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SOLVENT EXTRACTION OF ENRICHED URANIUM FUELS AT THE SAVANNAH RIVER PLANT*

MASTER

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ABSTRACT

A modified Purex process was developed in the Savannah River Laboratory and is now used routinely at the Savannah River Plant to recover enriched uranium (1 to >90% ^{235}U) and either plutonium or neptunium from irradiated fuel. Performance is improved over the previous process except for zirconium decontamination. Laboratory tests at lower scrub acid concentrations show that the zirconium decontamination can be improved. Modifications which further improve uranium-neptunium partitioning have been tested in the laboratory, but not in the plant.

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INTRODUCTION

The Savannah River Plant (SRP) recovers actinides from a wide variety of irradiated fuels containing ^{235}U enrichments of 1 to >90 isotopic percent. The fuels come from a variety of sources, including the SRP reactors, government test reactors, the nuclear ship Savannah, and the Piqua power reactor. These fuels, clad with aluminum, stainless steel, or nichrome, could be chemically or electrolytically dissolved and processed in existing equipment; however, for the lower ^{235}U enrichments, the previously used solvent extraction flowsheet would not provide adequate throughput. A more flexible flowsheet has been developed that enables efficient and rapid processing of fuels of a wide range of ^{235}U enrichments. With this modified flowsheet, the uranium concentration in the process may be varied to provide maximum throughput consistent with ^{235}U concentration limits required for nuclear safety.

Solvent extraction separations at SRP are conducted in multistage mixer-settlers through which organic and aqueous streams flow countercurrently. The uranium and either neptunium or plutonium are processed through two successive solvent extraction cycles, and the recovered products are also separated from fission products and other impurities (decontaminated).

The flowsheet used previously (3.5 vol % TBP extractant) had limited capacity for uranium and required 1.2M $Al(NO_3)_3$ as a scrub stream to prevent high losses of uranium to the aqueous raffinate.[1] The modified flowsheet utilizes 7.5 volume percent tri-n-butyl phosphate (7.5 vol % TBP) in the nitrate-nitric acid system. The flowsheet increases the flexibility of the separations equipment and lowers the amount of waste generated by about 20%. The development and use of this improved solvent extraction process are discussed in this paper.

PROCESS DESCRIPTION

In the first cycle (Figure 1), uranium and neptunium (or plutonium) are coextracted into the organic phase in the first mixer-settler (1A) and partitioned (separated) in the second mixer-settler (1B). Uranium is stripped in the third mixer-settler (1C). The organic extractant consists of 7.5 vol % TBP in n-paraffin hydrocarbon (C-12 to C-16).

Extractant flow is increased as the uranium concentration in the feed increases (100 grams per liter maximum) so that uranium does not reflux at any point to a concentration higher than that of the feed. The nominal nitric acid concentration of the feed and scrub streams in the first mixer-settler is 4.0M. Aluminum nitrate can be substituted for nitric acid in the feed, provided that sufficient acidity (0.5M) is maintained to prevent polymerization of plutonium.

When recovery of neptunium from the feed solution is desired, a reducing reagent (ferrous sulfamate) is added in the first cycle of solvent extraction to maintain plutonium in the nonextractable Pu(III) valence state and neptunium in the readily extractable Np(IV) state. Thus, the plutonium present will remain with the fission products in the waste streams, and neptunium will be extracted.

When recovery of plutonium is desired and the neptunium may be discarded, oxidizing conditions are maintained in the first cycle. Plutonium is extracted in the first mixer-settler as Pu(IV) and separated from uranium in the second mixer-settler by reducing Pu(IV) to Pu(III). Neptunium, if present in the feed, remains as Np(V) and is rejected to the aqueous waste stream.

In the second uranium cycle with two mixer-settlers, uranium is extracted into the organic phase and scrubbed with nitric acid in the first mixer-settler (1D). Uranium is stripped in the second mixer-settler (1E).

URANIUM RECOVERY

Using miniature mixer-settlers, laboratory tests were made with solutions simulating dissolved stainless steel clad fuels to determine the best solvent concentration for the uranium concentrations of interest. Boric acid, used in the plant to maintain nuclear safety in dissolving some fuels, was added to the test solutions. Test results for the first mixer-settler (Table 1) showed uranium losses to be high with 3.5 and 5.0 vol % TBP, although a very high extractant flow rate with 5.0 vol % TBP yields a marginally acceptable loss. Uranium losses with 7.5 vol % TBP were low at fairly low extractant flow rates. Thus, 7.5 vol % TBP allows greater flexibility for processing higher uranium concentrations.

