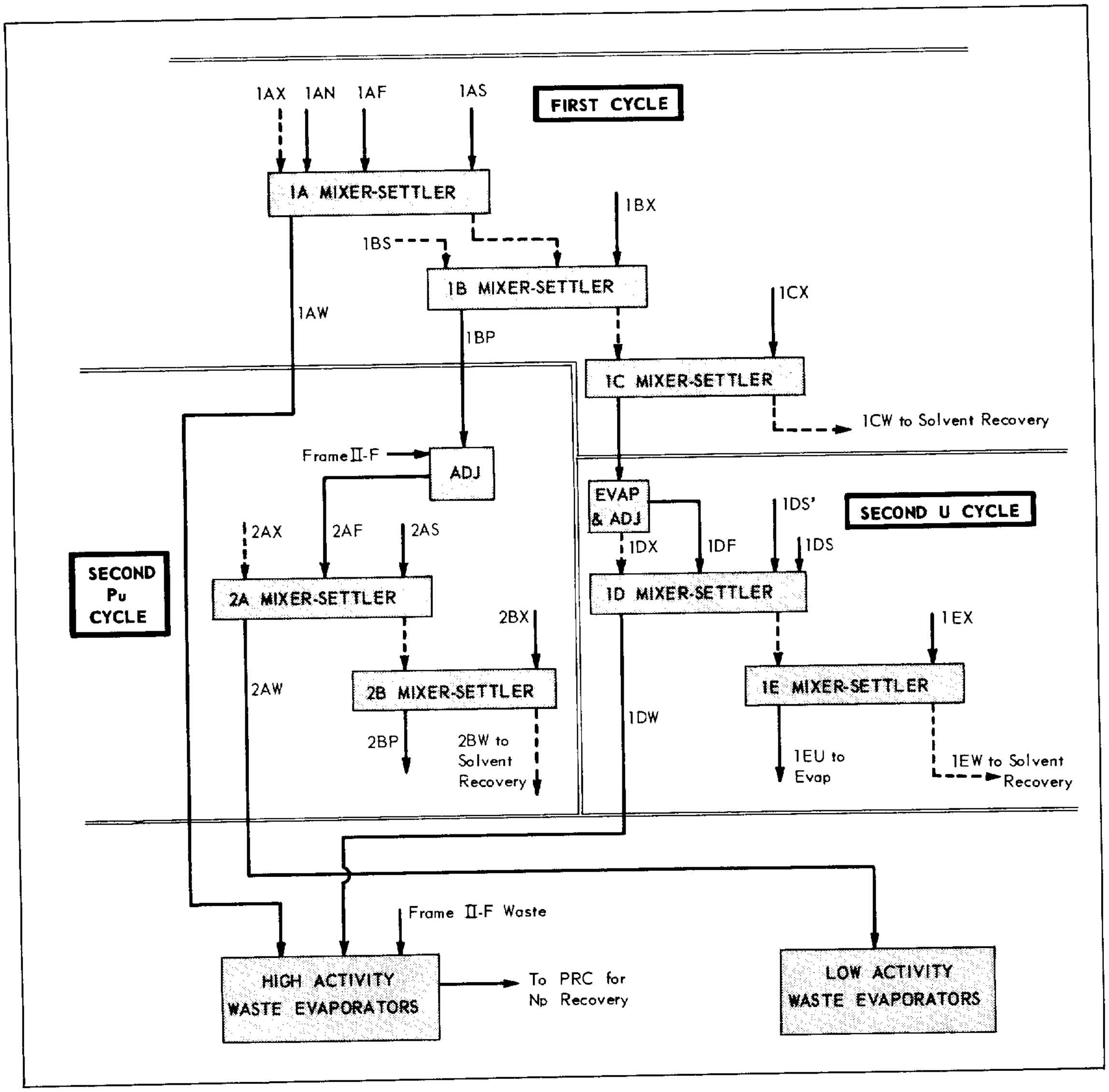


FIGURE 1. SRP PUREX

——— Aqueous Streams
