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AEC RESEARCH AND DEVELOPMENT REPORT

RECOVERY OF THORIUM AND URANIUM-233 FROM IRRADIATED THORIUM OXIDE AND METAL

W. E. PROUT
A. E. SYMONDS

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RECOVERY OF THORIUM AND URANIUM-233 FROM IRRADIATED THORIUM OXIDE AND METAL

by

William E. Prout

and

Albert E. Symonds

Approved by

L. H. Meyer, Research Manager
Separations Chemistry Division

January 1967

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ABSTRACT

Processes for recovering both ^{233}U and thorium from irradiated thorium oxide or thorium metal were adapted and modified as required for use in the existing Savannah River facilities that are normally used to process enriched uranium—aluminum alloy. In the resulting process, ^{233}U and thorium are separated and purified by solvent extraction with 30 vol % tributyl phosphate. The ^{233}U is subsequently concentrated by cation exchange and converted to uranium trioxide.

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INTRODUCTION

This report describes the development and testing of chemical processes for the recovery and purification of ^{233}U and thorium from irradiated thorium target elements. The prime objective of the project was to develop a process compatible with the Savannah River chemical separations plant that was previously converted from Purex operations(1,2) to the processing of irradiated ^{235}U-Al alloy.(3)

The elements to be processed were fabricated either as thorium metal cylinders clad with aluminum, or as annular aluminum cans that contained granular thorium oxide. These elements were irradiated at a flux of $\sim 3 \times 10^{14}$ n/(cm^2)(sec) to ~1500 grams of mass 233 per metric ton of thorium, and subsequently were cooled 90-120 days to allow adequate decay of ^{233}Pa and ^{131}I before chemical processing.

The requirements of the chemical process were (1) to separate the thorium and uranium from each other so that the thorium contained less than 20 ppm ^{233}U and the ^{233}U contained less than 0.1% thorium, (2) to purify ^{233}U so that residual fission product gamma activity would be less than 0.1 microcurie per milligram of ^{233}U, and (3) to purify the thorium so that residual fission products and ^{233}Pa would contribute less than 10% to the total gamma radiation of the thorium product at all times after chemical separation.

The particular irradiation history of the elements was a major factor in determining the purification required. During irradiation, production of ^{233}U by

$$^{232}\mathrm{Th}(n,\gamma)\ ^{233}\mathrm{Th} \xrightarrow[22m]{\beta^-} {}^{233}\mathrm{Pa} \xrightarrow[27d]{\beta^-} {}^{233}\mathrm{U}$$

is accompanied by undesirable side reactions (Figure 1) that form fission products and by uranium and thorium isotopes that produce energetic γ-emissions in their decay chains.(4) With the relatively high flux and short irradiation times, the quantity of fission products was small, and the quantity of ^{233}Pa was large because little of the ^{233}Pa had decayed to fissionable ^{233}U in the reactor. The ^{232}U, formed primarily by the sequence starting with the (n,2n) reaction of high-energy neutrons with ^{232}Th, was controlled to <6.5 ppm with the proper reactor lattice. ^{232}U increases radiation both in the ^{233}U product as ^{228}Th grows in

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and decays through ^{208}Tl, and in the thorium product as a result of the ^{228}Th formed before chemical separation. Because the ^{234}Th content is relatively high as shown in Figure 2, the purified thorium must be aged about nine months to reduce the ^{234}Th content to an acceptable low level, as shown in Figure 3. Fission products and ^{233}Pa also decay during this aging period; consequently, the decontamination requirements during purification of the thorium are lower than would be required if the long period were not provided for ^{234}Th decay. The calculated decontamination factors required to meet the product purity criteria are summarized in Table I.

TABLE I

Decontamination Requirements

Isotope	Overall Decontamination Factor	
	^{233}U Product	Thorium Product
^{103}Ru-^{103}Rh	1.5×10^4	5.5×10^3
^{106}Ru-^{106}Rh	2.8×10^2	1.2×10^4
^{95}Zr-^{95}Nb	1.6×10^4	3.0×10^4
^{233}Pa	1.0×10^8*	1.6×10^6

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* Protactinium removal required for uranium processing by cation exchange in the finishing step.

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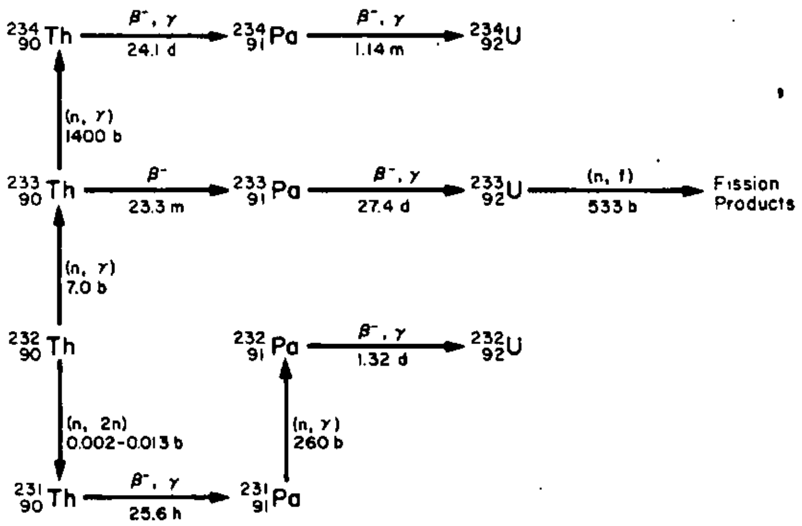
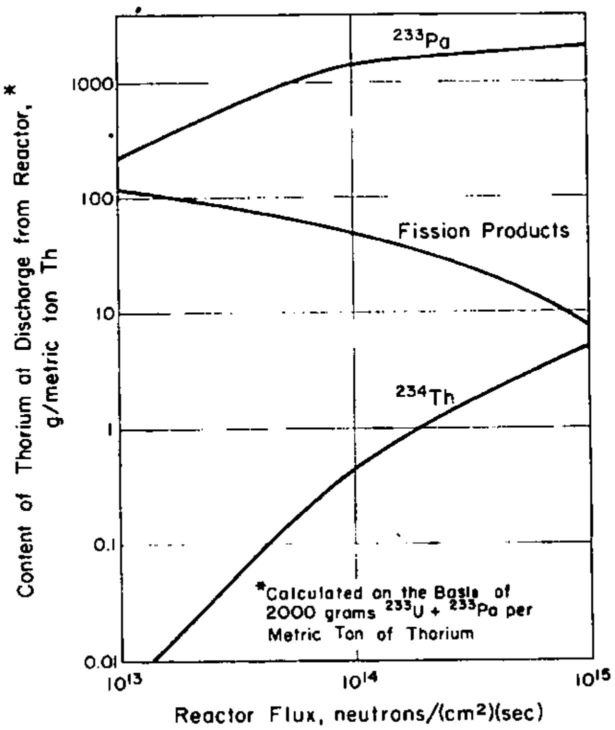


FIG. 1 MAJOR NUCLEAR REACTIONS DURING IRRADIATION OF THORIUM



*Calculated on the Basis of 2000 grams ^{233}U + ^{233}Pa per Metric Ton of Thorium

FIG. 2 ACTIVITY IN IRRADIATED THORIUM

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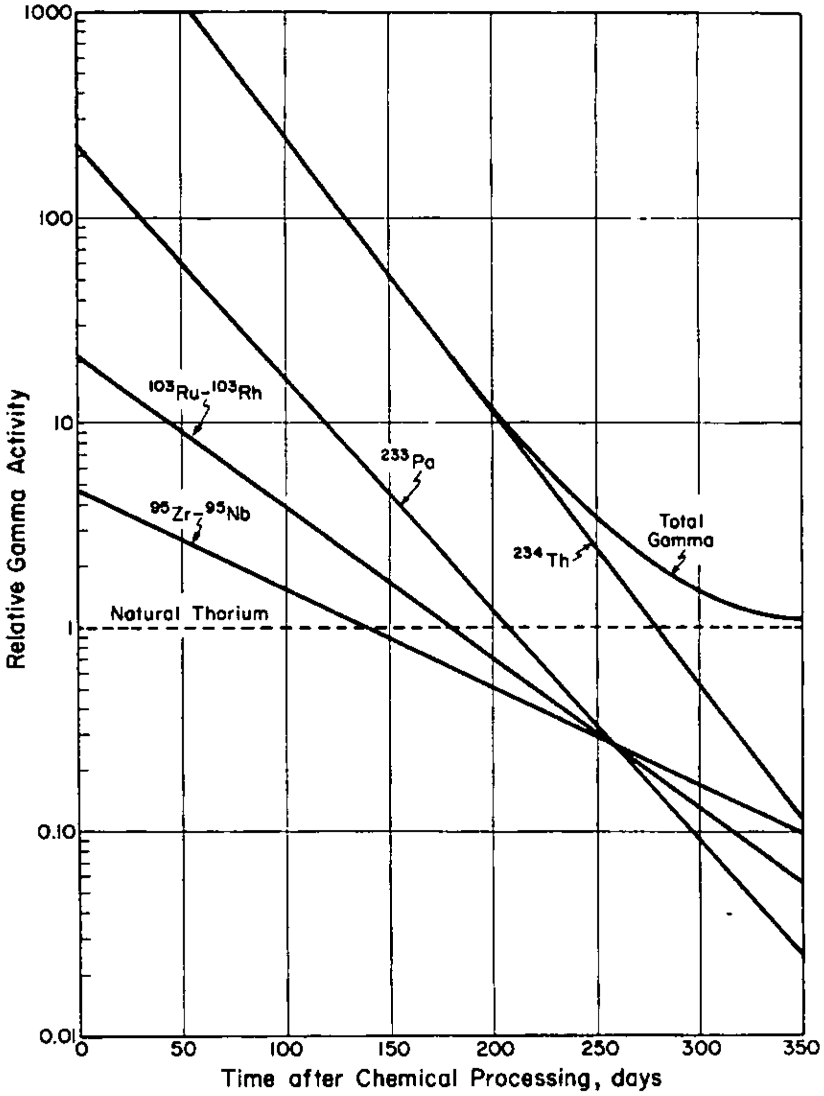

FIG. 3 RADIATION FROM SEPARATED THORIUM

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SUMMARY

Principal steps in the process for recovering and purifying ^{233}U from irradiated thorium oxide or metal are as follows:

- Dissolution of elements. For thorium metal cores, the aluminum cladding is dissolved in $NaOH—NaNO_3$ solution and then the thorium metal core is dissolved in 10-15M HNO_3—0.01M HF. For thorium oxide cores, the aluminum can and core are both dissolved in 12M HNO_3—0.025M HF—0.1M $Al(NO_3)_3$—2.5 x 10^{-4}M $Hg(NO_3)_2$.
- Sorption of ^{233}Pa on manganese dioxide.
- Separation of ^{233}U and thorium from each other and from aluminum, protactinium, and fission products in a first cycle of solvent extraction with 30 vol % TBP in kerosene.
- Purification of the thorium from residual protactinium and fission products in another cycle of solvent extraction with 30 vol % TBP in kerosene.
- Purification of the ^{233}U from residual thorium, protactinium, and fission products in a second cycle of solvent extraction with 7-1/2 vol % TBP in kerosene.
- Concentration of the ^{233}U by cation exchange.
- Precipitation of the ^{233}U as ammonium uranate.
- Calcination of the ammonium uranate to uranium trioxide.
- Storage of the recovered thorium as a concentrated solution of thorium nitrate.

In the development program, all process steps except the second uranium cycle were satisfactorily demonstrated in laboratory test facilities. (The second uranium cycle had already been demonstrated.(3)) The recovery efficiency for thorium and uranium was >99% in the first cycle of solvent extraction. Greater than 99% of the ^{233}Pa and ^{95}Zr-^{95}Nb, and 90 to 99% of the ruthenium were rejected in this cycle. In the second thorium cycle, thorium recovery efficiency was >99.9%, while >99% of the residual ^{233}Pa and ^{95}Zr-^{95}Nb, and ~99% of the ruthenium were rejected.

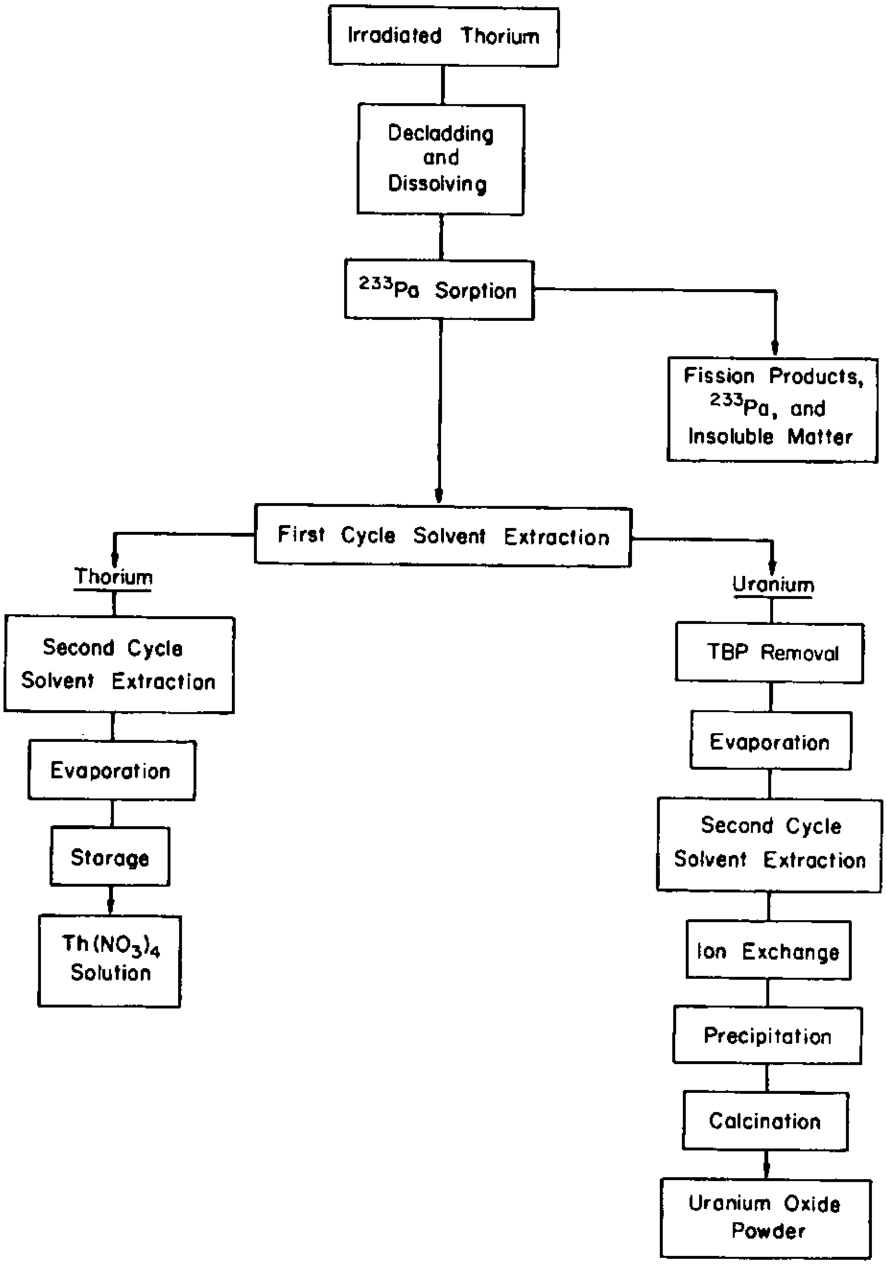

FIG. 4 FLOW DIAGRAM FOR THORIUM PROCESSING

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PROCESS DESCRIPTION

The overall process flow diagram is shown in Figure 4 and is described in the following sections.

