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**LABORATORY STUDIES ON THE USE OF URANIUM(IV)
AS A PLUTONIUM REDUCTANT IN A PUREX PROCESS**

J. E. MENDEL

JUNE, 1965

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for Plutonium and Uranium

LABORATORY STUDIES ON THE USE OF URANIUM(IV)
AS A PLUTONIUM REDUCTANT IN A PUREX PROCESS

By
J. E. Mendel

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LABORATORY STUDIES ON THE USE OF URANIUM(IV) AS A PLUTONIUM REDUCTANT IN A PUREX PROCESS

INTRODUCTION

This report describes laboratory-scale studies that evaluate the feasibility of using uranium(IV)-hydrazine instead of ferrous sulfamate-sulfamic acid as the partitioning agent in the Hanford Atomic Products Operation Purex separations plant. Iron(II), in combination with sulfamic acid as a holding reductant, has been used very successfully for many years as a plutonium reductant in solvent extraction separation processes for irradiated nuclear fuels. However, the iron remains in the radioactive wastes produced in the separation processes. Its presence in these wastes accelerates corrosion of waste concentration equipment, complicates treatment of the waste for by-product recovery, and adds to the total quantity of contaminated salts which must be stored.

Much effort has gone into searches for plutonium reductants which would not have the disadvantages of iron(II). Uranium(IV) has been proposed since, having acted as reductant, it is extracted along with uranium from the irradiated fuels and does not remain in the waste. Previous studies on the use of uranium(IV) in solvent extraction separations processes have involved mixer-settler contactors.^(1, 2) The HAPO Purex plant has pulsed-column contactors. This difference, plus certain problems due to the particular flowsheet conditions used, required additional laboratory studies to determine the feasibility of using uranium(IV) in the HAPO Purex plant. These are described in the succeeding sections.

SUMMARY AND CONCLUSIONS

A flowsheet was proposed for a plant production test of uranium(IV)-hydrazine instead of ferrous sulfamate-sulfamic acid as partitioning agent in HAPO Purex partition column. In this flowsheet, an aqueous stream containing uranium(IV) is contacted with Purex solvent and both phase are introduced into the 1BX column at the feed point. Hydrazine is added

to the 1BX column feed accumulation tank to destroy nitrite present in the organic feed stream. A hydrazine-nitric acid stream is introduced at the top of the 1BX column instead of the usual ferrous sulfamate-sulfamic acid stream. The solvent phase leaving the 1BS column is routed to the 1BX feed tank instead of to the co-decontamination cycle. Otherwise, the stream compositions and flows remain as at present.

These laboratory studies indicated that satisfactory operation of the 1B column should be obtained with the proposed flow sheet. Uranium(IV) was shown to be adequately stable in Purex solvent to allow reasonable hold-up time in the vessel in which the feed stream is made up. Plutonium reduction was sufficiently rapid and complete under 1BX extraction conditions to assure adequate separation of plutonium from the product uranium stream. Although some re-oxidation and re-extraction of plutonium in the 1BS column (scrub section) was anticipated, the results indicate that the plutonium reflux will be no greater than that which occurs with the present flowsheet. Extraction of uranium(IV) in the 1BS column should be adequate to insure acceptably low uranium in the product plutonium stream, particularly if high acidity ($4M\ HNO_3$) is maintained through addition of the 1BXP- HNO_3 stream.

FLowsheet Considerations

The Hanford Atomic Products Operation Purex 1B column is actually two concatenated columns locally designated as 1BX column and 1BS column. For purposes of discussion the columns can be considered a single compound 1B column in which the Purex 1BX column functions as the extraction section and the 1BS column functions as the scrub section. Figure 1 shows the present 1B or partition column flowsheet in solid lines. Present Purex nomenclature for the various entering and leaving streams is used.

The feed stream to the 1B column (1BXF) is Hanford Purex process solvent (30 vol% TBP in Soltrol) containing uranium, plutonium and trace amounts of impurities. The extractant or partitioning agent (a ferrous sulfamate-sulfamic acid solution) enters the top of the 1BX column as the 1BX stream. The 1BXP- HNO_3 stream is not necessary for operation of the 1B column. It is a plant stream containing a small amount of uranium, recovery of the uranium by adding the stream to the 1BX column is convenient.

FIGURE 1

Partition Cycle Flowsheet

Modifications to the flowsheet, proposed by HAPO Purex Process Engineering personnel to accommodate uranium(IV)-hydrazine instead of ferrous sulfamate-sulfamic acid as partitioning agent, are shown in dotted lines in Figure 1. Addition of the uranium(IV) as an aqueous stream above the 1BX feed point is not possible in the existing equipment because there is no feed point available. Addition of uranium(IV) as an aqueous stream at the existing feed point was considered not operable because uranium(IV) which followed the aqueous phase into the 1BS column would be extracted into the solvent and not returned to the column until after a long residence time in the feed tank. The alternate chosen was the introduction of uranium(IV) in a combined aqueous and solvent stream with most of the uranium(IV) in the solvent.