----- Organic Streams

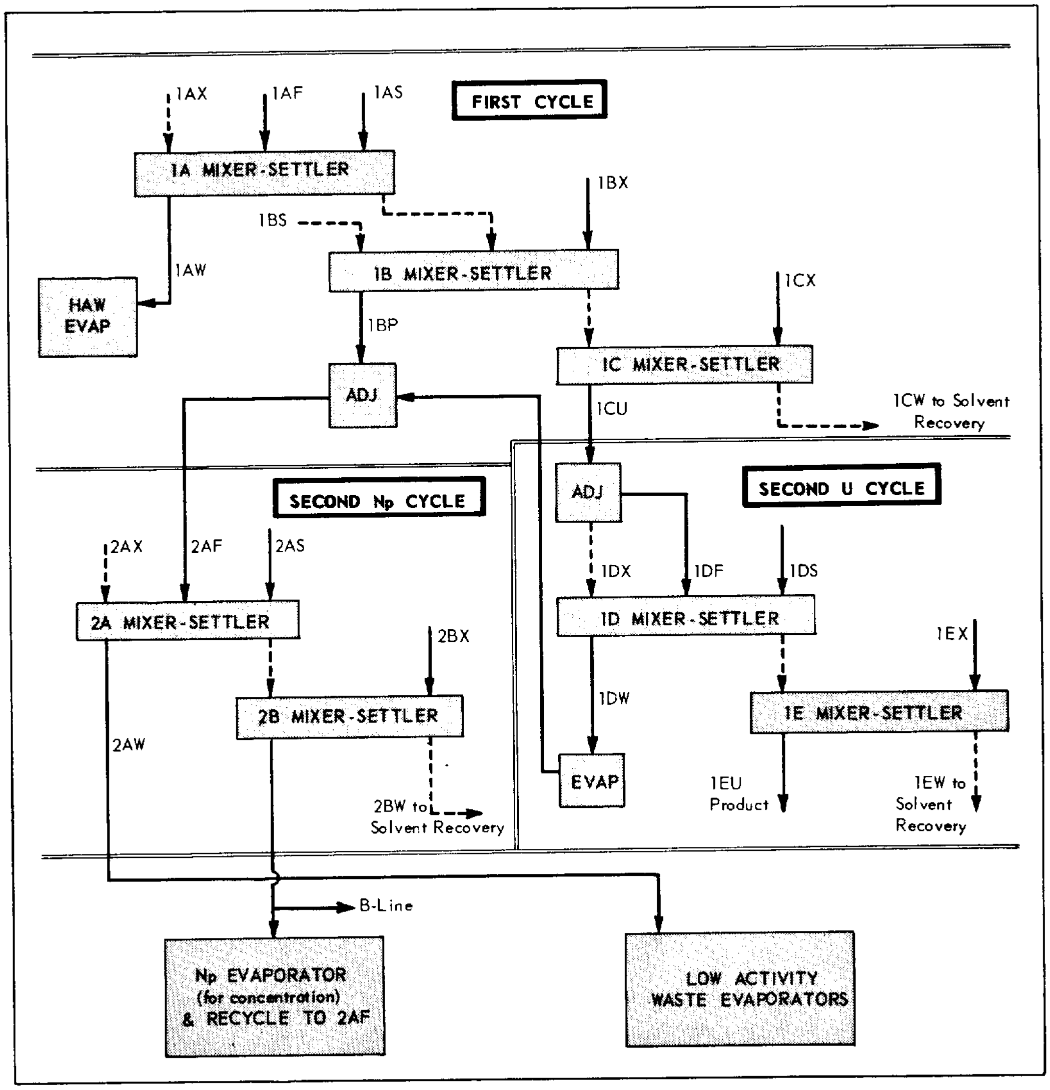


FIGURE 2. SRP