THORIUM OXIDE DISSOLUTION (Figure 5)

Thorium oxide and the aluminum cans are sequentially dissolved in the same 12M HNO_3—0.025M HF—0.1M $Al(NO_3)_3$ dissolvent.(7) Fluoride catalyzes the dissolution of thorium oxide, and mercuric ions catalyze the dissolution of aluminum. The sequence of operations starts with an oxide "heel":

- The heel is partially dissolved in new dissolvent at boiling.
- The solution is cooled when it reaches 1M $Th(NO_3)_4$, and the new elements are added.
- Mercuric nitrate is added, and then the newly charged elements are decanned in the boiling $Th(NO_3)_4$—HNO_3 solution, leaving a thorium oxide heel for the next dissolving cycle.
- The solution is cooled, diluted, and centrifuged to remove entrained thorium oxide; the centrifuged thorium oxide is returned to the dissolver.

The first heel of thorium oxide is formed by chemically decanning the first elements with 5M HNO_3—$Hg(NO_3)_2$ solution. A typical dissolving time cycle is shown in Figure 6. The solubility of thorium nitrate in the presence of nitric acid and aluminum nitrate is shown in Figure 7.

Laboratory studies(7) with thorium oxide showed that dissolution rates depended on the composition of the dissolvent, and on the particle size distribution of the thorium oxide, but are independent of irradiation. The most satisfactory dissolvent was 13M HNO_3—0.025M HF—0.1M $Al(NO_3)_3$; the $Al(NO_3)_3$ is added to decrease corrosion of the stainless steel dissolvers by fluoride. The particle size distribution given in Table II was typical of the ThO_2 used in the reactor target elements. Because fine particles with large surface-to-mass ratio dissolved more rapidly than coarse particles, the dissolution was initially rapid but slowed during dissolution of the residual coarse particles. The practice of dissolving the heel of coarse particles in new dissolvent, and then dissolving the fine particles of a new charge of elements in the depleted acid tended to yield more uniform dissolving rates.

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TABLE II

Particle Size Distribution of Thorium Oxide

U. S. Std. Screen	Weight %
+16	17.8
-16 +20	7.7
-20 +40	14.4
-40 +50	3.1
-50 +60	3.1
-60 +80	6.2
-80 +100	8.3
-100	39.4

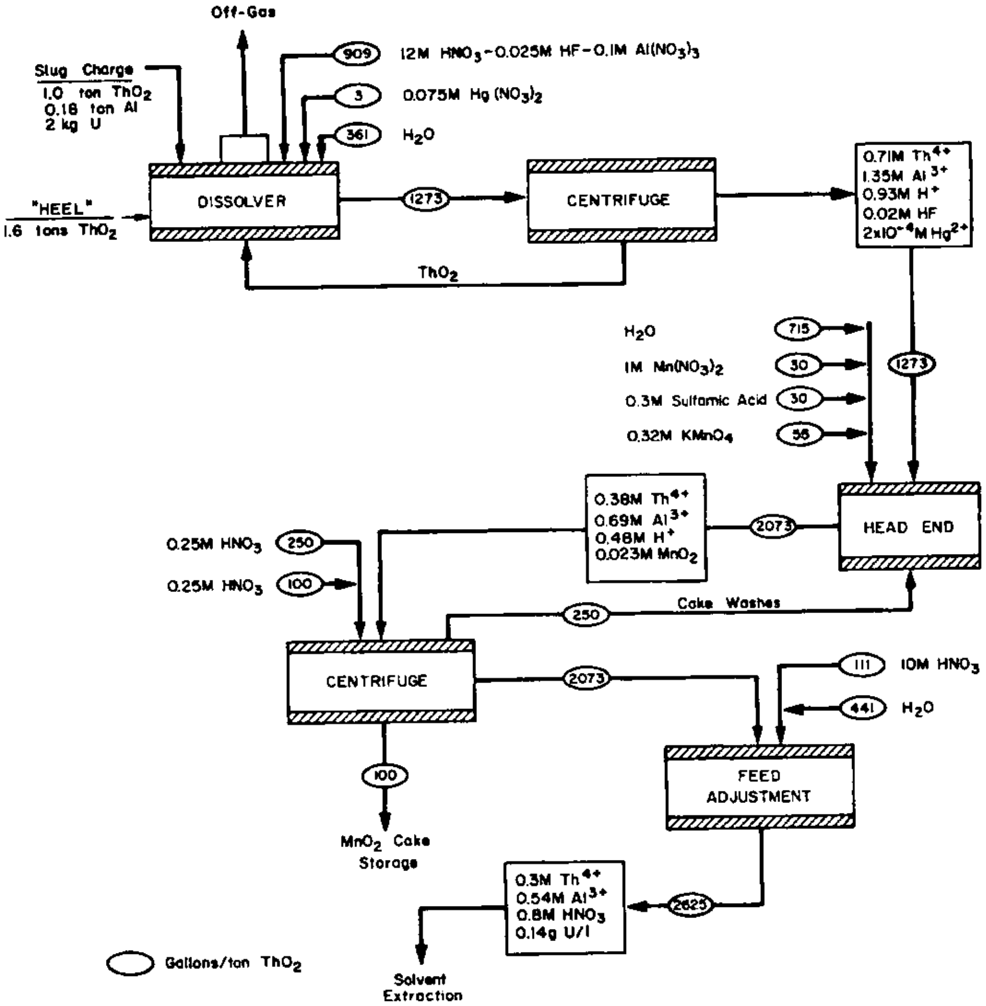

FIG. 5 THORIUM OXIDE DISSOLVING AND HEAD END

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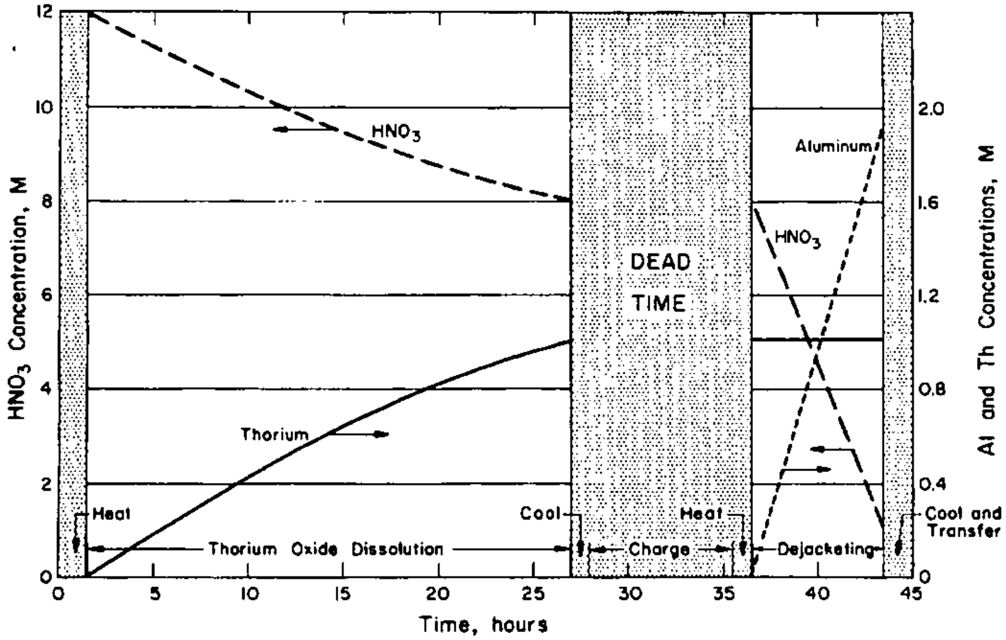
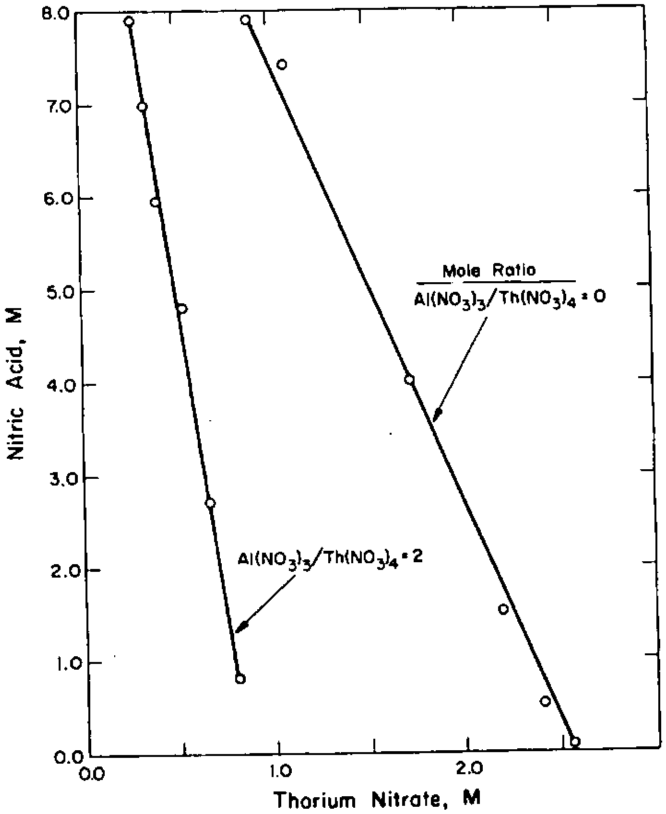


FIG. 6 TIME CYCLE FOR THORIUM OXIDE DISSOLUTION

FIG. 7 SOLUBILITY OF THORIUM NITRATE IN NITRIC ACID (20°C)



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THORIUM METAL DISSOLUTION (Figure 8)

The aluminum-clad thorium metal is dissolved by conventional procedures.(8,9) The aluminum cladding is dissolved in NaOH—$NaNO_3$ solution; the thorium core is then dissolved in 10-15M HNO_3 with 0.005M fluoride added as a catalyst. The dissolution reaction is

$$Th + 6HNO_3 \rightarrow Th(NO_3)_4 + 3H_2O + NO + NO_2$$

In practice 5 to 5.2 moles of nitric acid are consumed per mole of thorium dissolved. The effects of the concentrations of dissolved thorium nitrate, nitric acid, and hydrofluoric acid on the dissolving rate as determined by laboratory studies are shown in Figure 9.

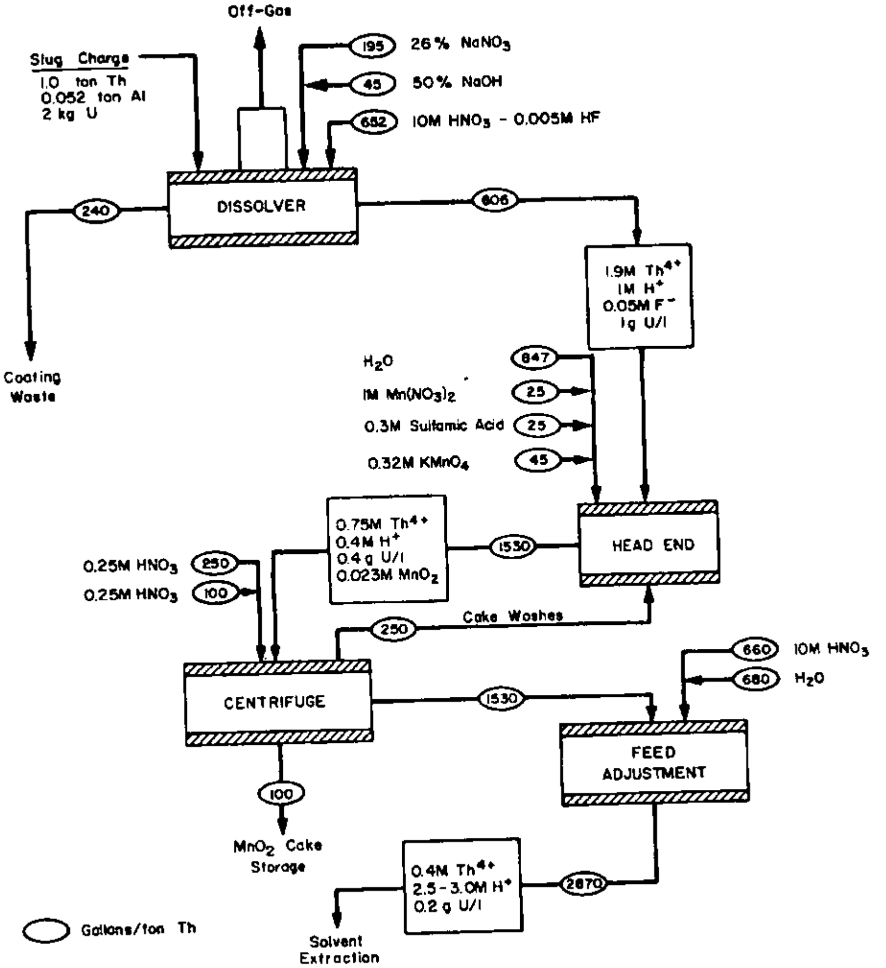

FIG. 8 THORIUM METAL DISSOLVING AND HEAD END

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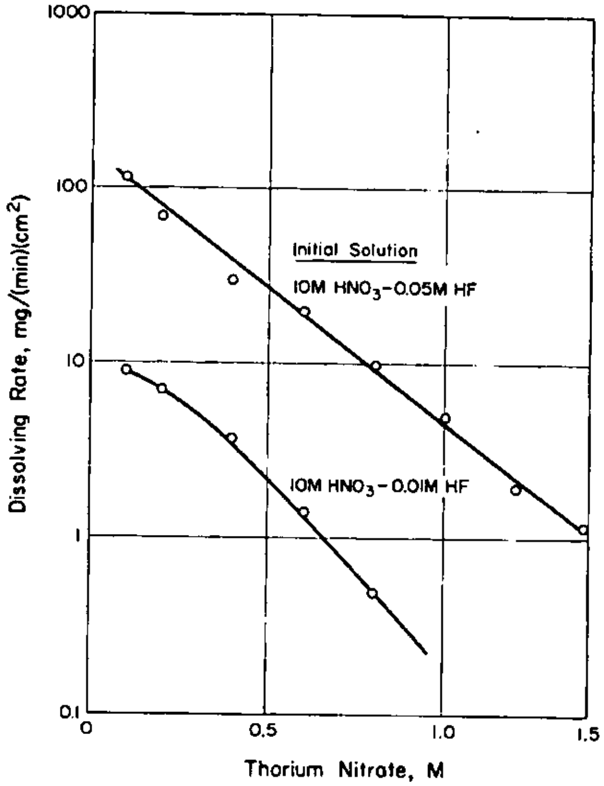

FIG. 9 THORIUM METAL DISSOLVING RATES DURING EXTENDED DISSOLUTIONS

^{233}Pa SORPTION (Figures 5, 8)

^{233}Pa is sorbed on freshly precipitated manganese dioxide with procedures similar to the "reverse strike" head end used in the Purex process.(5,10,11) The sequence of operations is listed below for either a solution of dissolved thorium oxide adjusted to 0.4M $Th(NO_3)_4$—0.72M $Al(NO_3)_3$—0.8M HNO_3 (Figure 5) or a solution of dissolved thorium metal adjusted to 0.7M $Th(NO_3)_4$—0.7M HNO_3 (Figure 8):

- Manganese nitrate and sulfamic acid are added, and the solution is heated to 70°C.
- Potassium permanganate is added with agitation to precipitate manganese dioxide.
- The slurry is digested for 30 minutes, cooled, and centrifuged.
- The cake is washed and slurried from the centrifuge.

