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## PUREX PROCESS PERFORMANCE WITH SHORT-RESIDENCE CONTACTORS

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#### ABSTRACT

Recovery and purification were satisfactory in miniature-pilot-plant tests of the Purex first cycle with the solvent residing four seconds in each extraction stage. Decontamination factors for both zirconiumniobium and ruthenium were greater than 10,000 with negligible crosscontamination of uranium and plutonium. The effects of the following on decontamination and plutonium partitioning were determined: irradiation level of the fuel, operating temperature, solvent saturation, accumulated exposure of the solvent, nitrous acid, and ferrous sulfamate versus U(IV) as a reductant for Pu(IV).

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## PUREX PROCESS PERFORMANCE WITH SHORT-RESIDENCE CONTACTORS

#### INTRODUCTION

The flexibility of existing solvent extraction facilities for processing irradiated nuclear fuels can be increased by short-residence contactors (contactors in which process solutions reside only a short time). Since such contactors have high capacity with low holdup, they can be used to process both irradiated natural uranium, where high processing rates are normally required, and plutonium or enriched uranium, where close control of the configuration and inventory of fissionable materials are important. An additional feature of short-residence contactors is a reduced exposure of organic solvent to chemical and radiolytic degradation. With mixer-settlers, pulse columns, and other conventional contactors, the residence time of the organic solvent is one to ten minutes per stage, and degradation necessitates purification of the solvent after each pass through the process. Since purification is only partially effective, the accumulated effects of exposure eventually become excessive, and the solvent must be replaced.

Practical equipment for short-residence extraction recently became available when the centrifugal contactor was developed.<sup>(1)</sup> This contactor is a mixer-settler that uses centrifugal force to promote phase separation and thereby reduces the holdup time or volume in the settling section. Many sizes of centrifugal contactors can be made. The miniature centrifugal contactor<sup>(2)</sup> in which the holdup is less than 10 ml per stage and which is operable with residence times of the organic solvent as low as two seconds per stage is an example.

The advantages of short-residence contactors can be realized only if the chemical reactions for the extraction process proceed to completion during the shorter time that the organic and aqueous phases are in contact. In preliminary investigations<sup>(2)</sup> with miniature centrifugal contactors, uranium was extracted efficiently by diluted tri-n-butyl phosphate (TBP) at residence times as low as two seconds per extraction stage. Other cations that are weakly complexed by reactions of the type

 $M^{n+} + n NO_3 + (6 - n) TBP \rightleftharpoons M(NO_3)_n (TBP)_{(6-n)}$ 

will probably be extracted efficiently. Limitations on fission

product extraction and scrubbing rates have not been reported, and it had not been demonstrated that plutonium can be partitioned from uranium at residence times as low as four seconds per stage.

#### SUMMARY

The first solvent extraction cycle of the Purex process was satisfactorily adapted to short-residence contactors. Process performance with an organic phase residence time of four seconds per stage was equivalent to performance normally obtained with conventional mixer-settlers. With normal levels of saturation in the 1A bank, purification of uranium and plutonium was excellent (gamma decontamination factor greater than  $10^4$ ), and crosscontamination of uranium and plutonium was negligible. The tests included a demonstration of satisfactory performance with solvent that had been exposed to typical levels of radiation for about 300 hours.

Two necessary process modifications were:

- Elevation of temperatures in the 1A bank. Decontamination from ruthenium was low (DF = 600) at normal operating temperatures ( $35-40^{\circ}C$ ), but was excellent (DF >  $10^{4}$ ) at  $55-65^{\circ}C$ .
- Destruction of excess nitrous acid in the feed solution (1AF). Without destruction of excess nitrous acid several partitioning stages of the 1B bank were needed to destroy nitrite with hydrazine, and insufficient stages remained for complete partitioning of uranium and plutonium.

The following characteristics of the process were also observed:

- Zr-Nb decontamination increased almost linearly with increases in the irradiation level of the fuel, indicating the possible presence of a "carrier" that carried a fixed amount of activity through the process.
- Excessive dibutyl phosphate did not form at elevated temperatures.
- Maximum solvent saturation at the feed stage of the IA bank gave the highest decontamination, as it does with conventional mixer-settlers.
- Decontamination with solvent that had been exposed 300 hours to radiolytic degradation in conventional mixer-settlers was satisfactory; therefore, longterm recirculation of solvent is possible with short-residence contactors.
- Ferrous sulfamate and hydrazine-stabilized uranous nitrate were equally effective reducing reagents for Pu(IV) in the partitioning contactor.