ENRICHED URANIUM RECOVERY

———	Aqueous Streams
- - - - -	Organic Streams

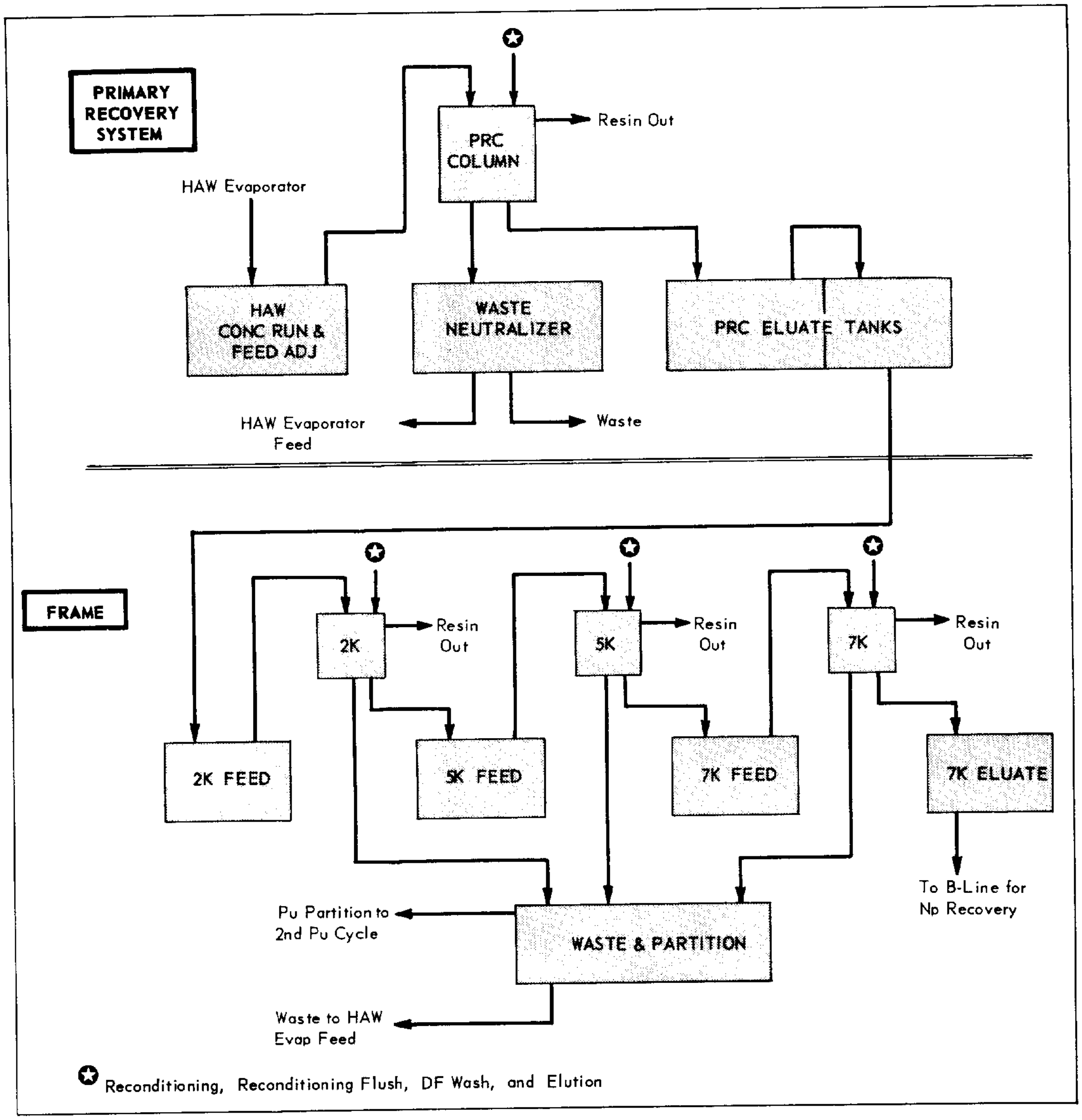


FIGURE 3. RECOVERY OF Np-237 FROM PUREX WASTE