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Results of laboratory tests showing the effects of solution composition and digestion time on sorption are shown in Figure 10. Other tests showed that sorption was essentially unchanged when the cake was re-slurried after standing 10 to 24 hours and then re-centrifuged, and that sorption is enhanced by:

- Maintaining the composition of the feed solution below 0.4M $Th(NO_3)_4$—0.72M $Al(NO_3)_3$—0.8M HNO_3.
- Adding sulfamic acid to scavenge radiolytically formed nitrous acid, which otherwise dissolves manganese dioxide.
- Centrifuging the precipitate promptly to decrease radiolytic decomposition of the manganese dioxide.

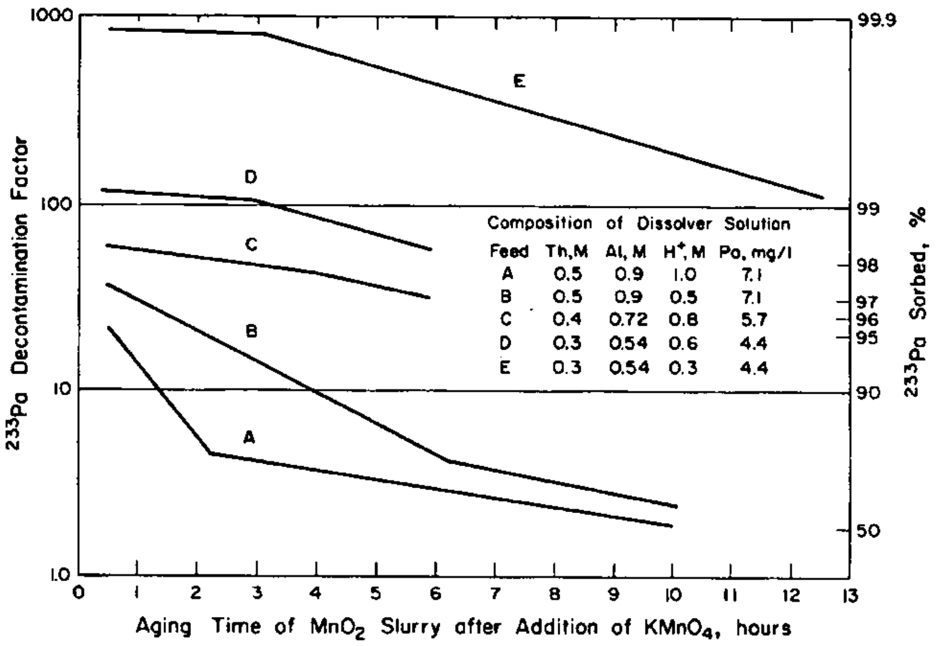

FIG. 10 SORPTION OF ^{233}Pa WITH MnO_2

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SOLVENT EXTRACTION

The uranium and thorium are separated by solvent extraction into two aqueous streams, each of which is adequately free of ^{233}Pa and fission products. The extraction is performed in mixer-settlers with 30% tributyl phosphate in kerosene.(e) The overall process includes a first cycle for the primary separation of thorium and uranium and decontamination of each, plus second cycles for both thorium and uranium. Each cycle involves extracting uranium and/or thorium from an aqueous stream into solvent, scrubbing the resulting product-bearing solvent to remove the small amounts of extracted ^{233}Pa and fission products, and stripping the thorium or uranium back into an aqueous stream. The transfer of thorium and uranium between phases is controlled by adjusting the aqueous concentrations of nitric acid and aluminum nitrate.

Seven mixer-settler banks are used in the solvent extraction process, as described below.

<u>First Cycle</u>

1A Bank (16 stages, center fed) - coextraction of thorium and uranium from fission products with 30 vol % TBP in "Ultrasene"*

1B Bank (16 stages, center fed) - back-extraction of thorium with dilute HNO_3

1C Bank (12 stages, end fed) - back-extraction of uranium with dilute HNO_3

<u>Second Thorium Cycle</u>

2A Bank (16 stages, center fed) - extraction of thorium from residual fission products with 30 vol % TBP in "Ultrasene"

2B Bank (12 stages, end fed) - back-extraction of thorium with dilute HNO_3

<u>Second Uranium Cycle</u>

1D Bank (16 stages, center fed) - extraction of uranium from residual fission products with 7.5 vol % TBP in "Ultrasene"

1E Bank (12 stages, center fed) - back-extraction of uranium with dilute HNO_3

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* A refined kerosene product of the Atlantic Refining Company.

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The extraction reactions are

$$Th(NO_3)^{2+}_{2\,aq} + 2NO^-_{3\,aq} + 2TBP_{org} \rightleftharpoons Th(NO_3)_4 \cdot 2TBP_{org}$$

$$UO^{2+}_{2\,aq} + 2NO^-_{3\,aq} + 2TBP_{org} \rightleftharpoons UO_2(NO_3)_2 \cdot 2TBP_{org}$$

$$H^+_{aq} + NO^-_{3\,aq} + TBP_{org} \rightleftharpoons HNO_3 \cdot TBP_{org}$$

Nitric acid is less extractable than thorium nitrate, which is less extractable than uranyl nitrate; the ratio of extraction coefficients E_U/E_{Th} = ~14 for most conditions. The operating parameters of the solvent extraction flowsheets were designed with McCabe-Thiele diagrams and the distribution data given in Tables III and IV, in Figures 11 and 12, and in DP-181.(12) The solubility of thorium nitrate in 30 vol % TBP is given in Figure 13.(12,13) Exceeding this solubility limit produces a second organic phase in which a thorium nitrate—TBP complex is the solvent. As seen in Figure 13, the solubility increases with temperature and decreases with acid concentration.

TABLE III

Equilibrium Distribution of Thorium Nitrate and Uranyl Nitrate Between TBP in "Ultrasene" and Aqueous Nitric Acid Solutions*

TBP, vol %	Temp, °C	Concentration, M			$(E^o_a)_{Th}$	Concentration, M		$(E^o_a)_U$	E_U/E_{Th}
		H_a	Th_a	Th_o		U_a	U_o		
30	30	0.26	0.233	0.080	0.343	2.81×10^{-3}	1.42×10^{-2}	5.05	14.7
30	30	0.56	0.219	0.093	0.425	2.46	1.45	5.89	13.9
30	30	0.27	0.342	0.133	0.389	2.48	1.95	5.85	15.0
30	30	0.55	0.336	0.137	0.408	2.25	1.47	6.53	16.0
30	50	0.26	0.225	0.062	0.275	3.13	1.14	3.64	13.2
30	50	0.53	0.207	0.076	0.367	2.63	1.15	4.37	11.9
30	50	0.30	0.322	0.111	0.345	2.57	1.15	4.48	13.0
30	50	0.55	0.306	0.124	0.405	2.32	1.16	5.00	12.3
42.5	30	0.28	0.230	0.120	0.522	2.70	2.08	7.70	14.7
42.5	30	0.57	0.213	0.135	0.634	2.36	2.09	8.86	14.0
42.5	30	0.29	0.334	0.199	0.596	2.46	2.07	8.42	14.1
42.5	30	0.56	0.328	0.218	0.665	2.30	2.08	9.04	13.6

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* Subscripts "a" and "o" denote aqueous and organic phases, respectively.

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TABLE IV

Equilibrium Distribution of Thorium Nitrate Between 30 vol % TBP in "Ultrasene" and Aqueous Solutions of Nitric Acid and Aluminum Nitrate (30°C)

Concentration, M				
$Al(NO_3)_3$	HNO_3	Th_a	Th_o	$(E_a^o)_{Th}$
0.6	0.0	2.87×10^{-3}	0.0138	4.81
0.6	0.0	0.0876	0.183	2.09
0.6	0.2	0.0012	0.0276	23.0
0.6	0.2	0.0713	0.180	2.52
0.6	0.97	0.0020	0.013	6.5
0.6	0.97	0.105	0.142	1.32

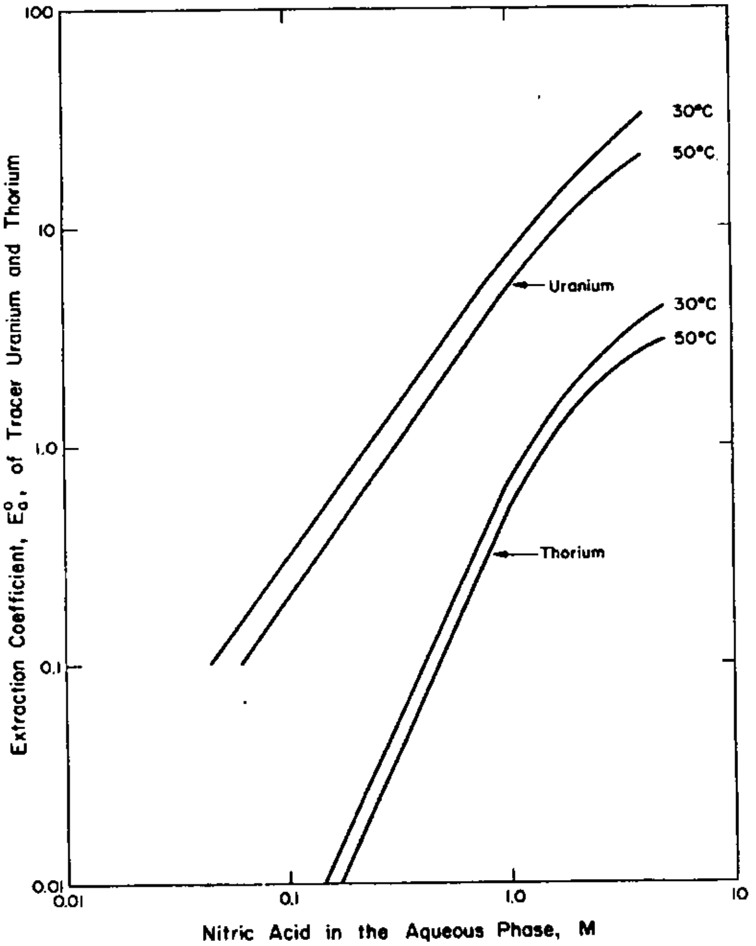

FIG. 11 DISTRIBUTION OF TRACER ($<10^{-2}$M) URANYL NITRATE AND THORIUM NITRATE BETWEEN 30 vol % TBP - "ULTRASENE" AND AQUEOUS NITRIC ACID

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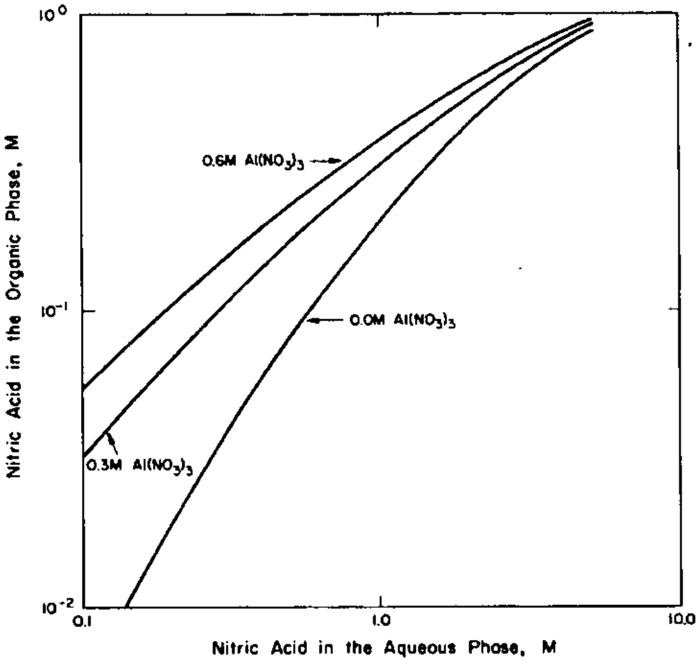

FIG. 12 EQUILIBRIUM DISTRIBUTION OF NITRIC ACID BETWEEN 30 vol % TBP - "ULTRASENE" AND AQUEOUS NITRIC ACID IN THE PRESENCE OF $Al(NO_3)_3$ (30°C)

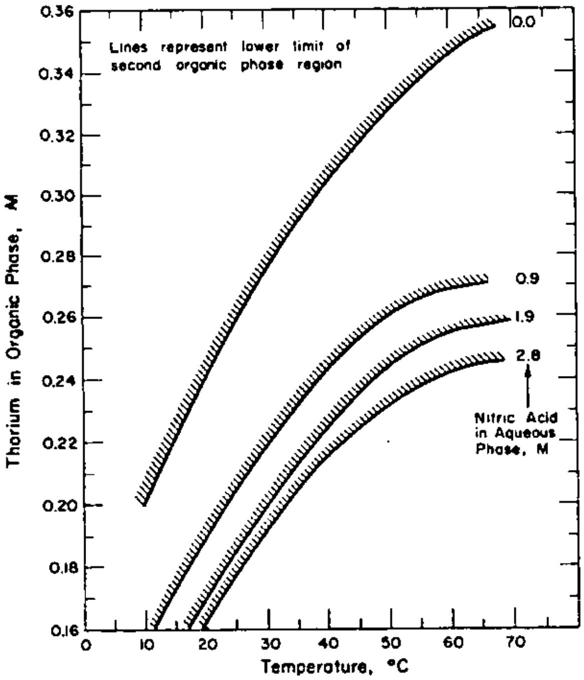

FIG. 13 LIMITS FOR THE EXISTENCE OF A SECOND ORGANIC PHASE IN 30 vol % TBP - "ULTRASENE"

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First Cycle

Adjusted feed from the clarification (^{233}Pa sorption) step is processed through the 1A, 1B, and 1C mixer-settlers according to the flowsheet shown in Figure 14. Thorium and uranium are coextracted from fission products and ^{233}Pa and scrubbed with HNO_3—Na_3PO_4 in the 1A bank. The thorium is partitioned from uranium into dilute nitric acid in the 1B bank, and the uranium is stripped in the 1C bank.