The plant feed solution from Piqua fuel (last test in Table 1) contained 5×10^9 dis/(min)(ml) from gamma-emitting fission products and 4.2×10^7 alpha dis/(min)(ml) from plutonium. The apparent uranium loss was higher than expected. Piqua fuel contains molybdenum which interferes with colorimetric uranium analyses; therefore, uranium recovery in the product is a more accurate indicator of loss than uranium in the waste. The losses observed during plant operation with this solution were much lower (0.01% uranium loss by isotope dilution analyses).

PLUTONIUM RECOVERY AND PARTITIONING

With the modified flowsheet, plutonium recovery was expected to be equivalent to uranium recovery because the distribution coefficients of plutonium and uranium are similar. Three tests were made to determine plutonium recovery (Table 2). Except for the test with the plant solution obtained from dissolution of Piqua power reactor fuel, plutonium mass balances were excellent. Plutonium losses were low in all tests. With plant feed, neptunium split approximately 85 to 15% between product and waste, respectively, in the first mixer settler, and 35% of the neptunium was in the uranium product after partitioning. Similar neptunium behavior has been observed in plant operation and is due to the high nitric acid concentrations in plant feeds (4.5 - 6.0M). The oxidation of extractable Np(IV) to inextractable Np(V) is inversely dependent on the fourth power of the hydrogen ion activity.[2] Thus, Np(IV) is stabilized and can be extracted along with uranium and plutonium.

The partitioning mixer-settler must be operated to separate uranium and either neptunium or plutonium with no reflux of uranium above the concentration in the feed to the first mixer-settler. Plutonium is partitioned by reducing extractable Pu(IV) to inextractable Pu(III) with ferrous sulfamate. The acid concentration can be high enough to prevent uranium reflux because Pu(III) is essentially inextractable.

Plutonium partitioning was tested in the miniature mixer-settlers (Table 3), and results were satisfactory as long as sufficient ferrous sulfamate was present. Nitric acid reacts slowly with ferrous sulfamate to destroy sulfamate and oxidize ferrous to ferric.[3] Thus, at the higher acid concentration used in the tests with plant solutions, more ferrous sulfamate was needed to ensure plutonium reduction. The higher acid was required to prevent uranium reflux which occurred in the first two tests with low acid.

During plant operation with the low enrichment uranium flowsheet, uranium losses have averaged <0.05% to waste and 0.01% to plutonium product stream. Plutonium losses have averaged <0.05% to the waste and uranium streams. Operation at higher than predicted extractant flows have been necessary to obtain these low product losses. Consequently, the ultimate uranium capacity of the process is limited by the equipment in the plant.

NEPTUNIUM RECOVERY AND PARTITIONING

With highly enriched uranium feed solutions, neptunium is recovered by extraction along with uranium, and plutonium is rejected to the waste. Neptunium and uranium are then partitioned in the second mixer-settler by adjusting the acid concentration of the strip stream and flows of the aqueous and organic streams. Tests were made to investigate recovery and partitioning. Neptunium losses were low (<0.3%) in all the tests (Table 4). The first five tests were made with simulated feeds, and ferrous sulfamate was added to the feed immediately before each test. The sixth test was made with plant feed in which ferrous sulfamate was added 3 to 4 days before the test. Bibler[4] has shown that the ferrous ion is rapidly oxidized by radiolytically produced hydroxyl radicals. The ferric ion, thus produced, hindered reduction of neptunium in the mixer-settler and resulted in higher than expected neptunium losses.[3] The presence of ferric ion also results in incomplete reduction of plutonium[3] as shown by the presence of only 81% of the plutonium in the waste. The apparent high uranium losses are probably due to inaccurate colorimetric analysis at these very low uranium concentrations. Uranium extracts better than neptunium; therefore, the actual uranium loss is believed to be less than the neptunium loss in all cases.

For neptunium partitioning, the flows of the various streams and the acid concentration of the strip must be carefully controlled to maintain the neptunium distribution coefficient as low as possible while maintaining the uranium distribution coefficient close to one. This condition necessarily requires operation with some uranium reflux to ensure low neptunium in the uranium product. The conditions necessary to partition neptunium and prevent reflux of uranium were calculated and confirmed by laboratory tests (Table 5). The first three tests demonstrate the effects of the flow ratios on partitioning. To effectively strip neptunium, a high strip-to-extractant flow ratio is necessary. However, a high scrub-to-strip flow ratio is necessary to re-extract uranium which is stripped along with neptunium. The fourth test illustrates the combined effects of high flow ratios. Uranium reflux did not occur in any of these tests.