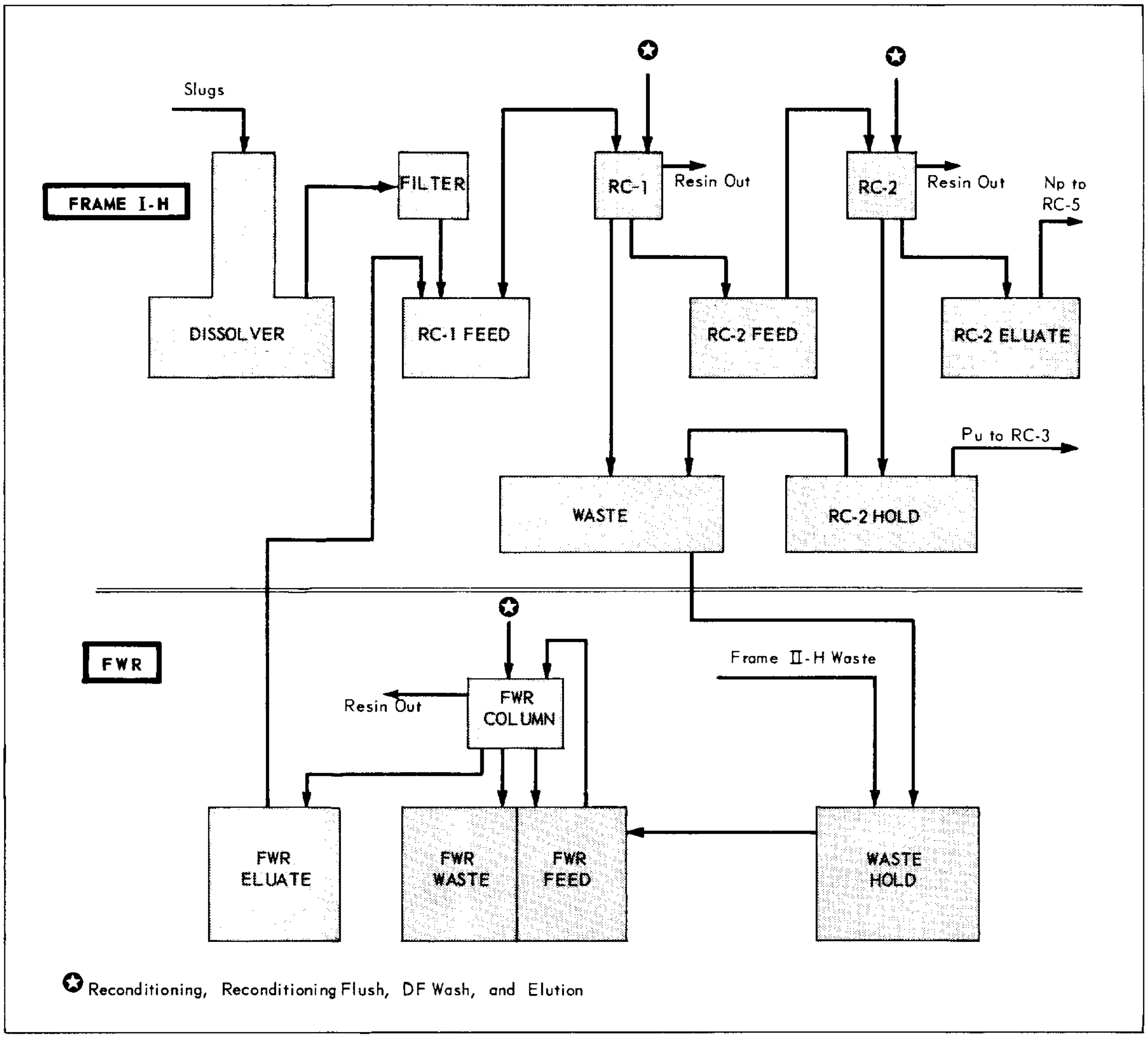
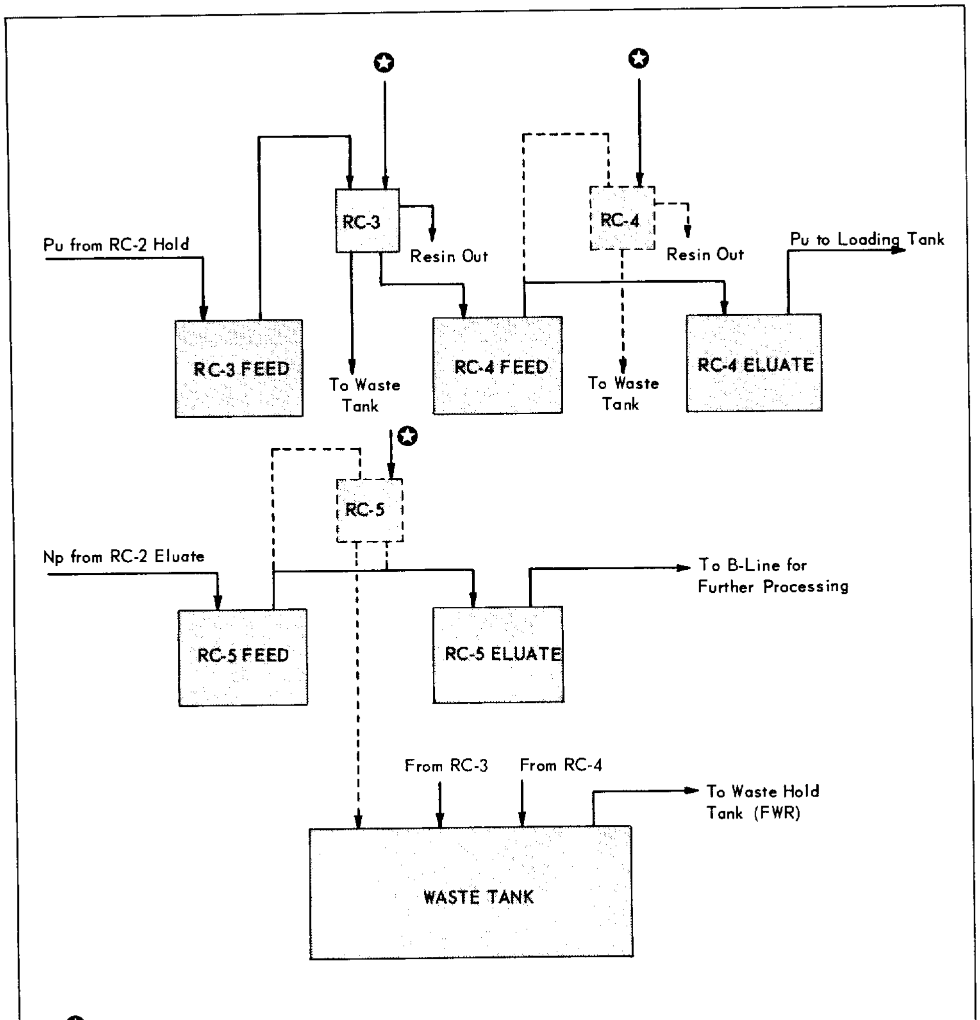