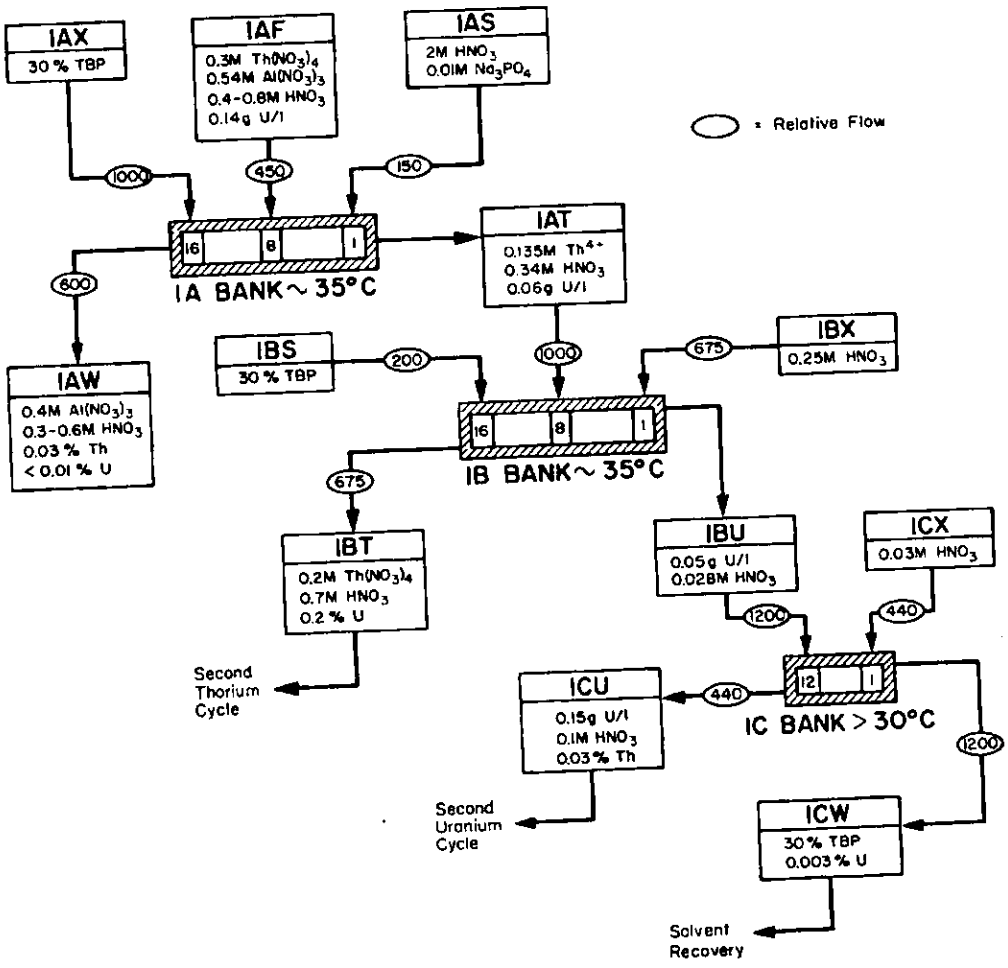

FIG. 14 FIRST CYCLE OF SOLVENT EXTRACTION

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The limits of operating conditions calculated from distribution data are summarized in Table V and in Figures 15, 16, and 17. These limits were calculated for a cocurrent stage efficiency of $\geqslant$80% to provide ranges as wide as possible without violating any of the following criteria:

- $\leqslant$1.0% loss of ^{233}U to the thorium stream (1BT).
- $\leqslant$1.0% loss of thorium to the uranium stream (1CU).
- Uranium concentration in the 1C bank $\leqslant$ uranium concentration in the 1CU.
- No second organic phase [which occurs at high concentrations of extracted $Th(NO_3)_4$—HNO_3], no excessive reflux of ^{233}Pa, and no formation of $Th_3(PO_4)_4$ gel (which occurs in the extraction section of the 1A bank when the concentration of nitric acid is $\leqslant$0.2M).

Details of the design of the most important of these limits are discussed in the following paragraphs.

TABLE V

Operating Limits for Solvent Extraction

Variable*		Minimum	Maximum
	First Cycle		
1A Bank			
Temperature, °C	(1,2,3)	30	40
TBP in 1AX, %	(1,2,3,7,8)	28	32
HNO_3 in 1AS, M**	(1,2,3,4)	1	2
Phosphate in 1AS, M	(6)	0.005	0.015
$Th(NO_3)_4$ in 1AT, M	(1,2,3)	0.10	0.135
1AS/1AX flow ratio	(1,2,3)	0.12	0.18
1AX/(1AF + 1AS) flow ratio	(1,2,3,5)	1.0	2.4
1AF composition			
Thorium nitrate, M	(1,2,3)	0.165	0.45
Nitric acid	(1,2,3,4,6)	See Figure 15	
Aluminum nitrate	(1,2,3,6)	See Figure 15	
1B Bank			
Temperature, °C	(1,2,7)	30	40
TBP in 1BS, %	(1,2,7,8)	28	32
1BX/1BS flow ratio	(1,2,7,8)	3.0	3.6
1BX/1AX flow ratio	(1,2,7,8)	See Figure 16	
HNO_3 in 1BX	(1,2,7,8)	See Figure 16	
Entrainment in 1BU			
% aqueous in organic	(8)	-	3.0
1C Bank			
Temperature, °C	(8)	30	-
HNO_3 in 1CX, M	(8)	0.0	0.05
Loss of thorium to 1CU, %	(8)	-	1.0
(1AX + 1BS)/1CX flow ratio	(8)	See Figure 17	

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TABLE V (Continued)

	Variable*		Minimum	Maximum
	Second Thorium Cycle			
2A Bank				
	Temperature, °C	(3)	30	60***
	TBP in 2AX, %	(3)	28	32
	HNO_3 in 2AS, M**	(3,4)	1	2
	Phosphate in 2AS, M	(6)	0.005	0.015
	$Th(NO_3)_4$ in 2AT, M	(3)	0.10	0.135
	2AS/2AX flow ratio	(3)	0.12	0.18
	2AX/(2AF + 2AS) flow ratio	(3,5)	1.0	2.4
	2AF composition			
	Thorium nitrate, M	(3)	0.165	0.45
	Nitric acid	(3,4,6)	See Figure 15	
2B Bank				
	Temperature, °C	(9)	30	-
	HNO_3 in 2BX, M	(9)	0.0	0.05
	2AX/2BX flow ratio	(9)	-	1.0
	Second Uranium Cycle			
1D Bank				
	Temperature, °C	(8)	-	50
	TBP in 1DX, %	(8)	7.1	8.4
	HNO_3 in 1DS, M	(8)	0.8	1.2
	1DS/1DX flow ratio	(8)	0.13	0.17
	1DX/1DF flow ratio	(8)	2.6	3.9
1E Bank				
	Temperature, °C	(8,9)	-	50
	HNO_3 in 1EX, M	(8,9)	0.0	0.05
	1DX/1EX flow ratio	(8,9)	-	9.0

---

* Code indicates that operation outside the limits will affect the following:

1. ^{233}U in the thorium product stream.
2. Thorium in the uranium product stream.
3. Third phase formation in the 1A bank (2A bank).
4. Precipitation of thorium phosphate in the 1A bank (2A bank).
5. Hydraulic operability of the extraction section of the 1A bank (2A bank).
6. Reflux of ^{233}Pa in the 1A bank (2A bank).
7. Reflux of ^{233}U in the 1B bank.
8. Reflux of ^{233}U in the 1C bank (1D bank)(1E bank).
9. Loss of thorium or uranium to solvent washers.

** See Figure 16 (Acid concentration of 1AS affects partitioning of thorium and uranium in the 1B bank.)

*** Plant operating experience showed that the maximum operating temperature is between 40 and 55°C.

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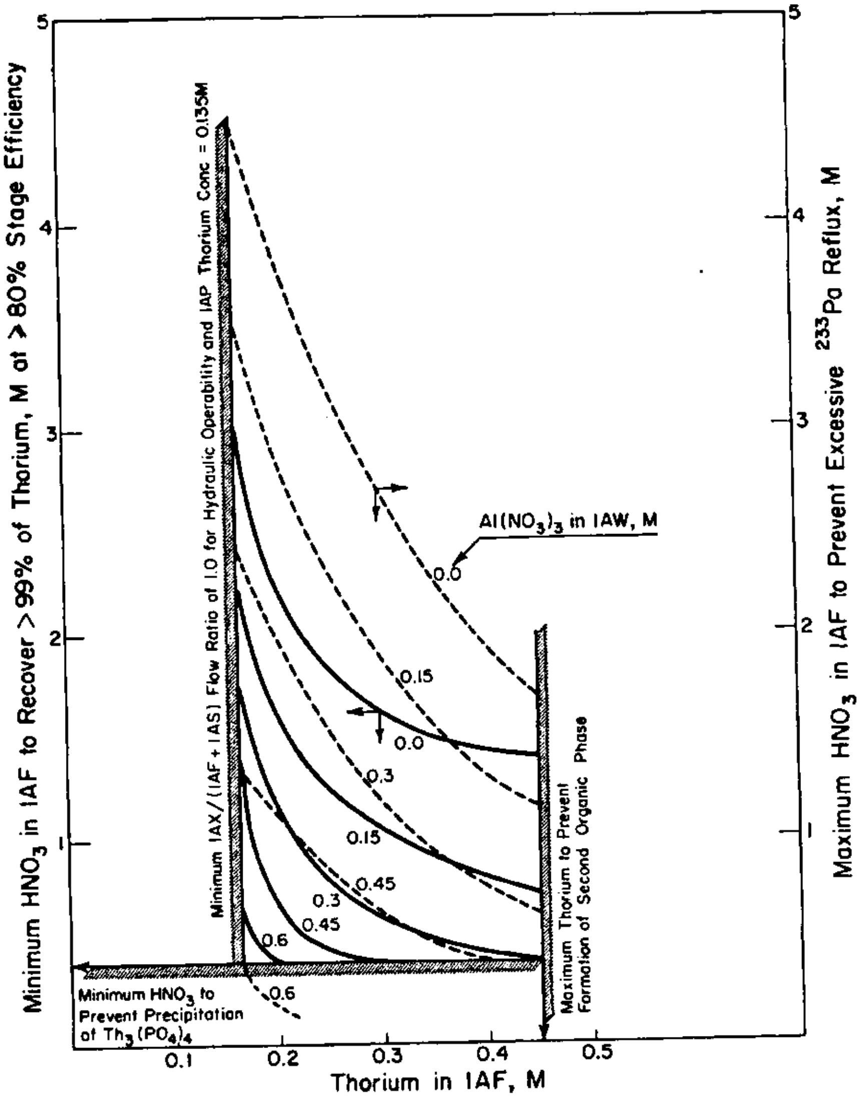

FIG. 15 OPERATING LIMITS FOR 1A BANK

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FIG. 16 OPERATING LIMITS FOR 1B BANK

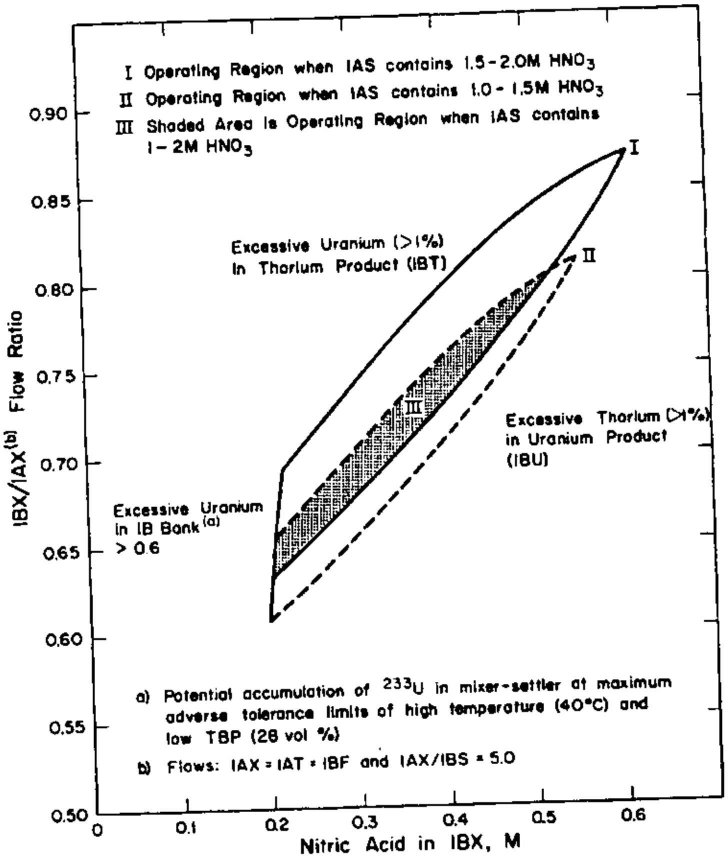
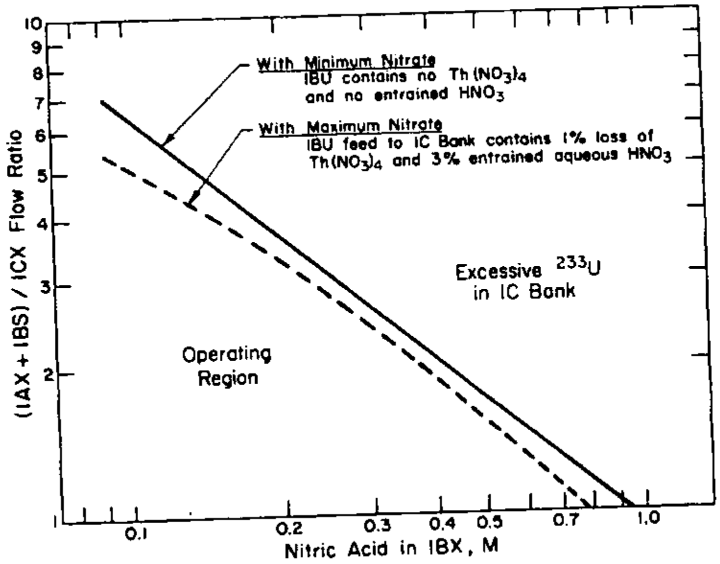


FIG. 17 OPERATING LIMITS FOR 1C BANK



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1A Bank

The 1A bank is operated to recover thorium with the maximum decontamination that is compatible with a thorium loss of <1%; uranium recovery efficiency is greater than that for thorium because of its greater extractability. Factors that pertain to the operation or the 1A bank are:

- High scrub (1AS) flow improves decontamination, but causes thorium to reflux in the scrub section. If refluxing is severe and thorium concentration exceeds ~0.19M in the organic phase, a second organic phase will form.
- Low scrub flows decrease the reflux of thorium, but generally result in lower decontamination from fission products.
- Increasing the temperature decreases capacity. As the temperature is raised the distribution coefficient for thorium between 30 vol % TBP and nitric acid solution decreases. Consequently, the second organic phase will form unless the effect is compensated by reducing the flow of 1AF, which will decrease the thorium nitrate in the organic product stream (1AT). Figure 18 shows the maximum thorium concentration that can be achieved in the 1AT as a function of temperature and 1AS/1AX flow ratio. For example, with a scrub-to-extractant flow ratio of ~1/7 (used in the Purex flowsheet(1)), the thorium in the organic product stream (1AT) must be ⩽0.135M to prevent exceeding the thorium nitrate solubility. In this case the second organic phase is just avoided at ⩽50°C, as shown in Figure 19. Typical stage profiles are illustrated in Figures 20, 21, and 22. Since these were constructed to fit specific flowsheet conditions, they illustrate only one of many workable conditions.
- Thorium phosphate precipitates in the extraction section if the acid concentration is ⩽0.2M.

The complete set of operating limits were calculated using McCabe-Thiele diagrams and are summarized in Table V and Figures 15 and 18.