In initial plant operation with the new flowsheet, valence adjustment of plutonium and neptunium with ferrous sulfamate was ineffective. Rejection of neptunium to the aqueous waste stream (1AW) ranged from 5% to 75%, while more than 90% of plutonium present was extracted, rather than rejected, in the first cycle.

Laboratory studies demonstrated that ferrous sulfamate, added to both the aqueous feed solution and the aqueous scrub stream in the first stage (1AS), was largely destroyed during the relatively long time between preparation and use of these solutions.[3] Radiolysis destroyed the ferrous ion in the feed in a few hours, and nitric acid oxidized much of the ferrous ion in the scrub solution. To achieve the desired neptunium-plutonium separation in plant operation, the process flowsheet was modified as follows: ferrous sulfamate is added to the 1AS stream immediately before it enters the mixer-settler, and ferrous sulfamate is not added to the feed solution where it would be destroyed by radiolysis.

During subsequent routine plant operation, uranium losses have averaged <0.05% to waste and 0.01% to the neptunium product stream. These losses are within the range of expected losses based on laboratory tests and computer calculations. Neptunium losses have averaged <1% to waste. The losses to waste are considerably higher than those observed in laboratory tests, but better than the 2% obtained with the 3.5 vol % TBP flowsheet.

Neptunium losses to the uranium stream in the partitioning step have averaged 4%, but these losses are lower than those observed in the 3.5 vol % TBP process. Neptunium in the uranium stream is sent to the waste in the second uranium cycle (1DW) and is recovered in the second neptunium cycle. The large volumes and impurities in the recycled waste increase neptunium loss and decrease decontamination in the second neptunium cycle.

If partitioning could be improved so that only 1 to 2% of the neptunium remains with the uranium, neptunium would not have to be recovered from the second uranium cycle waste, and second neptunium cycle performance would be improved. Neptunium and uranium are partitioned by adjusting the acid concentration in the 1B mixer-settler so that the extraction factor of uranium is greater than one while the extraction factor of neptunium is less than one. Computer calculations were made to examine potential improvement in partitioning obtained by lowering the acid concentration in the strip solution (1BX). The calculations showed and tests confirmed that changes which decrease neptunium losses to the uranium stream increase uranium losses to the neptunium stream.

An alternative method of improving partitioning is to (1) lower the acidity of the strip solution (1BX) to ensure stripping neptunium, and (2) inject a high acid stream in the 1B scrub section to recover any uranium stripped with the neptunium. The feasibility of a low acid strip stream with a high acid side stream was tested (Figure 2). The tests (Table 6) showed that neptunium losses to the uranium stream can be reduced below the current nominal 4%. Computer calculations were made with a high acid stream in Stage 9 at a flow of 0.10 relative to the feed and a strip acid concentration of 1.0M. The results of the calculations showed that partitioning could be improved with side stream acidities of 4 to 8M. The plant will test this flowsheet change in the near future.

DECONTAMINATION

Decontamination of uranium, plutonium, and neptunium from fission products and inorganic ions is of prime importance to the separations process. Decontamination of the uranium product stream from inorganic ions (except for aluminum, molybdenum, and mercury) was better than decontamination of the neptunium or plutonium stream (Table 7). Decontamination is expected to be higher for the uranium stream because the partitioning mixer-settler acts as additional scrub stages for the uranium stream.

Molybdenum decontamination was studied extensively because Piqua fuel solutions contained 0.01M molybdenum. Molybdenum was moderately extractable, but did not easily strip into acid solutions. Decontamination was improved by addition of 0.05M phosphate ion and 0.5M aluminum ion which reduced molybdenum extractability by formation of heteropolymolybdates.

Fission product decontamination was measured with plant solutions with low and high ^{235}U enrichments. The fission product activities of these solutions and the decontamination factors are shown in Table 8. At higher activities (high ^{235}U enrichments) the fission product decontamination is better. Analyses of the product solutions for inorganic ions and fission products resulted in concentrations less than or equal to the detectability limit of the method used for analysis. Therefore, as fission product concentration in the feed increased, the decontamination increased because the product concentration remained unchanged.