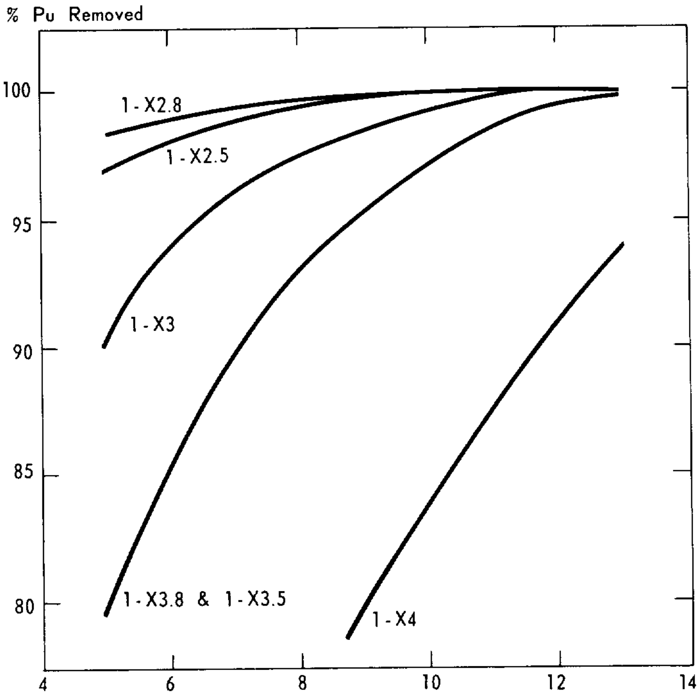
FIGURE 4. FRAME I-H AND FWR



✪ Reconditioning, Reconditioning Flush, DF Wash, and Elution

FIGURE 5. FRAME II-H

—— Aqueous Streams
- - - - - Organic Streams



5.7M Nitric Acid, 0.05M Hydrazine 0.05M Ferrous Sulfamate

FIGURE 6. LABORATORY PARTITIONING WITH DOWEX RESIN

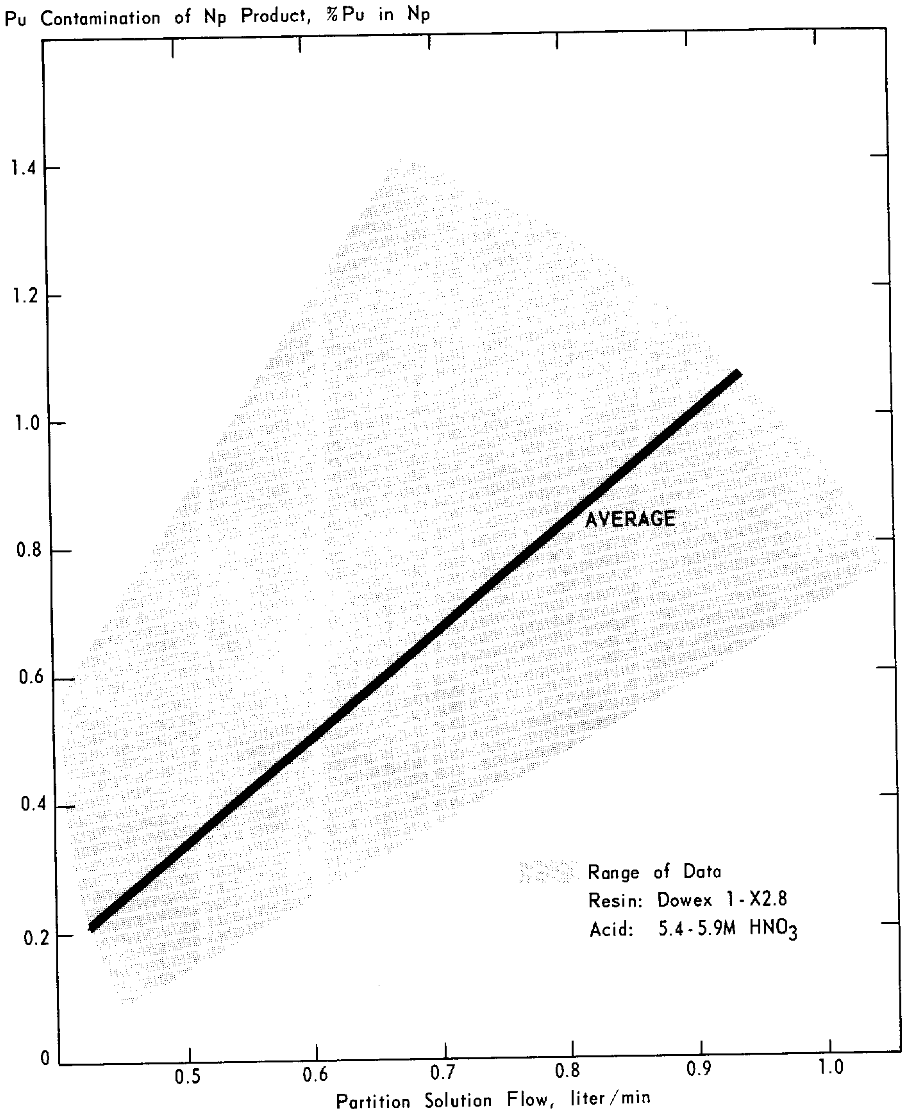


FIGURE 7. EFFECT OF FLOW ON PARTITION