#### DISCUSSION

#### TESTS IN MINIATURE CENTRIFUGAL CONTACTORS

Feed solutions were prepared by dissolving irradiated natural uranium in 10-13M  $HNO_3$ , clarifying the solution with gelatin or  $MnO_2$  precipitation and filtration, adjusting the composition, and adjusting the plutonium with NaNO<sub>2</sub>.

The adjusted feed solutions were processed through miniature centrifugal contactors, which are described in Appendix A. The flowsheet and operating parameters are given in Figure 1 and Tables I and II. The process monitoring and control techniques are described in Appendix B. A summary of process performance is given in Table III.

#### TABLE I

#### Solvent Extraction Flowsheet

Bank	Temp, °C	Impeller Speed, rpm	Bowl Speed, rpm	Stream	Entry Stage	Flo Relative	w cc/min	Composition
lA	Varia- ble(a)	4000	7000	laf	<b>A-</b> 8	100 <sup>(b)</sup>	11(b)	Nominal: 1.5M UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> , 1M HNO <sub>3</sub>
				lAX	A-16	374(c)	41.2	30 vol % TBP - "Adakane 12"(d)
				1AS	A-1	67	7.4	3M HNO3
1B	40 ±5	4000	7000	1AP	в-8	-	-	-
				1BX	B-1	50	5.5	Variable <sup>(a)</sup>
				1BS	в <b>-</b> 16	160	17.6	30 vol % TBP - "Adakane 12"
				1BX'	B <b>-</b> 9	Varia- ble(a)	-	Variable(a)
1C	35 ±3	4000	7000	1BU	C-16	-	-	-
				lCX	C-1	710	78	0.025M HNO3

(a) Variations are given in Table II.

(b) Flow when lAF composition was nominal; varied (see Table II) when composition was not nominal.

(c) Adjusted to control saturation; see Table II.

(d) Trademark of Archer-Daniels-Midland Co.





### TABLE II: Variations in Solvent Extraction Flowsheet

(Major variations are inside boxes.)

	Activity Level			1A Bank Variables					
	Irradiation	Cooling	·····	lAF	Composi	tion	1AF		
	Level	Time	U,	HNO <sub>3</sub> ,	HNO2,	$\gamma$ Activity,	Flow		
Run	<u>(relative)</u>	<u>(relative)</u>	M	<u>M</u>	<u> </u>	<u>c/(min)(ml)</u>	(relative		
1	1.0	1.33	1.25	1.1	0.05	2.1x10 <sup>8</sup>	120		
2	3.3	1.33	1.25	1.1	0.05	6.9x10 <sup>8</sup>	120		
3	3.7	1.33	1.37	1.4	0.07	1.3x10 <sup>9</sup>	110		
4	3.7	1.33	1.37	1.4	0.07	1.3x10 <sup>9</sup>	110		
5	3.7	1.33	1.37	1.4	0.07	1.3x10 <sup>9</sup>	110		
6	7.9	1.0	1.63	1.0	<sub>O</sub> (a)	5.3x10 <sup>9</sup>	92		
7	7.9	1.0	1.63	1.0	0(a)	5.3x10 <sup>9</sup>	92		
8	7.9	1.0	1.63	1.0	<sub>O</sub> (a)	5.3x10 <sup>9</sup>	92		
9	7.9	1.0	1.63	1.0	<sub>O</sub> (a)	5.3x10 <sup>9</sup>	92		
10	-	-	1.56	1.3	0	0	100		
11		æ	1.56	1.3	0	0	100		
12	-	-	1.56	1.3		0	100		

(a) 0.01M NaNO<sub>2</sub> added and then heated to eliminate excess. (b)  $Fe(SO_3NH_2)_2$ .