In plant operation, the zirconium decontamination factor for the neptunium product during highly-enriched uranium processing with 3.5% TBP varied from 20 to 2600 and averaged ∿600.

Since 1974 when processing with 7.5 vol % TBP began, the zirconium decontamination factor for neptunium has varied from 10 to 450 and averaged 90. The zirconium distribution coefficient ($D_{o/a} = [Zr]_o/[Zr]_a$) increases as nitric acid concentration increases. Therefore, the zirconium decontamination should increase if the scrub acidity in the 1A mixer-settler is decreased from the present 4.0M HNO_3. Several laboratory tests were made to determine the minimum feasible scrub acid concentration (Table 9). If neptunium were not to be recovered, the scrub acid could be the same as in the second uranium cycle, i.e., a minimum of 0.8M HNO_3. Recovery of neptunium, however, prevents use of very low acid scrubs because the Np(IV) distribution coefficient is much lower than uranium. The tests (Table 9) show that neptunium loss is unaffected down to 2.0M acid in the scrub as long as the extractant flow rate is increased when the scrub acidity is below 2.5M. Uranium losses at 2.0M acid should not be so high (0.46%) since the uranium distribution coefficient is greater than 1.5 in the scrub section. In these tests, the total uranium inventory increased, but no change was observed in the maximum uranium concentration in the 1A bank. The maximum uranium concentration in the bank will not change so long as the uranium distribution coefficient in the scrub section is greater than one.

Two tests were made using tracer zirconium to illustrate the improvement in decontamination obtained by lowering the scrub acid concentration. The zirconium decontamination factor increased from ∿200 to >1000 when the scrub acidity was decreased from 4.0 to 2.0M.

SECOND URANIUM CYCLE

Increasing the concentration of uranium in the feed to the second uranium cycle made it necessary to change the scrub acid concentration to prevent reflux of uranium above the feed concentration. For less than 5 g/l uranium concentrations in the feed, a minimum of 0.8M HNO_3 in the scrub was adequate. Several tests were made with high uranium concentrations in the feed, and the results were checked with computer calculations. The first four tests showed excessive reflux of uranium (Table 10). The last two tests were typical of normal operation except for low acid in the feed to one test which resulted in high uranium loss to the waste stream. Plutonium was added to the feed of the last test to determine the decontamination factor. The decontamination factor of 25 found in this test was not sufficient to ensure acceptable uranium product.

Uranium losses to waste have been less than 0.01% during plant operation. With ferrous sulfamate added in the scrub section of the mixer-settler, plutonium contamination of the uranium product has been about 1000 alpha disintegrations per gram of uranium. Fission product decontamination has been sufficient to give an acceptable product.

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TABLE 1

URANIUM RECOVERY

Feed 25 g/l U, 4M HNO$_3$, 4 g/l B, 0.001M Al, 0.15M Fe,
0.16M Ni, 0.041M Cr, 0.004M Mn

Scrub 4M HNO$_3$

Temperature 45°C

Extractant, % TBP	Flow Ratio Extractant-To-Feed	 Scrub-To-Extractant	Uranium, % In Product	 In Waste
3.5	3.0	0.17	40.1	53.7
3.5	5.0	0.10	78.0	24.4
5.0	3.0	0.17	94.6	5.4
5.0	5.0	0.10	99.2	0.4
7.5	2.0	0.17	96.8	0.54
7.5	3.0	0.17	99.9	0.06
7.5[a]	4.0	0.15	99.6	0.96

---

[a] Plant feed solution 40.2 g/l U.

TABLE 2

PLUTONIUM RECOVERY

Feed 25 g/l U, 4-5M HNO$_3$, 4 g/l B, 0.001M Al, 0.15M Fe,
0.016M Ni, 0.041M Cr, 0.004M Mn

Scrub 4M HNO$_3$

Extractant 7.5% TBP

Temperature 45°C

Feed Pu, dis/(min)(ml)	Flow Ratio Extractant-To-Feed	 Scrub-To-Extractant	Plutonium, % In Product	 In Waste
1.96×10^7	2	0.25	100	0.14
2.30×10^7	2	0.17	100	0.04
4.2×10^7[a]	4	0.15	92	0.03

---

[a] Plant feed solution 40.2 g/l U.