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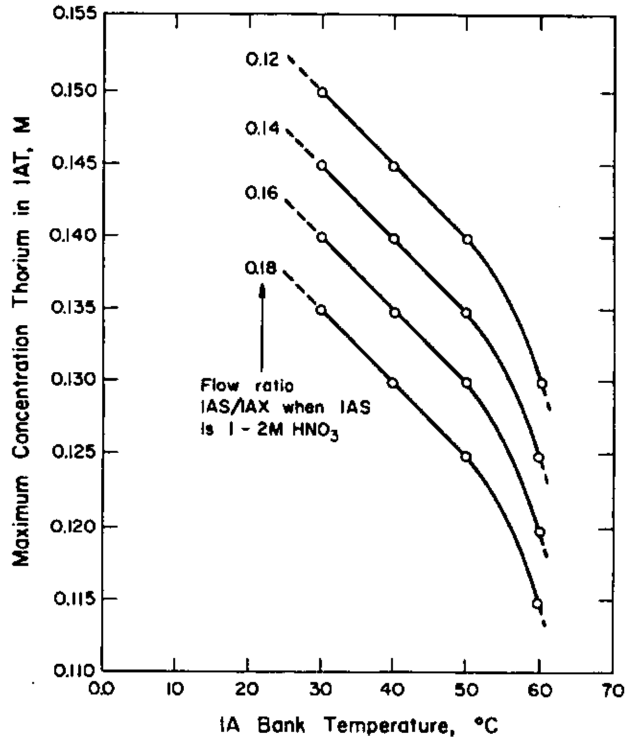

FIG. 18 EFFECT OF TEMPERATURE AND AQUEOUS-TO-ORGANIC FLOW RATIO IN THE SCRUB SECTION ON THORIUM EXTRACTION CAPACITY OF THE 1A BANK

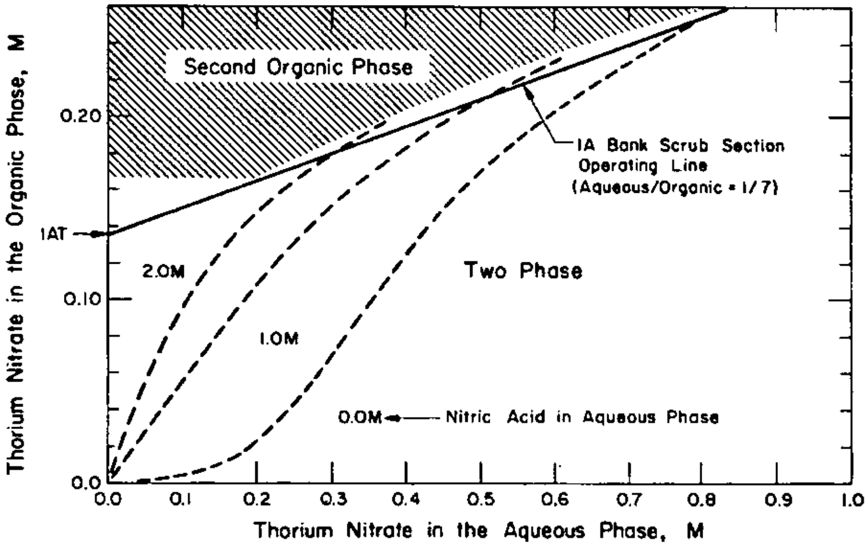

FIG. 19 1A BANK SCRUB SECTION OPERATING LINE OF THE DILUTE THOREX FLOWSHEET (30 vol % TBP - "ULTRASENE", 50°C)

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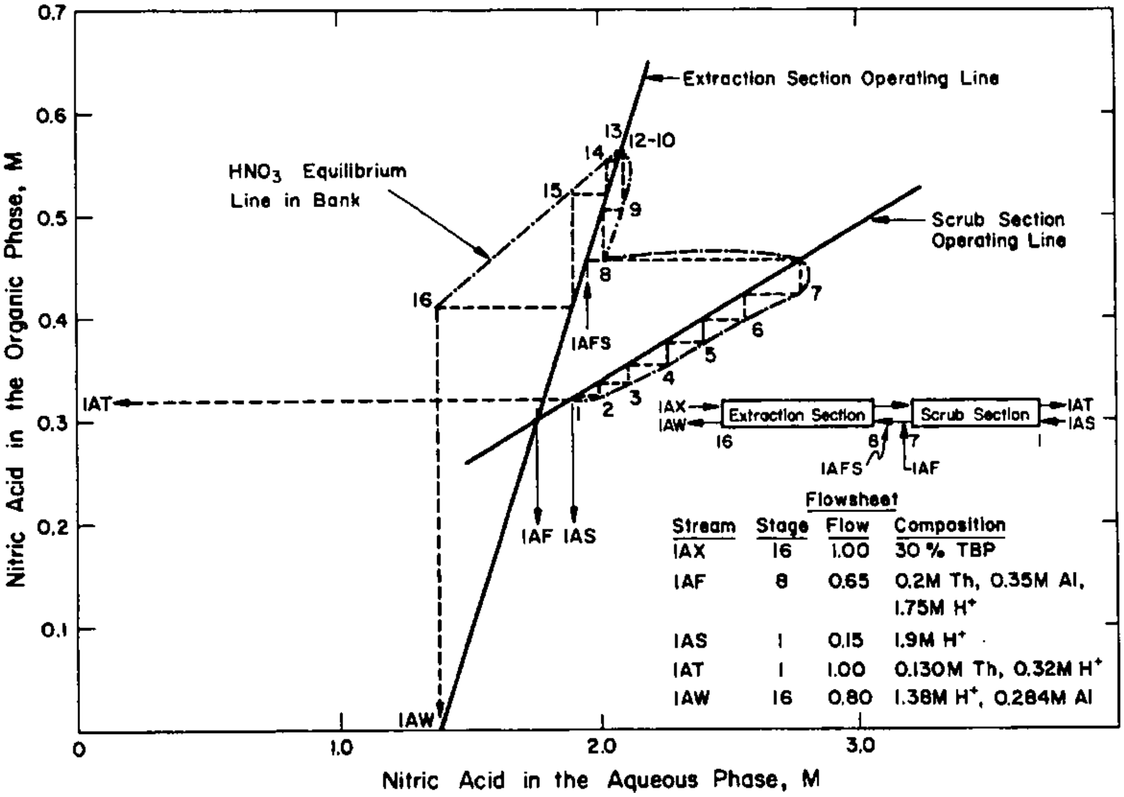

Flowsheet

Stream	Stage	Flow	Composition
IAX	16	1.00	30 % TBP
IAF	8	0.65	0.2M Th, 0.35M Al, 1.75M H^+
IAS	1	0.15	1.9M H^+
IAT	1	1.00	0.130M Th, 0.32M H^+
IAW	16	0.80	1.38M H^+, 0.284M Al

FIG. 20 DISTRIBUTION OF NITRIC ACID IN THE 1A BANK

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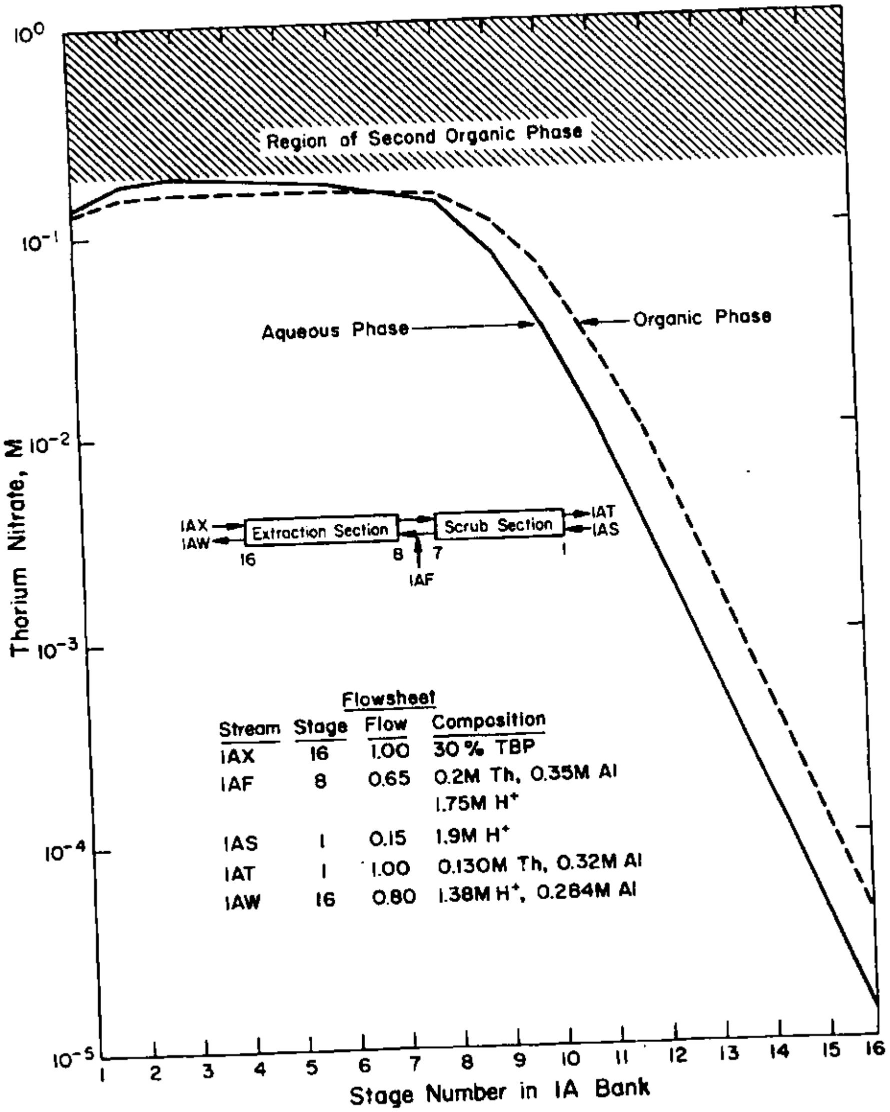

Flowsheet

Stream	Stage	Flow	Composition
IAX	16	1.00	30 % TBP
IAF	8	0.65	0.2M Th, 0.35M Al 1.75M H^+
IAS	1	0.15	1.9M H^+
IAT	1	1.00	0.130M Th, 0.32M Al
IAW	16	0.80	1.38M H^+, 0.284M Al

FIG. 21 THORIUM PROFILE OF THE 1A BANK

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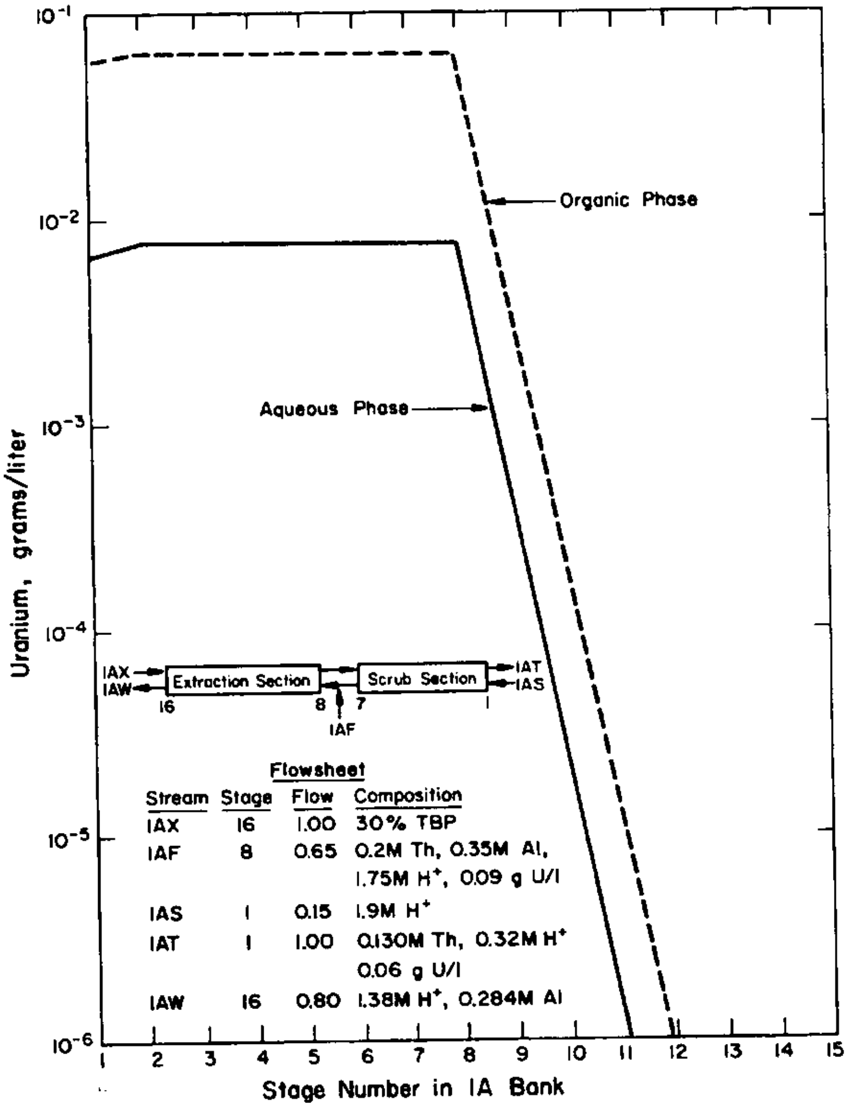

Flowsheet

Stream	Stage	Flow	Composition
IAX	16	1.00	30% TBP
IAF	8	0.65	0.2M Th, 0.35M Al, 1.75M H^+, 0.09 g U/l
IAS	1	0.15	1.9M H^+
IAT	1	1.00	0.130M Th, 0.32M H^+ 0.06 g U/l
IAW	16	0.80	1.38M H^+, 0.284M Al

FIG. 22 URANIUM PROFILE OF THE 1A BANK

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1B Bank

Thorium and uranium are partitioned in the 1B bank by controlling the salting (HNO_3) strength of the aqueous phase and the organic-to-aqueous flow ratio to exploit the differences in extraction factors for thorium and uranium. Acid concentration and flow ratios must be selected carefully to achieve efficient thorium-uranium partition without excessive reflux of uranium.

In designing the flowsheet for operation of the 1B bank, McCabe-Thiele calculations were first made to determine the effects of varying (a) the organic-to-aqueous flow ratios, (b) the acid concentrations of the organic feed (1AT), and (c) the acid concentrations of the aqueous extractant (1BX). Criteria for these calculations were:

- Cocurrent stage efficiency of $\geqslant 80\%$
- $\leqslant 1\%$ loss of ^{233}U to the thorium stream (1BT)
- $\leqslant 1\%$ loss of thorium to the uranium stream (1BU)
- ^{233}U concentration in the 1B bank $\leqslant 0.6$ g/l when processing $\leqslant 2000$ g/T of feed
- 1AS nitric acid concentration of 1 to 2M
- 1BF/1BS flow ratio = 5

Operating limits were then calculated as follows:

1. Preliminary calculations showed that most of the acid in the 1AT is stripped into the aqueous phase; the acid content of the aqueous phase in the uranium scrub section is therefore the sum of the acid in the 1AT and 1BX streams. Also, the acid content of the thorium stripping section is essentially that of the 1BX stream.
2. The thorium profile of the 1B bank was calculated on the basis of the nitric acid, thorium, and uranium distribution data. The flow and acid concentrations of the 1BX were varied until 1% of the thorium was lost to the uranium stream (1BU) under the most adverse conditions of TBP concentration (32 vol %) and stage efficiency (80%).