TABLE	TTT •	Summary	of	Process	Performance
LADDE	****	Common y	OT.	11000000	I CI I OI Manoo

	Ura	nium and F	lutonium L	osses				
	% to	lAW	% Pu	% U	lAF to lAP			
Run	U	Pu	to lCU	to 1BP	Gross $\gamma$	Zr-Nb	Ru	
1	-	-	0.8	9x10 <sup>…4</sup>	1.1x10 <sup>3</sup>	1.3x10 <sup>3</sup>	4.2x10	
2	2x10 <sup>-3</sup>	5x10-2	6.3	9x10-4	2.1x10 <sup>3</sup>	3.6x10 <sup>3</sup>	4.7x10	
3	7x10-4	3.6x10-3	3.6	4.2x10-3	2.7x10 <sup>3</sup>	4.6x10 <sup>3</sup>	5.1x10	
4	6.6x10-4	3.7x10-3	3.0	3.9x10-3	1.0x10 <sup>4</sup>	1.0x10 <sup>4</sup>	7.3x10	
5	5.8	3x10 <del>-1</del>	2.6	3.4x10-3	2.3x104	2.1x10 <sup>4</sup>	2.7x10	
6	1.3x10-2	1.8x10-2	0.24	3.7x10-4	1.4x104	1.6x104	7.1x10 <sup>.</sup>	
7	1.1x10-3	1.0x10 <sup>-2</sup>	0.33	3.7x10-4	3.0x10 <sup>3</sup>	2.8x10 <sup>3</sup>	3.8x10	
8	4.9x10-1	4.5	5.9x10 <sup>-2</sup>	2.2x10 <sup>-4</sup>	6.6x10 <sup>3</sup>	5.6x10 <sup>3</sup>	2.8x10	
9	7.7x10-3	1.0x10-2	0.28	6.9x10 <sup>-5</sup>	1.4x10 <sup>3</sup>	1.3x10 <sup>3</sup>	1.2x10	
10	-	<b></b>	5x10-2	5.7x10-2(a)		-	649	
11	840		4x10-2	9.9x10-6		-	1429	
12	-	-	6x10-2	4.1x10-6	92.9	-		

(a) Low HNO3 in 1BX'.

		Solvent	1B Bank Variables						
	No. of	Quality,	1BX Composit	ion	1BX'	Composi	tion	1BX'	
Ъmр,	Saturated	hours of	N <sub>2</sub> H <sub>4</sub> ,	HNO <sub>3</sub> ,	U(IV),	N₂H₄,	HNO3,	Flow	
°C	<u>Stages</u>	exposure	<u> </u>	<u>M</u>	M		M	Ratio	
38 ±1	2	~0	0.022	0.18	0.08	0.11	1.8	20	
38 ±1	2	~0	0.022	0.18	0.08	0.11	1.8	20	
39 ±2	2	$\sim 0$	0.025	0,29	0.065	0.10	1.7	20	
5 ±2	2-3	~0	0.025	0.29	0.067	0.10	2.3	20	
5 ±2	3	~0	0.025	0.29	0.067	0.10	2.3	20	
o5 ±3	5	~0	0.12	0.29	0.20	0.10	1.5	20	
5 ±3	0	~0	0.12	0.29	0.20	0.10	1.5	20	
5 ±3	5	300	0.12	0.29	0.20	0.10	1.5	20	
5 ±3	0	300	0.12	0.29	0.20	0.10	1.5	20	
30		~0	0.14	0.10	0.09	0.16	0.4	10	
<u></u> SO	-	~0	$(0.08 \text{M FeSA}^{(b)})$	-				0	
		~0	$(0.14M \text{ HNO}_3)$						
30	-		$\begin{pmatrix} 0.32M \text{ FeSA}^{(0)} \\ 0.14M \text{ HNO}_3 \end{pmatrix}$	-	-	-		0	