TABLE 3

PLUTONIUM PARTITIONING

Temperature 30-35°C

Flow Ratio			Strip, M		Pu Product, %		U Product, %	
Extractant-To-Feed	Strip-To-Extractant	Scrub-To-Strip	HNO_3	FS	U	Pu	U	Pu
2.0	0.225	3.33	0.90	0.02	0.10	99.9	100.0	0.14
2.0	0.225	3.33	0.84	0.03	0.16	100.0	97.9	0.01
4.0[a]	0.20	3.0	1.99	0.075	<0.02	82.8	99.6	0.27
4.0[a]	0.20	3.0	1.99	0.075	0.14	90.7	100.0	0.10

---

[a] Plant feed solution 40.2 g/l U.

TABLE 4

NEPTUNIUM RECOVERY

Scrub 4.0M HNO_3, 0.02-0.04M FS

Extractant 7.5% TBP

Feed 6 g/l U, 0.1 g/l Np, 1.1M $Al(NO_3)_3$,
0.7M HNO_3, 0.02M FS

Extractant-to-Feed Flow Ratio	Waste, %		
	U	Np	Pu
1.3	0.92	0.28	~100
1.5	0.29	0.12	~100
1.5	0.20	0.09	~100
2.0	0.43	0.08	>99
2.0	0.17	0.03	>99
2.5[a]	0.09	0.24	81

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[a] Run made with plant feed 5.45 g/l U, 2.08 × 10^8 dis/(min)(ml) Pu, 6 × 10^3 dis/(min)(ml) Np, 1.47M Al, 1.93M HNO_3, and 8.36 × 10^{10} dis/(min)(ml) gross gamma activity.

TABLE 5

NEPTUNIUM PARTITIONING

Extractant and Scrub 7.5% TBP

Strip-to-Extractant Flow Ratio	Strip[a] HNO_3, M	Scrub-to-Strip Flow Ratio	Neptunium Product, %		Uranium Product, %	
			Np	U	Np	U
0.20	1.59	3.0	22.4	0.25	77.5	79.5
0.38	1.59	2.4	93.1	6.1	1.9	95.0
0.46	1.59	2.0	98.0	13.8	0.09	133.6
0.45	1.50	3.0	103.4	0.6	0.6	106.8

---

[a] Strip also contained 0.02M ferrous sulfamate.

TABLE 6

NEPTUNIUM-URANIUM PARTITIONING WITH A LOW ACID STRIP STREAM AND A HIGH ACID SIDE STREAM

1BX' - 8.2M HNO_3 added at Stage 9

1AS Acid, M	1BX Acid, M	Flow Ratios			Np[a] in U, %	U Reflux Ratio[b]
		1BX'/1BX	1BX/1AX	1BS/1BX+1BX'		
2.0	1.0	0.13	0.36	2.00	1.8	0.43
2.0	1.0	0.11	0.31	2.50	0.48	0.35

---

[a] Current nominal % Np in U is 4%.

[b] Ratio of maximum uranium concentration in any stage to uranium concentration in 1AF.

TABLE 7

INORGANIC ION DECONTAMINATION

Ion or Element	Concentration, M	Feed-to-Product Concentration Ratio	
		U Product	Np or Pu Product
Al	0.1	>600	>12,000
B	0.4	>400	320
Fe	0.15	>1,000	-
Ni	0.016	>1,100	>350
Cr	0.041	>1,000	>1,000
Mn	0.004	>500	>500
PO_4^{3-}	0.066	-	5,000
Hg	0.003	>70	>350
Mo	0.01	80[a]	>1,400

---

[a] Uranium product decontamination increased to >300 by addition of PO_4^{3-} and Al^{3+} to the feed.

TABLE 8

FISSION PRODUCT DECONTAMINATION

Isotope	Feed Activity, dis/(min)(ml)		Feed-to-Product Activity Ratio			
			U Product		Np or Pu Product	
	Low Enriched	High Enriched	Low	High	Low	High
^{95}Zr	-	2.81 × 10^{9}	-	>10^{4}	-	10^{4}
^{95}Nb	-	1.72 × 10^{9}	-	>10^{5}	-	>10^{4}
^{103}Ru	-	5.98 × 10^{9}	-	10^{5}	-	10^{5}
^{106}Ru	5.74 × 10^{8}	5.14 × 10^{9}	3600	>10^{5}	10^{4}	>10^{5}
^{134}Cs	3.41 × 10^{8}	2.34 × 10^{9}	>10^{4}	>10^{6}	>10^{4}	>10^{6}
^{137}Cs	2.03 × 10^{9}	6.02 × 10^{9}	>10^{5}	>10^{5}	>3 × 10^{4}	10^{5}
^{144}Ce	2.35 × 10^{9}	5.51 × 10^{10}	8600	>10^{5}	4900	10^{5}