[redacted]

3. The distribution of uranium was calculated by considering the effects of salting by both the thorium and acid. As in Step 2 above, the 1BX flow and acidity were varied until the maximum concentration of uranium in the bank was 0.6 g/l, and the loss of uranium to the thorium stream (1BT) was 1%.

The operating limits for the 1B bank were derived from graphical representations of the results of these calculations, as explained below.

Reflux of uranium in the 1B bank depends on the 1BX/1BF flow ratio, the acid concentration of the 1BX, and the temperature. Decreasing either the 1BX/1BF flow ratio or the acid in the 1BX, or increasing the temperature will cause uranium to reflux to higher concentrations. Figure 23 shows this effect for a fixed 1BX/1BF flow ratio.

The flowsheet parameters considered in the above calculations affect the uranium-thorium partitioning and uranium reflux as shown in Figures 24, 25, and 26. Significant effects are:

- High acid in the 1AS* and 1BX and low temperature increase loss of thorium to the uranium stream, and reduce loss of uranium to the thorium stream.
- Low acid in the 1BX and high temperature (Figure 23) cause excessive reflux of uranium in the extraction section and high loss of uranium to the thorium stream.
- If the 1AS contains 1M HNO_3 (Figure 24), uranium and thorium cannot be partitioned when the temperature is greater than 63°C; at 2M HNO_3 (Figure 25), the maximum operating temperature is 69°C.

If the 1AS contains between 1 and 2M HNO_3, the maximum operating temperature is 55°C (Figure 26). The operating restrictions for 1M and 2M HNO_3 must thus be combined if the acid concentration is allowed to vary over the range 1-2M (see Figure 16).

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* All of the acid in the 1AS is extracted in the 1A bank (see Figure 20) and enters the 1B bank with the 1BF (1AT).

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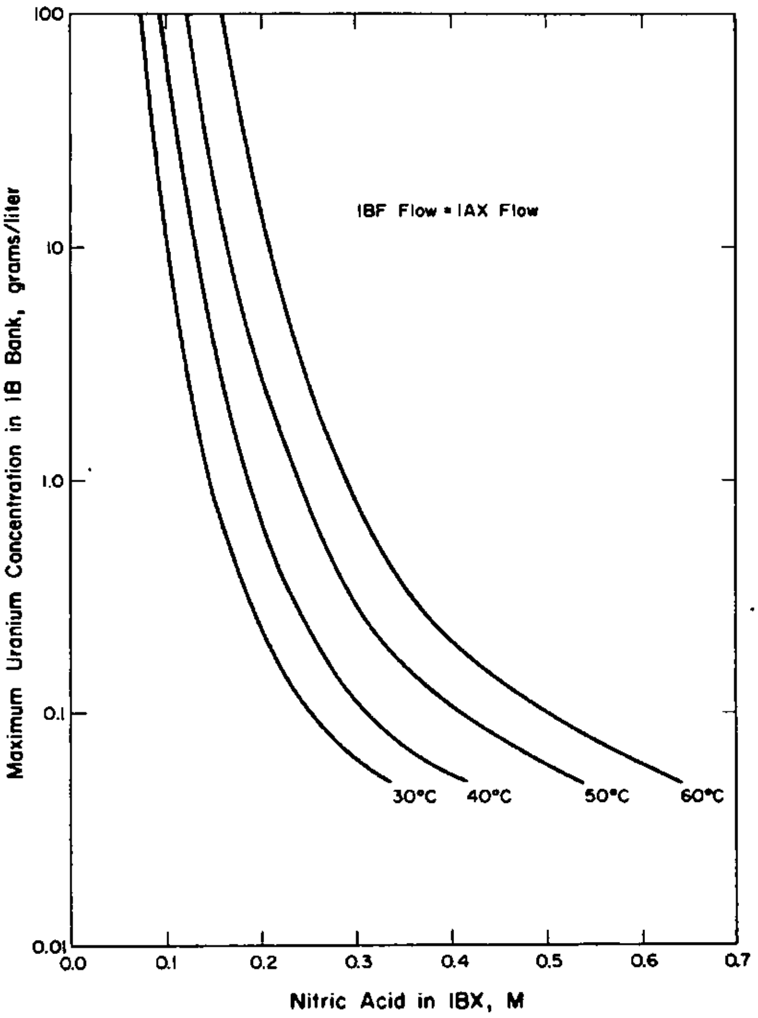

FIG. 23 EFFECT OF TEMPERATURE AND ACIDITY OF 1BX ON REFLUX OF URANIUM IN 1B BANK (1BX/1BF = 0.650 FLOW RATIO)

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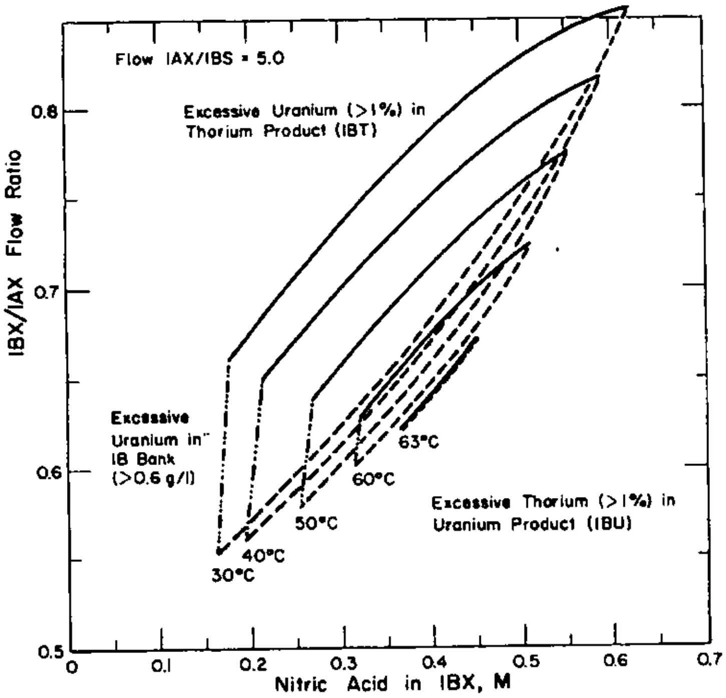

FIG. 24 EFFECT OF TEMPERATURE ON OPERATING LIMITS OF 1B BANK WHEN 1AS CONTAINS 1M HNO_3

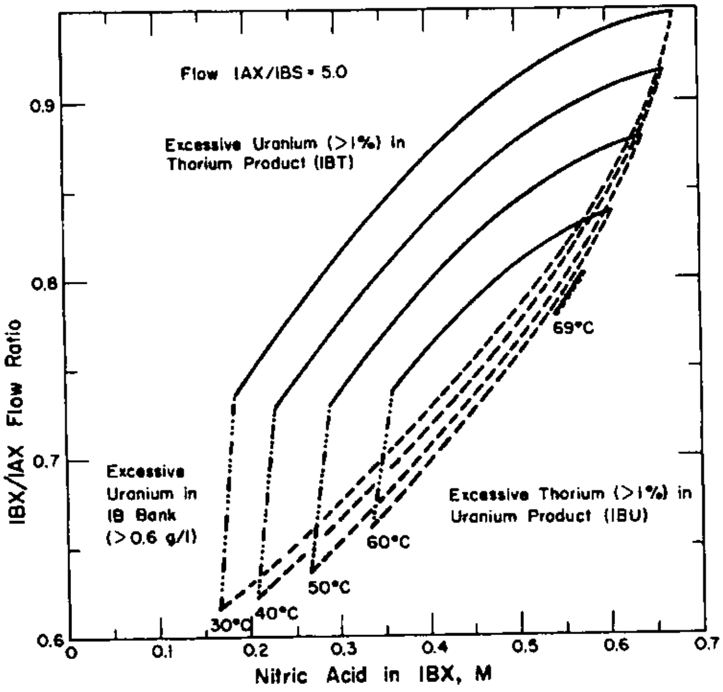

FIG. 25 EFFECT OF TEMPERATURE ON OPERATING LIMITS OF 1B BANK WHEN 1AS CONTAINS 2M HNO_3

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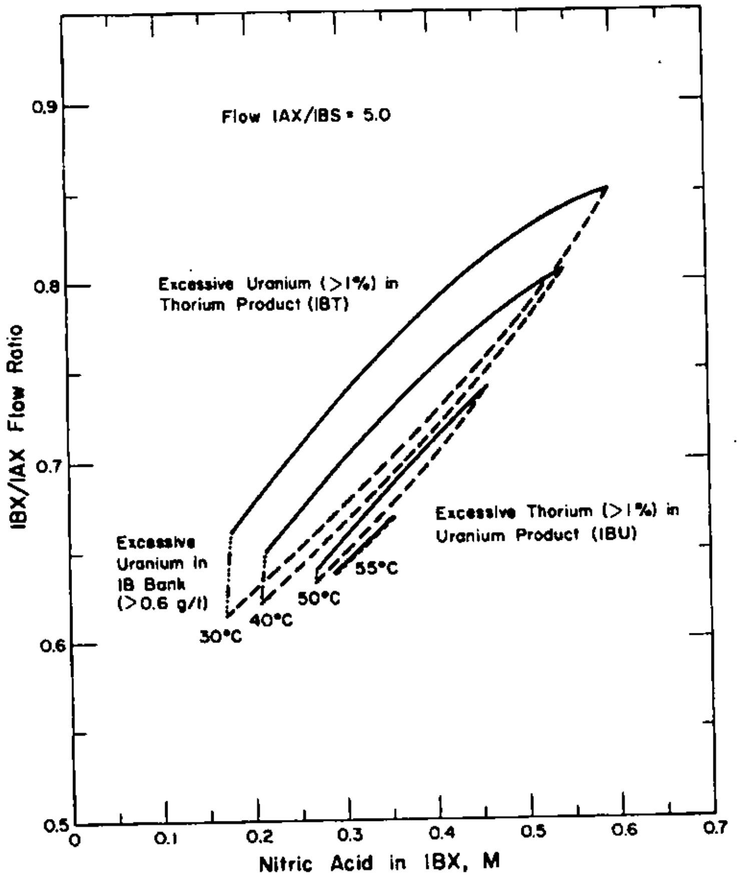

FIG. 26 EFFECT OF TEMPERATURE ON OPERATING LIMITS OF 1B BANK WHEN 1AS CONTAINS 1 TO 2M HNO_3

1C Bank

The operating limits for the 1C bank were calculated as a function of the 1BU/1CX flow ratio and the acid content of the 1BX to assure the uranium does not reflux in the 1C bank. Reflux of uranium is affected by the acid delivered to the 1C bank via the 1BU stream, and by the 1BU/1CX flow ratio. The acid content of the 1BU is controlled by the concentration of nitric acid used in the 1BX. To prevent reflux of uranium, the acid content of the 1CU, which determines the extraction coefficient for uranium (E_U) in the first strip stage, and the 1CX flow were set to give a uranium extraction factor $E^{o}_{aU}\cdot$(Flow Ratio 1BU/1CX) $\leqslant 1$. The results are presented in Table V and Figure 17.

[redacted]

[redacted]

Second Thorium Cycle

The thorium product stream (1BT) of the first cycle is evaporated and adjusted, and then processed by the flowsheet shown in Figure 27. Operating conditions for the second thorium cycle are shown in Table V.

Thorium and any uranium contaminant are extracted while the protactinium and fission products are discharged to the aqueous waste (2AW). The extracted thorium is scrubbed with HNO_3—Na_3PO_4 and is then stripped into dilute nitric acid in the 2B bank; the aqueous thorium solution (2BT) is evaporated and stored.