----

Deconta	mination	Factors				Ratio of	' lCU Ac	tivity
1	AF to 1BP	•	1	AF to 1CU	ſ	to ICW Activity		
$ross \gamma$	Zr-Nb	Ru	Gross $\gamma$	Zr-Nb	Ru	Gross $\gamma$	Zr-Nb	Ru
.9x10 <sup>3</sup>	2.2x10 <sup>3</sup>	7.6x10 <sup>2</sup>	2.4x10 <sup>3</sup>	3.3x10 <sup>3</sup>	9.6x10 <sup>2</sup>		-	-
}.9x10 <sup>3</sup>	6.7x10 <sup>3</sup>	7.5x10 <sup>2</sup>	4.7x10 <sup>3</sup>	7.9x10 <sup>3</sup>	9.3x10 <sup>2</sup>	5.5	3.2	15.4
1.2x10 <sup>3</sup>	7.5x10 <sup>3</sup>	7.4x10 <sup>2</sup>	7.3x10 <sup>3</sup>	1.2x10 <sup>4</sup>	1.5x10 <sup>3</sup>	4.0	3.9	4.7
.6x104	1.6x104	1.1x10 <sup>4</sup>	2.7x104	2.8x10 <sup>4</sup>	2.3x10 <sup>4</sup>	1.4	1.5	1.1
3.1x10 <sup>4</sup>	2.6x104	3.7x104	9.6x104	8.2x10 <sup>4</sup>	1.4x10 <sup>5</sup>	0.53	0.56	0.83
.3x10⁴	2.7x104	1.1x10 <sup>4</sup>	6.6x10 <sup>4</sup>	6.5x104	5.1x10 <sup>4</sup>	0.94	1.16	0.54
}.2x10 <sup>3</sup>	3.0x10 <sup>3</sup>	4.1x10 <sup>3</sup>	7.3x10 <sup>4</sup>	6.5x10 <sup>4</sup>	9.8x104	-		-
.3x104	1.2x104	1.4x104	2.8x104	2.4x104	1.1x10 <sup>5</sup>	0.32	0.63	0.024
.9x10 <sup>3</sup>	2.7x10 <sup>3</sup>	2.7x10 <sup>3</sup>	5.3x10 <sup>3</sup>	4.4x10 <sup>3</sup>	1.8x104	0.73	0.97	0.11
	-	-			-		-	
-	-	•••	-		-		-	-
	-	-		***	-		-	-



#### Irradiation Level

Decontamination from Zr-Nb increased almost linearly, as the irradiation level of the uranium increased; decontamination from ruthenium was independent of the irradiation level. The results of three tests (Runs 1, 2, and 3 of Table III) are shown in Figure 2. The linear variation of Zr-Nb decontamination suggests the presence of an organophilic ligand that carries a fixed quantity of Zr-Nb through the process. With larger amounts of Zr-Nb in the feed, this fixed amount of Zr-Nb becomes a smaller fraction of the total, so decontamination is relatively better. This hypoth-



FIG. 2 EFFECT OF URANIUM IRRADIATION LEVEL ON DECONTAMINATION

esis is supported by the distribution of Zr-Nb activity among scrub stages of the 1A bank (shown in Figure 3). The Zr-Nb activity decreased sharply in the two or three scrub stages adjacent to the feed stage, but did not decrease significantly in the remaining scrub stages. These data suggest that treating the feed solution with sufficient inactive zirconium to react with the major portion of the carrier might be an effective way to increase removal of active zirconium from the uranium and plutonium.



FIG. 3 Zr-Nb SCRUBBING PROFILES

#### Temperature

The marked effect of elevated temperatures on ruthenium decontamination (Runs 3, 4, and 5 of Table III) is shown in Figure 4 and is consistent with previous studies with miniature mixer-settlers. Removal of Zr-Nb was also slightly better at elevated temperatures, confirming that DBP does not form to a harmful extent when residence times are short.





Improved ruthenium decontamination at elevated temperatures is attributed to a lower ruthenium distribution coefficient in the scrub section of the 1A bank. Ruthenium scrubbing profiles (Figure 5) show that all increased decontamination at the higher temperatures occurs in the scrub section.

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At  $37^{\circ}$ C, the ratio of the ruthenium concentrations in the organic and aqueous phases was constant at about 0.4 throughout the scrub section. With an organic-to-aqueous flow ratio of 5.7, the extraction factor was greater than unity with the result that no ruthenium was scrubbed from the solvent. At  $65^{\circ}$ C, the ratio of concentrations was about 0.1, the resulting extraction factor was less than unity, and ruthenium scrubbing was equivalent to an additional decontamination factor of almost 300. The ruthenium profiles are not consistent with a chemical-reaction-controlled mechanism; such a mechanism would be indicated by a profile that exhibits an apparent increasing distribution coefficient (concentration ratio) from stages 1 to 7 like the Zr-Nb profiles in Figure 3.