TABLE 9

EFFECT OF SCRUB ACID CONCENTRATION ON URANIUM AND NEPTUNIUM RECOVERY AND DECONTAMINATION

HNO_3 in Scrub, M	Extractant Flow Relative to Feed	Np Loss to 1AW, %	U Loss to 1AW, %	U Reflux Ratio[a]	Zr, DF[b]
4.0	1.6	<0.66	<0.10	0.68	200
2.5	1.6	0.15	<0.09	0.70	-
2.0	2.1	0.22	0.46	0.67	>1000

---

[a] Ratio of maximum uranium concentration in any stage to uranium concentration in feed.

[b] Decontamination factor = activity in 1AF/activity in 1AP.

TABLE 10

MINI-MIXER SETTLER TESTS OF SECOND URANIUM CYCLE

Extractant 7.5% TBP
Temperature 45°C

Feed		Extractant-to-Feed Flow Ratio	Scrub HNO_3, M	Scrub-to-Extractant Flow Ratio	% U		U Reflux Ratio[a]
U, g/L	HNO_3, M				Product	Waste	
15.25	4.18	2.00	0.89	0.145	100	<.08	1.56
29.9	4.08	1.85	1.73	0.17	100	<.04	1.37
29.7	4.19	2.50	1.04	0.10	98.1	.03	1.87
29.7	4.19	3.00	1.04	0.14	97.0	<.05	1.30
17.8	3.46	3.00	1.2	0.12	99.7	.29	0.73
21.34	3.87	3.00	1.2	0.14	100	<.08	0.43

---

[a] Ratio of maximum uranium concentration in any stage to uranium concentration in feed.

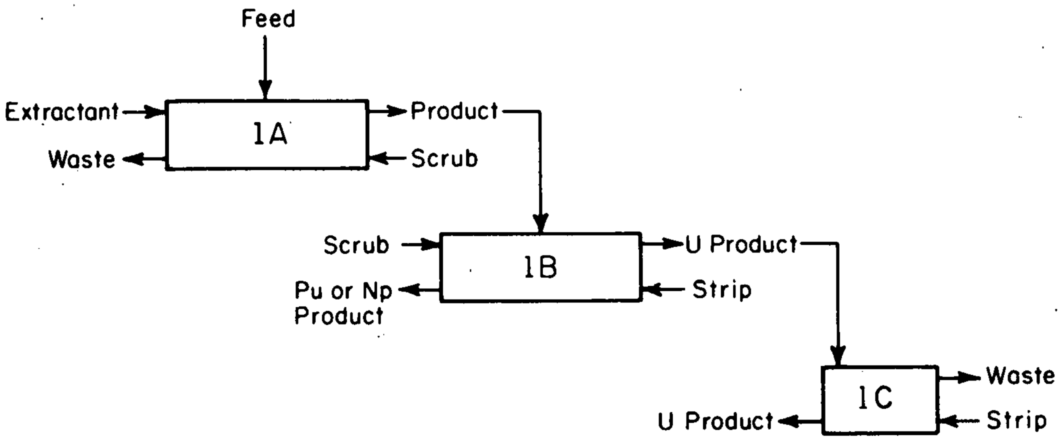

FIGURE 1. Present First Cycle Operation

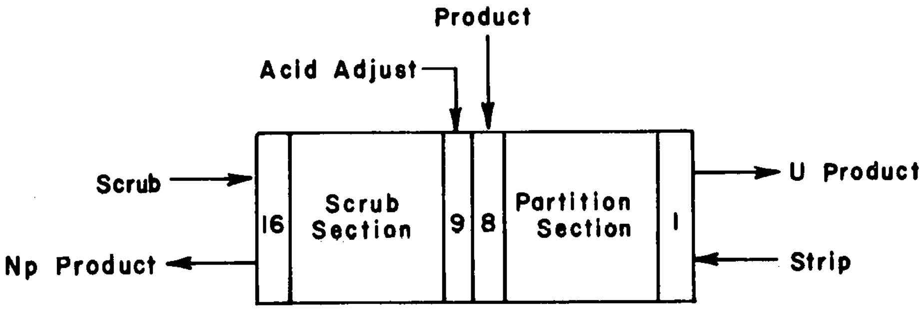

FIGURE 2. Proposed Partitioning Bank (1B) Operation