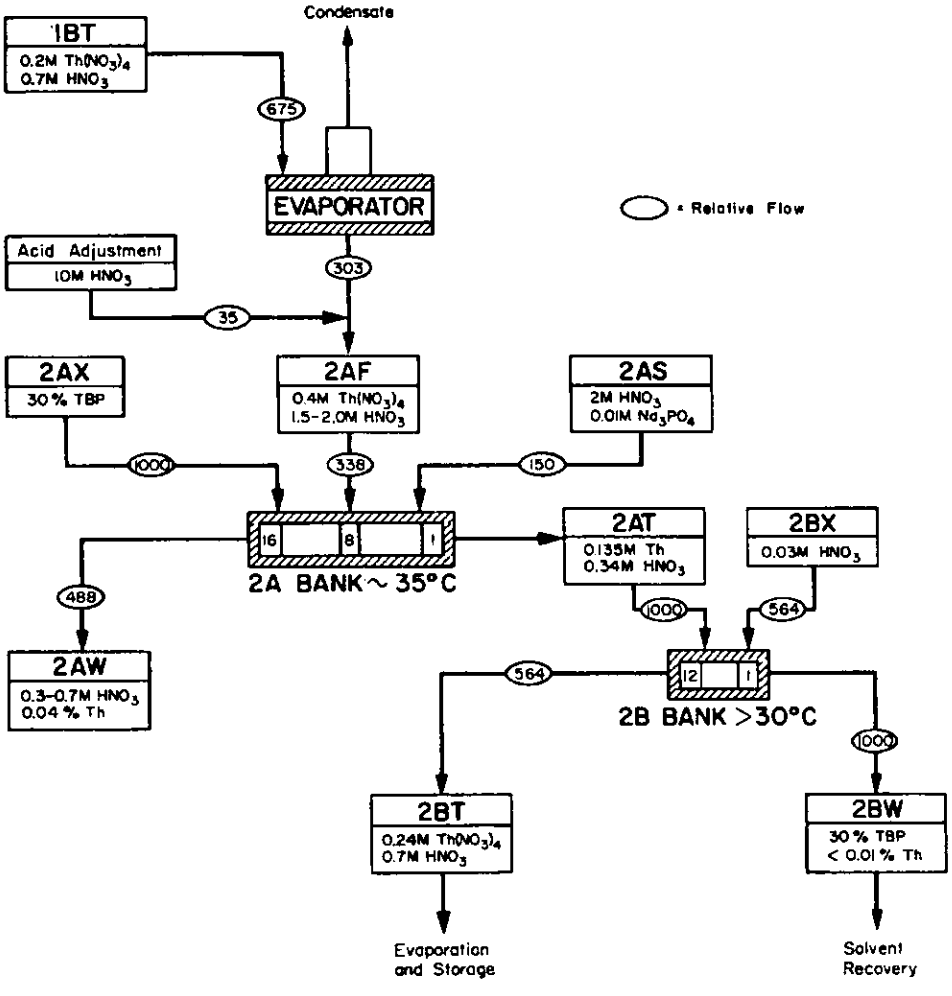

FIG. 27 SECOND THORIUM CYCLE

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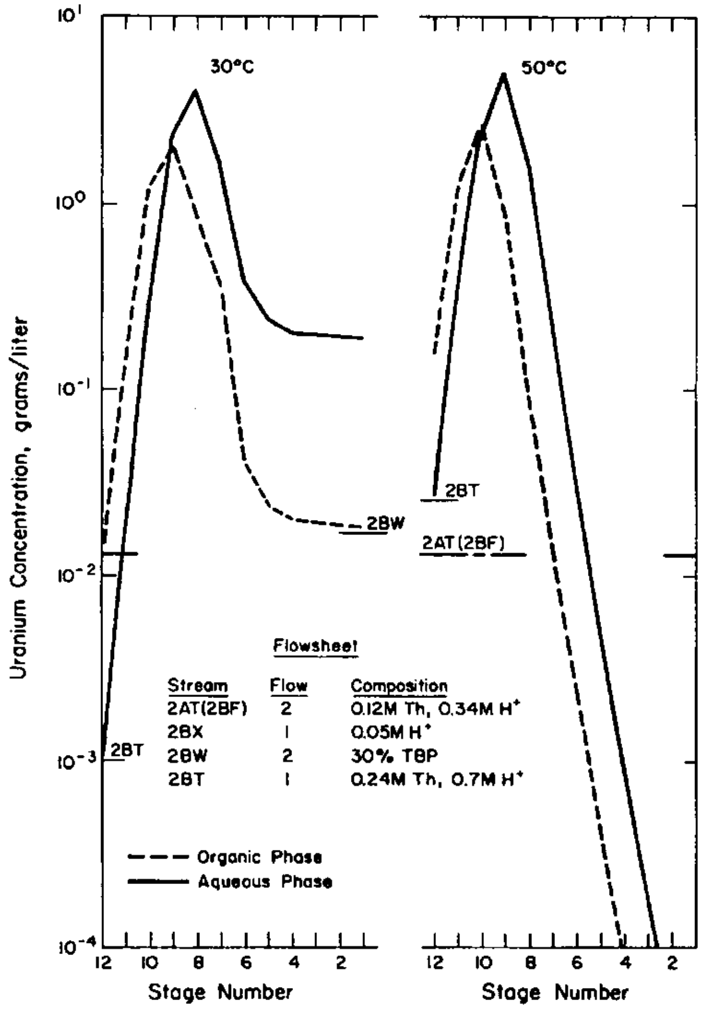
A key limit in the flowsheet is that the feed (2AF) should not contain more than 1% of the uranium present in the 1AF, because uranium refluxes in the 2B bank as a result of high concentrations of thorium and nitric acid in Stages 10, 11, and 12 and low concentration of nitric acid in Stages 1, 2, 3, and 4. For example, the extraction factor for uranium in Stage 12 is 10 whereas in Stage 1 it is 0.1. Typical uranium profiles are shown in Figure 28. At the specified operating temperature of 50°C, the inventory of ^{233}U in the 2B bank will not approach the maximum safe mass (510 g) unless more than 25% of the ^{233}U is lost to the thorium product stream of the first cycle (1BT). At an operating temperature of 30°C, the inventory of ^{233}U approaches the maximum safe mass if only 1% of the ^{233}U is lost to the 1BT stream.



Flowsheet

Stream	Flow	Composition
2AT(2BF)	2	0.12M Th, 0.34M H^+
2BX	1	0.05M H^+
2BW	2	30% TBP
2BT	1	0.24M Th, 0.7M H^+

FIG. 28 URANIUM PROFILE OF 2B BANK

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Second Uranium Cycle

The uranium product of the first cycle (1CU) is evaporated, adjusted, and then processed by the flowsheet shown in Figure 29. However, before evaporation, the uranium product stream from the first cycle (1CU) is washed with kerosene as a precaution to remove dissolved TBP. If the TBP is not removed, its degradation products formed during evaporation could precipitate ^{233}U or form uranium complexes that cannot be stripped from the organic phase in the subsequent solvent extraction step. The maximum allowable concentration of TBP in the kerosene wash was established as 0.2M on the basis of measurements of uranium extraction in the presence of DBP.(14,15)

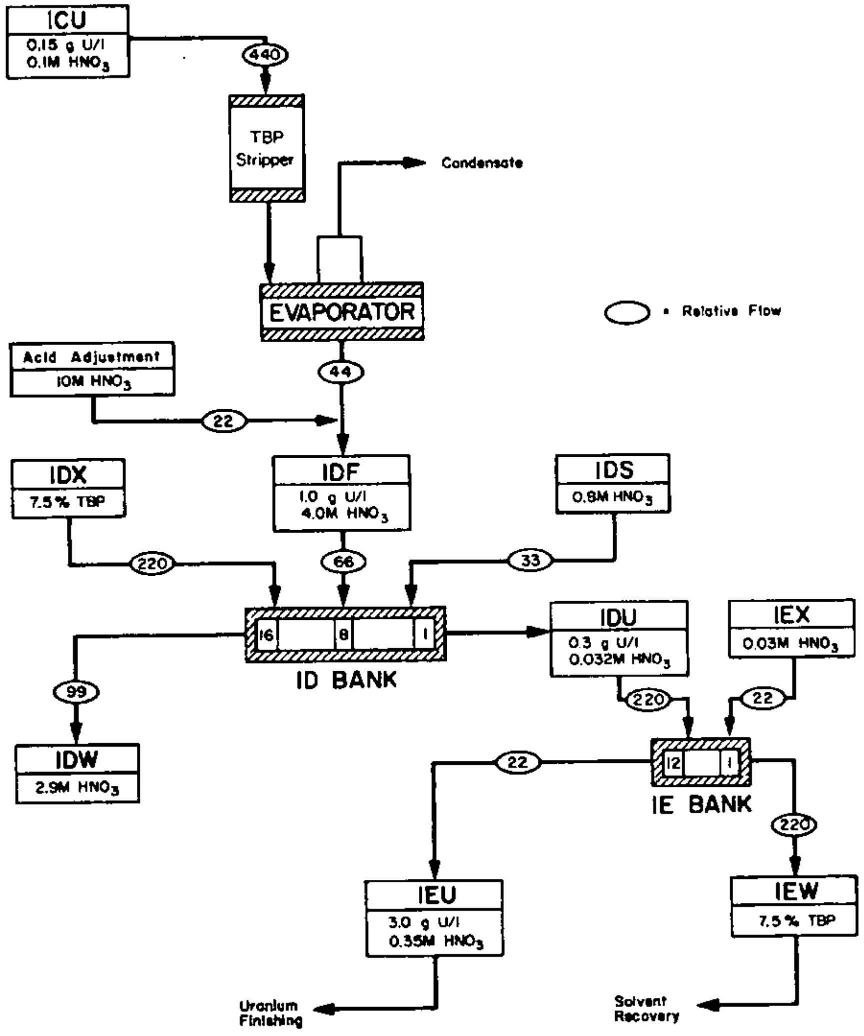

FIG. 29 SECOND URANIUM CYCLE

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In the 1D bank, uranium is separated from residual thorium, protactinium, and fission products by selective extraction from a nitric acid-salted aqueous solution into 7-1/2 vol % TBP. Fission products, protactinium, and thorium are rejected to the aqueous waste (1DW). In the scrub section, a nitric acid stream removes small quantities of fission products, protactinium, and thorium extracted into the uranium-bearing organic stream. In the 1E bank, uranium is stripped into a dilute nitric acid stream. The operating limits for the second uranium cycle are shown in Table V.

Startup and Shutdown Procedures

Thorium phosphate can precipitate in Stages 8, 9, and 10 of the 1A and 2A banks during startup and shutdown. The transient condition that causes precipitation is eliminated by conditioning the entire bank with nitric acid as follows:

- On startup, the extraction banks (1A and 2A) are operated with feed streams containing $\geq 0.5M$ HNO_3 instead of the thorium feed.
- On shutdown, feed streams containing $\geq 2M$ HNO_3 are continued until thorium is flushed from the system.

URANIUM FINISHING (Figure 30)

The ^{233}U product from solvent extraction (1EU) is concentrated by cation exchange, precipitated as ammonium uranate, and calcined to uranium trioxide.(18a)

Cation Exchange

The uranyl ion is absorbed and eluted from sulfonic acid cation exchange resin by the following reaction:

$$UO_2^{2+} + 2HR \underset{\text{elution}}{\overset{\text{absorption}}{\rightleftharpoons}} UO_2R_2 + 2H^+$$

where R represents the resin anion, and HR is the resin in the acid form. The capacity of the resin for uranyl ion depends on the composition of the solution that is in contact with the resin. With solutions containing only uranium and nitric acid, the percentage of the resin exchange sites that can absorb uranyl ions (designated S_u) is related to the acid and uranium concentrations in the feed, as shown in Figure 31.(18b) "Dowex"* 50W-X8 resin

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* Trademark of Dow Chemical Co.

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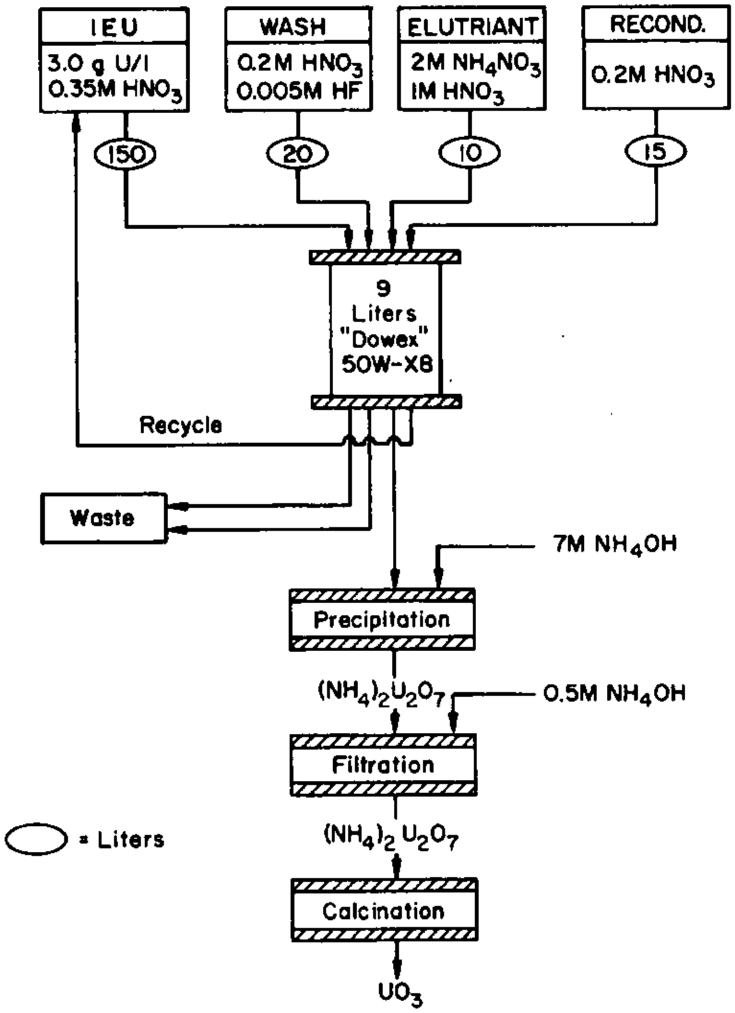

FIG. 30 URANIUM FINISHING OPERATIONS

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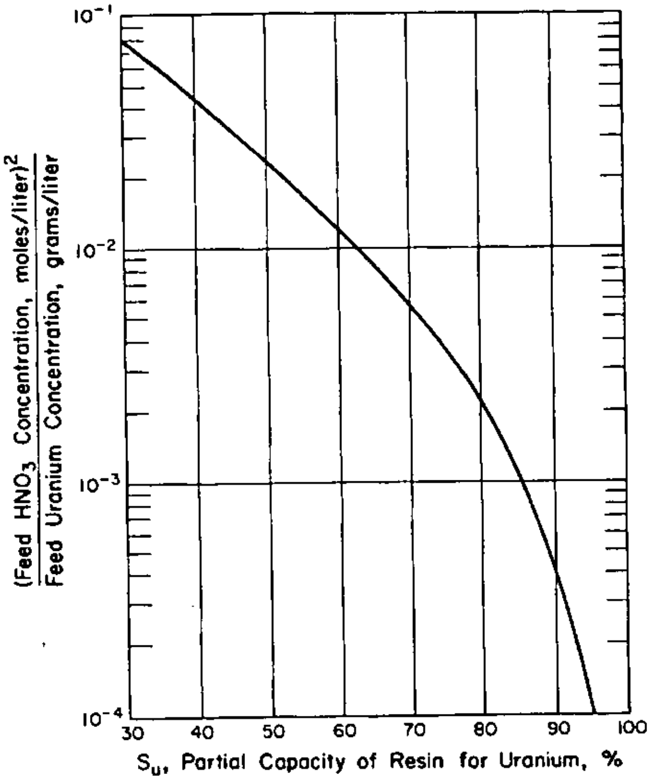

FIG. 31 ABSORPTION OF URANIUM ON "DOWEX" 50W-X8 ION EXCHANGE RESIN

(total capacity: 244 grams of uranium per liter of settled resin) absorbs about 122 grams of uranium per liter of settled resin from a feed solution containing 0.3M HNO_3 and 3 g of U/l; the value of S_u in this case is 50%.

Thorium in the feed decreases absorption of uranium because the thorium is more strongly absorbed and occupies some of the exchange sites of the resin. Data indicate that the thorium will accumulate on the resin and can be eluted with sodium bisulfate or nitric acid solutions. Solutions containing either 1M $NaHSO_4$ or 4.5M HNO_3 are considered optimum elutriants, with the sodium bisulfate more effective than nitric acid.

The absorbed uranium can be eluted with ~2 column volumes of ammonium nitrate solution. A solution of 2M NH_4NO_3—1M HNO_3 was chosen as elutriant, the small quantity of nitric acid being required to prevent hydrolysis of the uranyl ion.

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Uranium Precipitation

Uranium in the ion exchange eluant is precipitated as ammonium uranate by adding ammonium hydroxide.(17,18,19) Precipitation begins at pH 3.2-3.6 and is complete at pH 7.5-8.0. The solubility of uranyl ion at pH 8.0 is $\leqslant$0.1 mg of U/l.

Conversion to Uranium Oxide

Ammonium uranate is air-dried and then decomposed to uranium trioxide by heating to ~550°C. Thermogravimetric analyses indicate that the desired UO_3 product is stable between 450 and 600°C. Decomposition of the uranate is incomplete below 450°C, and conversion to U_3O_8 occurs above 600°C.

THORIUM CONCENTRATION AND STORAGE

The thorium product (2BT) of the second cycle is concentrated to 2.5M $Th(NO_3)_4$ by evaporation and stored to permit decay of ^{234}Th and other residual contaminants. To prevent crystallization during storage at normal ambient temperature (~20°C), the nitric acid must be steam-stripped to $\leqslant$0.25M HNO_3. The solubility of thorium nitrate in nitric acid is shown in Figure 7.

PROCESS PERFORMANCE

The solvent extraction processes* were demonstrated in miniature mixer-settler(22) tests using feed solutions prepared by dissolving irradiated thorium oxide. Adequate removal of ^{233}Pa by sorption on manganese dioxide was also an integral part of the demonstration. Compositions before and after ^{233}Pa sorption on manganese dioxide are summarized in Table VI. The sorption of ^{233}Pa was less than the expected value of 95% because the filter that was used in the tests did not retain the manganese dioxide completely; the net removal was 80 to 85%. Higher sorption was achieved in separate small-scale tests in which the manganese dioxide slurry was centrifuged. These results, summarized in Table VII, show that sorption of ^{233}Pa exceeded 97% even with a fourfold variation of the quantity of manganese dioxide.

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* The demonstrations of the dissolving and finishing procedures are reported in References 7 and 16a, respectively.