Ruthenium scrubbing was similar to that predicted from extensive studies of the chemistry of trinitrato nitrosylruthenium complexes.<sup>(3)</sup> The chemical reactions for ruthenium extraction are:



Highly Extractable

Poorly Extractable

Experimental and calculated values of distribution coefficients and rates of chemical conversion for conditions in the scrub section of the 1A bank are listed in Table IV.

#### TABLE IV

Properties of Trinitrato Nitrosylruthenium

Temp,	Organic-to Distribution of RuNO(	-Aqueous Coefficient NO <sub>3</sub> ) <sub>3</sub>	Half-time for Conversi of RuNO(NO <sub>3</sub> ) <sub>3</sub> to RuNO(NO <sub>3</sub> ) <sub>2</sub> (a), min			
	Experimental	Calculated	Experimental	Calculated		
37	0.42	0.47	>>0.06	1.45		
55		0.19	-			
65	0.1	0.08	>0.06	0.15		

(a)  $RuNO(NO_3)_2$  has a much lower organic-to-aqueous distribution coefficient than  $RuNO(NO_3)_3$ .

The calculated values for normal temperatures indicate that ruthenium scrubbing is limited by the rate of conversion of trinitrato nitrosylruthenium to dinitrato nitrosylruthenium, but both calculated and experimental values for elevated temperatures indicate the scrubbing at the shorter residence time of the centrifugal contactors is limited by the distribution coefficient of trinitrato nitrosylruthenium. The results also indicate that increased ruthenium decontamination can probably be obtained by adding more scrub stages.

#### Solvent Saturation

Decontamination with two to three saturated stages was as good as that with more than three saturated stages, but decontamination was poor when only the feed stage was saturated. Typical results (Runs 4, 6, and 7 of Table III) of the effect of solvent saturation are given in Figure 6. In most runs, two to three saturated stages (containing a relatively high concentration of uranium) were maintained in the extraction section of the 1A bank.



FIG. 6 EFFECT OF SOLVENT SATURATION ON DECONTAMINATION

The relative independence of decontamination on number of saturated stages, with more than two stages saturated, is consistent with the stage profiles of Figures 3 and 5. These profiles indicate that with highly saturated solvent some decontamination occurs in the saturated stages of the extraction section of the bank. Since the profiles tend to level out near the IAP end of the bank (stage 1), higher decontamination of solvent near the feed stage results in more of the scrub stages being ineffective.

#### Solvent Quality

Decontamination was satisfactory with solvent that had been exposed about 300 hours to acid and radiation fields typical of the 1A bank and an additional 600 hours to acid alone. The results obtained at two feed-to-solvent ratios (Runs 6-9 of Table III) are summarized in Table V.

#### TABLE V

	New Solvent		Exposed S	olvent(b)
Saturation(a)	Low	High	Low	High
Pu loss to lAW, %	0.01	1	0.01	4.5
Gamma decontamination factor of Pu effluent (lAF/lBP)				
Zr-Nb	3000	30,000	2,700	12,000

Ru	4100	65,000	2,700	14,000
~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	,			

(a) Flow of IAX was increased 7% to change the uranium saturation of the IA bank extraction section from low to high.

(b) Exposed 300 hours to acid and radiation fields typical of the IA bank and an additional 600 hours to acid alone.

Although decontamination with new solvent was slightly superior, the decreased decontamination with exposed solvent is insignificant compared with the advantages of extended use of the exposed solvent.

#### FACTORS AFFECTING PLUTONIUM PARTITIONING

#### Nitrous Acid

With no effort to destroy  $HNO_2$  in the feed (1AF) before processing through the 1A bank (Runs 1-5 of Table III), plutonium losses to the uranium stream (1BU) were between 0.8 and 4.3%. The stage profile of plutonium in the 1B bank organic phase, shown in Figure 7, shows that Pu(IV) was not reduced to Pu(III) near the center of the bank. Since some of the stages in the extraction section were then required to destroy the nitrite in the product stream (1AP) of the 1A bank, the number of remaining stages was insufficient to completely reduce the plutonium. With the 1AF heated to decompose residual  $HNO_2$  after the valence adjustment step in feed preparation (Runs 8 and 9 of Table III), plutonium partitioning was satisfactory (less than 0.1% loss to the uranium product stream). Profiles of plutonium in the 1B bank without  $HNO_2$  present in the feed are also shown in Figure 7; these profiles illustrate the improved partitioning in the extraction section of the bank.



FIG. 7 PLUTONIUM PARTITIONING PROFILES



The poor partitioning with  $HNO_2$  in the lAF probably results from a slow reaction in the destruction of nitrite in the lB bank:

$$2HNO_2 + N_2H_4 \rightarrow N_2 + N_2O + 3H_2O$$

or

 $2HNO_2 + 2NH_2SO_3 \rightarrow 2N_2 + 2SO_4 + 2H_2O + 2H^+$ 

Complete destruction of  $HNO_2$  is a prerequisite to chemical reduction of Pu(IV) to Pu(III), and a slow step in the destruction of  $HNO_2$  would have the effect noticed in the experimental work.

#### Chemical Reductant

Ferrous sulfamate and hydrazine-stabilized uranous nitrate were equally effective reducing reagents for plutonium. Without  $HNO_2$  in the lAF stream (Runs 8-12 of Table III), losses of plutonium to the uranium product were as low as 0.05% with either 0.08 or 0.32M ferrous sulfamate in the lBX stream or with uranous nitrate introduced into the scrub section of the lB bank at a rate of 0.01 mols per liter of lBX. The stage profiles of Figure 7 illustrate that both chemical reductants are equally useful.

#### APPENDIX A

#### THE MINIATURE CENTRIFUGAL CONTACTOR

#### Description

Details of the development and construction of the miniature centrifugal contactor were described previously.<sup>(2)</sup> The basic components are the mixer and settler (Figures A-1 and A-2, respectively). These components are located in mixing and settling chambers that are wells in the stainless steel housing shown in Figure A-3. Figure A-4 shows an assembled 16-stage contactor.

Ducts for solutions to flow between the component parts are visible in the plastic, single-stage contactor of Figure A-5. The light (organic) and heavy (aqueous) phases flow into the mixer along the shaft and are discharged radially from the bottom periphery of the mixer to the bottom of the settler. The mixed phases are pumped by centrifugal action up through the settler, where they are separated at 350 x gravity(G). The separated phases then flow over circular discharge weirs within the settler into collector rings in the stationary housing. The ducts out of the collector rings are tangential to the collector rings, so the solutions ejected from the settler are pumped to the next mixer by centrifugal force.

The housing for the 16-stage contactor also contains access ports for introducing solutions to any stage, for removing samples, and for inserting in-line instrumentation into flowing streams within the contactor. Thermocouple wells and internal ducts for circulating heating or cooling water are available also.

Figure A-6 shows two contactors and associated auxiliary equipment installed in the miniature pilot plant for processing irradiated fuels.<sup>(4)</sup>

(Text continued on page 25)



FIG. A-2 SETTLER



FIG. A-3 HOUSING OF 16-STAGE CONTACTOR



DPSTF - 1 - 6640

FIG. A-4 ASSEMBLED 16-STAGE CONTACTOR



DPSTF - 1 - 5029

SIDE VIEW



DPSTF - 1 - 5027

#### BOTTOM VIEW

## FIG. A-5 PLASTIC MODEL OF SINGLE-STAGE CONTACTOR

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FIG. A-6 CONTACTORS INSTALLED IN PILOT PLANT

#### Operating Characteristics

The major operating characteristics that are important in transposing performance on a miniature scale to that on a much larger scale are residence time, mixing intensity, and centrifugal force in the settler. These characteristics for Purex operations in the miniature centrifugal contactors are:

- The residence time varies with throughput and organicto-aqueous flow ratio, since the dynamic holdup of each phase is fixed by the position of the overflow weirs. The variations in the residence time within the 1A bank are shown in Figure A-7. The corresponding variations for flow ratios for the 1B and 1C banks are probably similar.
- Mixing intensity varies with the agitator speed, throughput, and flow ratio. Uranium extraction efficiency, which is controlled by diffusion and mixing intensity, was used as the indicator of mixing intensity. The uranium extraction efficiency varied with mixer speed as shown in Figure A-8. On the basis of prior experience with mixer-settlers, the extraction efficiency of components other than uranium should be similar for the 1A bank flowsheet, but the efficiency for other flowsheets, for example scrubbing in the 1A bank, partitioning plutonium in

the 1B bank, and stripping uranium in the 1C bank, could be lower. Therefore, uranium extraction efficiency should be used only as an indicator of mixing intensity for comparing different-sized contactors; it should not be used as a measure of extraction or scrubbing efficiency for all parts of the flowsheet.

• The centrifugal force in the settlers was maintained at 350G by maintaining the bowl speed at 7000 rpm.



FIG. A-7 RESIDENCE TIMES IN 1A BANK



FIG. A-8 URANIUM EXTRACTION EFFICIENCY IN 1A BANK

#### APPENDIX B

#### PROCESS MONITORING AND CONTROL TECHNIQUES

Where practical, conventional instruments and control techniques were used with only minor modifications for the miniaturized equipment. Remote-indicating in-line instrumentation with rapid read-out of process performance was used to get the rapid monitoring required by short-residence contactors. The indicators of process performance were centralized with precise controllers for rapid response to transients. The control methods used are listed in Table B-I.

The most critical variable to be monitored and controlled was the ratio of solvent flow to feed flow (1AX/1AF) in the 1A bank. With the optimum ratio, solvent leaving the 1A bank is almost saturated with uranium, and a minimum amount of solvent is available to extract fission products. With the ratio below optimum, the solvent cannot extract all the uranium and plutonium; the excess builds up in the extraction section and is eventually lost to the aqueous waste. With the ratio above optimum, excess extractant carries more fission products, causing a decrease in uranium and plutonium purification.

In practice, the 1AX/1AF was adjusted to maintain a controlled inventory of plutonium in the first two stages of the extraction section; changes in the ratio were monitored from changes that occurred in the plutonium inventory when the flow ratio deviated from optimum. The plutonium inventory was indicated by the plutonium extraction profile, which was continuously monitored with silicon alpha detectors<sup>(5)</sup> (see Figure B-1) inserted in aqueous interstage ducts of the contactor. Typical profiles of plutonium and uranium in the extraction section of the 1A bank are shown in Figure B-2; profile changes resulting from small changes in the 1AX/1AF are illustrated in Figure B-3. Normal adjustments to maintain the plutonium profile were too small to be detected by the rotameters used as flow indicators.

#### TABLE B-I

#### Monitoring Instrumentation and Controls

Item Monitored	Monitoring Instrumentation		
and Controlled	Sensor	Indicator	Controlling Mechanism
Agitator speed	DC generator coupled to air motor	Microammeter	Air pressure to air motor
Centrifuge speed	DC generator coupled to <b>air</b> motor	Microammeter	Air pressure to air motor
Temperature	Iron-constantan thermocouple	Potentiometer	Mix ratio of hot-to-cold water
Flow	Rotameter	Direct reading	Needle valve in line from pressurized feed tank
lAF/lAX flow ratio	Silicon alpha probes in aqueous interstage ducts of IA bank	Count rate meter	lAX flow
Gamma activity of product streams	Well-type scintillation crystals	400-channel pulse height analyzer	-
Pu in lAW	Silicon alpha probes in aqueous inter- stage ducts of 1A bank	Count rate meter	lAX flow
Stage profiles and end stream activities	Samples		

	Ce	oaxial Connector
N type Silicon Wafer 1/8 ' dia , sealed in glass tubing Platinum Reference Electrode	SS Tubing	
		ST 55

FIG. B-1 SILICON ALPHA DETECTOR



FIG. B-2 1A BANK EXTRACTION PROFILES



FIG. B-3 EFFECT OF 1AX/1AF FLOW RATIO ON PLUTONIUM EXTRACTION PROFILE

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