[redacted]

TABLE VI

Compositions of Feed Solutions

	Dissolver Solutions 1	2	3
$Th(NO_3)_4$, M	0.40	0.40	0.40
$Al(NO_3)_3$, M	0.7	0.7	0.8
HNO_3, M	0.02	0.25	0.47
^{233}U, g/l	0.3	0.3	0.3
MnO_2/Th	0.053	0.053	0.053
Before Sorption of ^{233}Pa			
^{233}Pa, 10^8 dis/(sec)(ml)	120	110	62
^{103}Ru-^{103}Rh, 10^6 dis/(sec)(ml)	-	-	10
^{95}Zr-^{95}Nb, 10^5 dis/(sec)(ml)	330	-	440
After Sorption of ^{233}Pa			
^{233}Pa, 10^8 dis/(sec)(ml)	1.2	16	12
^{103}Ru-^{103}Rh, 10^6 dis/(sec)(ml)	9.1	-	4.0
^{95}Zr-^{95}Nb, 10^5 dis/(sec)(ml)	2.5	200	280

TABLE VII

Sorption of ^{233}Pa* and ^{95}Zr-^{95}Nb

Feed Solution: 0.4M Th^{4+}, 0.72M Al^{3+}, 0.5M HNO_3, 4.2×10^{10} dis ^{233}Pa/(sec)(ml), and 4.0×10^7 dis ^{95}Zr-^{95}Nb/(sec)(ml).

	Decontamination Factor$^{(a)}$			
	^{233}Pa		^{95}Zr-^{95}Nb	
MnO_2/Th Molar Ratio	1 hr	3 hr	1 hr	3 hr
0.053	706	632	2.7	4.0
0.026	194	100	2.8	2.7
0.013	67	43	2.4	2.3

---

* Test slurries centrifuged 1 and 3 hr after precipitation of MnO_2.

[redacted]

The first-cycle test conditions, results, and the effects of variations among the different tests are shown in Table VIII. The following conclusions were drawn from these results and from additional observations and measurements:

- Uranium and thorium were extracted satisfactorily in all tests; losses were small, and cross-contamination of the two product streams was within acceptable limits of $\leqslant 1\%$ ^{233}U in the thorium and $\leqslant 1\%$ thorium in the uranium. Other data verified that the maximum concentration of thorium in the organic phase in the 1A bank was identical to the calculated values, indicating that the flowsheet was properly designed to prevent formation of a <u>third phase</u>. The reflux of ^{233}U in the 1B bank at the normal operating temperature of 35°C was small; however reflux of ^{233}U in this bank was excessive (>0.6 g/l) at elevated temperatures.

- Although removal of ^{233}Pa was satisfactory in all tests, the reflux of ^{233}Pa in the extraction section of the 1A bank with concentrated feeds (Figure 32, Test 1D) would degrade the solvent excessively in the much larger plant mixer-settlers in which the fraction of radiation absorbed is 50 times greater than in the miniature mixer-settlers. Dilution of the feed eliminated the reflux, as illustrated in Figure 32, Tests 2C and 2D.

- Zirconium-niobium extraction was almost identical with that of ^{233}Pa, and the reflux was very similar.

- Removal of ruthenium was low, with decontamination factors of only 50-400, except in Tests 1A-1D (Table VIII), for which the feed preparation steps involved abnormally low nitric acid concentration and in Tests 3C-3D for which the 1A bank was operated at higher temperature. The stage concentrations indicated that 5 to 10% of the ruthenium was extracted in the 1A bank but was not separated in the scrub section of the 1A bank. This extracted ruthenium was then distributed erratically among the streams of thorium (1BT), uranium (1CU), and unwashed solvent (1CW). The erratic behavior of ruthenium indicates that its removal in the first cycle from the thorium (1BT) by factors exceeding 50 cannot be expected.

- Activity was readily removed from the used solvent (1CW) by Na_2CO_3—NaOH solvent wash solution. In batch washing tests, residual solvent activity after a single wash was only 6000 counts/(min)(ml), which is considered adequate for reuse of the solvent as 1AX.

[redacted]

[redacted]

TABLE VIII

First-Cycle Test Conditions and Results

Underlined values denote principal variations.

	Dissolver Solution 1				Dissolver Solution 2				Dissolver Solution 3		
	Test 1A	1B	1C	1D	2A	2B	2C	2D	3B	3C	3D
Composition of 1AF											
$Th(NO_3)_4$, M	0.40			0.40	0.40	0.40	0.20	0.20	0.40		
$Al(NO_3)_3$, M	0.67			0.69	$\underline{0.75}$	$\underline{0.75}$	$\underline{0.48}$	$\underline{0.51}$	0.93		
HNO_3, M	0.35			0.33	$\underline{0.35}$	$\underline{0.94}$	$\underline{0.42}$	$\underline{1.1}$	1.1		
U, g/l	0.31			0.32	0.29	0.30	0.17	0.15	0.28		
^{233}Pa, 10^7 d/(sec)(ml)	6.3			$\underline{12}$	$\underline{120}$	96	120	94	61		
^{103}Ru-^{103}Rh, 10^6 d/(sec)(ml)	10			9.1	9.6	9.4	5.2	5.1	4.0		
^{95}Zr-^{95}Nb, 10^6 d/(sec)(ml)	1.7			1.9	20	12	17	11	20		
1AF Flow (Figure 14)	$\underline{270}$	$\underline{320}$	$\underline{338}$	$\underline{338}$	338	338	676	676	338		
Temperature, °C	35				35	35	35	35	35	45	55
Loss of ^{232}Th, %											
1AW	0.03				0.03	0.03	0.03	0.02	0.01	-	-
1CU	0.1				0.03	0.13	0.35	0.39	0.1	0.1	0.1
Loss of ^{233}U, %*											
1BT	0.2				0.2	0.2	0.2	0.2	0.3	0.3	0.6
1CW	0.03				0.04	0.07	0.03	0.03	0.02	0.03	0.04
Removal of activity from thorium stream (1BT), DF**											
^{233}Pa	2 x 10^4				6 x 10^4	4000	7 x 10^4	1 x 10^4	1200	1300	1500
^{103}Ru-^{103}Rh	750				200	75	50	100	50	1000	3500
^{95}Zr-^{95}Nb	1 x 10^4				1 x 10^4	2300	1.6 x 10^4	>1.3 x 10^4	1000	1500	750
Removal of activity from uranium stream (1CU), DF**											
^{233}Pa	2 x 10^4				>3 x 10^3	>2000	>10^5	6 x 10^4	1.6 x 10^4	2.6 x 10^4	1 x 10^5
^{103}Ru-^{103}Rh	1500				750	400	100	150	50	1500	1 x 10^4
^{95}Zr-^{95}Nb	1 x 10^5				1.9 x 10^5	1.7 x 10^5	2.6 x 10^6	>4 x 10^6	2 x 10^4	7 x 10^4	2 x 10^5

---

* Loss of ^{233}U to the 1AW stream was less than loss of Th.

** Ratio, (Activity in 1AF)/(Activity in product stream).

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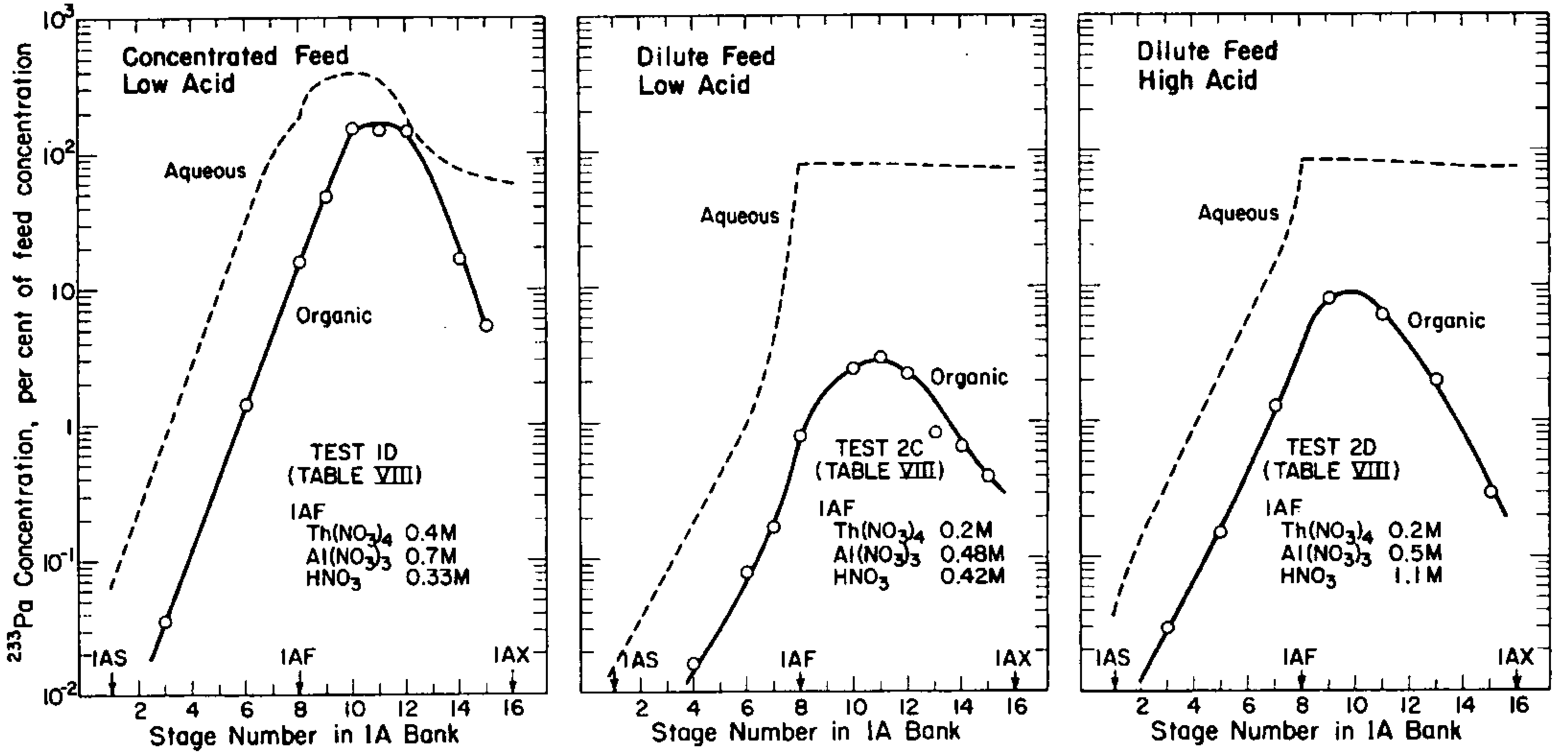

FIG. 32 PROTACTINIUM PROFILES IN 1A BANK

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Test conditions for the second thorium cycle are shown in Figure 33. The results summarized in Table IX illustrate the highly beneficial effect of elevated temperature for removing ruthenium; a tenfold improvement was obtained by increasing the temperature from 35 to 55°C. Removal of ^{95}Zr-^{95}Nb was also better at the high temperatures, but the removal of ^{233}Pa was not affected.

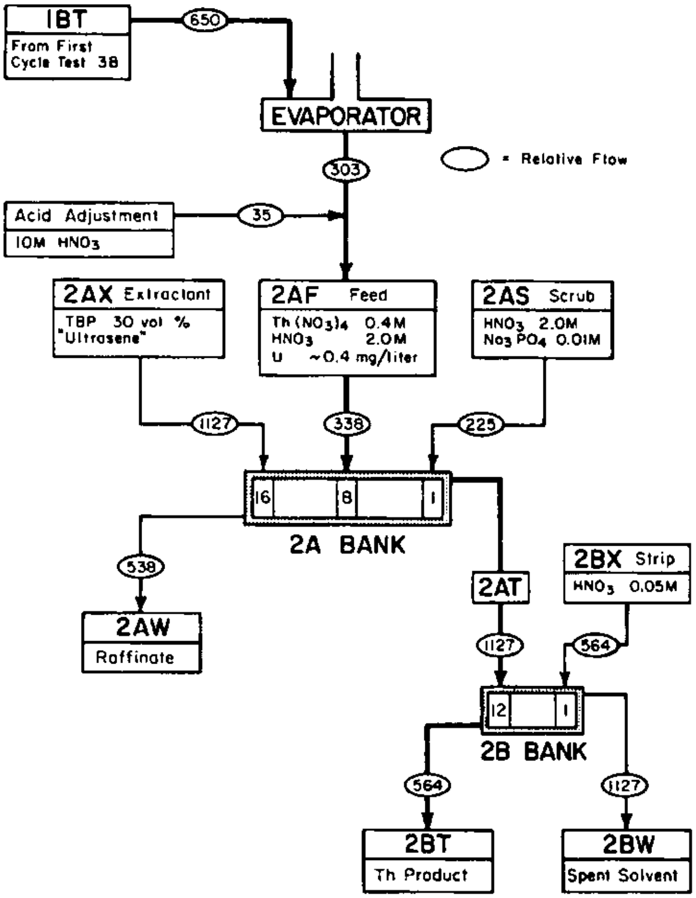

FIG. 33 SECOND THORIUM CYCLE TEST CONDITIONS

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The overall purification results for the thorium product, which are also summarized in Table IX, indicate that only the removal of ruthenium is marginal for providing a highly purified thorium oxide product (a decontamination factor of 1×10^4 is required for the removal of ruthenium, as noted in Table I); the overall removals of ^{233}Pa and ^{95}Zr-^{95}Nb are about ten times the necessary values.

TABLE IX

Second Thorium Cycle Test Results

Temperature	35-40°C	40-50°C	50-55°C
Loss of Th to 2AW, %	0.02	0.07	0.13
Removal of activity in second cycle, DF*			
^{233}Pa	2300	2800	2200
^{103}Ru-^{103}Rh	23	60	200
^{95}Zr-^{95}Nb	12	50	100
Overall removal of activity, DF**			
^{233}Pa			1.2×10^7
^{103}Ru-^{103}Rh			1×10^4
^{95}Zr-^{95}Nb			2×10^5

---

* Ratio, (Activity in 2AF)/(Activity in 2BT)

** Ratio, (Activity in dissolver solution)/(Activity in 2BT from Test 3B).

No tests of the second uranium cycle were made, since the conventional (7-1/2 vol % TBP) conditions of the normal ^{235}U process would be used in the plant.(3) The satisfactory purification of the uranium product stream (1CU) from ^{233}Pa and ^{95}Zr-^{95}Nb in the first cycle and good plant experience with removal of ruthenium in the ^{235}U process indicated that ^{233}U of adequate quality would be obtained.

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DP-1036, RECOVERY OF THORIUM AND URANIUM-233 FROM IRRADIATED THORIUM OXIDE AND METAL

by W. E. Prout and A. E. Symonds

The attached report is a detailed description of the development of the process that was used in the Savannah River separations plant to recover both thorium and ^{233}U from irradiated thorium oxide and metal. The development work was performed in support of the first large-scale separations campaign for simultaneous Th-U recovery and purification, which was carried out at Savannah River in H Area during the period September 1965 to January 1966. During this campaign, ~150 kg of ^{233}U with <6.5 ppm ^{232}U was separated from ~88 tons of thorium oxide and ~17 tons of thorium metal. Despite difficult control problems, process performance was very satisfactory.

This report is concerned primarily with the solvent extraction steps of the process, the development of which is based partly on earlier work at KAPL and ORNL. The detailed developments of other steps of the process are reported in References 5, 6, 7, and 16a cited in the bibliography to this report.

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