Since the 1BXF stream contains a small amount of nitrite, and nitrite oxidizes uranium(IV) rapidly, the modified flowsheet includes addition of hyrazine to the feed tank to destroy the nitrite. In the modified flowsheet, the solvent stream from the 1BS column is added to the 1BX feed tank instead of being routed back to the coextraction cycle.

The following sections describe laboratory studies made to evaluate the feasibility of the modified flowsheet.

EXPERIMENTAL PROCEDURES AND DISCUSSION OF RESULTS

Uranium(IV)

Preparation

Uranium(IV) used in these studies was prepared from uranyl nitrate solution obtained from the HAPO Redox plant. In most cases, reduction of uranium(VI) to uranium(IV) was done with aluminum metal dust. In some cases reduction was by sodium formaldehyde sulfoxalate⁽³⁾ or by formic acid catalyzed by platinum dispersed on alundum⁽⁴⁾. The method of preparing uranium(IV) has process significance because in the flowsheet proposed, the aqueous phase in which uranium(IV) is produced enters the extraction column.

Analysis

Uranium(IV) was determined by two procedures: (1) a direct spectrophotometric method⁽⁵⁾ and (2) a method in which uranium(IV) reduces iron(III) to iron(II) which is then measured⁽⁶⁾. The former procedure has a limit of detection of about 0.001M and requires correction for nitric acid concentration. The latter has a lower limit of less than 5×10^{-6} M and does not require correction for nitric acid. Both procedures were also used for analysis of uranium(IV) in 30% TBP-Soltrol solutions.

Liquid-liquid Contacting

Batch Contacting

Kinetic considerations were of primary importance in these studies and it was important to duplicate column conditions as closely as possible. Therefore, for the batch contact, the phases were dispersed with a reciprocating plunger equipped with sieve-plates and operating at 60 cpm.

TABLE I
URANIUM(IV) STABILITY IN 30% TBP-SOLTROL** AT 25 C

Run no.		Method of Uranium(IV) Preparation	Percent Theoretical Uranium(IV) Remaining*					
			Approx 5 min	1 hr	2 hr	5 hr	7 hr	24 hr
1	Organic Separated from Initial Aqueous, Air Sparged†	DeLeone	62.5	69.1	78.1	65.5	57.4	12.1
5	Organic Separated from Initial Aqueous, Air Sparged	Al-reduced	75.2	----	56.3	61.6	----	28.7
9	Organic Separated from Initial Aqueous, Air Sparged	Discolite	56.2	----	35.8	33.3	----	11.1
2	Organic in Contact with Initial Aqueous, Air Sparged	DeLeone	73.4	74.5	75.7	83.0	81.8	5.3
6	Organic in Contact with Initial Aqueous, Air Sparged	Al-reduced	64.2	----	44.9	36.5	----	0.0
10	Organic in Contact with Initial Aqueous, Air Sparged	Discolite	82.1	----	84.4	84.8	----	103.4
3	Organic Separated from Initial Aqueous, N ₂ Sparged	DeLeone	80.1	68.9	68.4	66.4	61.5	20.0
7	Organic Separated from Initial Aqueous, N ₂ Sparged	Al-reduced	65.5	----	46.6	20.9	----	0.0
11	Organic Separated from Initial Aqueous, N ₂ Sparged	Discolite	79.5	----	85.1	82.5	----	84.8
4	Organic in Contact with Initial Aqueous, N ₂ Sparged	DeLeone	69.8	----	60.6	67.2	----	0.0
8	Organic in Contact with Initial Aqueous, N ₂ Sparged	Al-reduced	56.3	----	59.8	25.9	----	0.0
12	Organic in Contact with Initial Aqueous, N ₂ Sparged	Discolite	79.3	----	85.5	94.5	----	108.6
13	Organic in Contact with Initial Aqueous, Air Sparged	DeLeone	67.3	----	76.7	68.6	----	61.5
14	Organic in Contact with Initial Aqueous, Air Sparged	Al-reduced	106.4	----	88.6	108.2	----	0.7
15	Organic in Contact with Initial Aqueous, Air Sparged	Discolite	72.3	----	49.0	35.2	----	0.6

* Theoretical initial concentrations were 0.023, 0.026, and 0.025M, for runs with DeLeone, Al-reduced, and Discolite prepared Uranium(IV), respectively.

† Sparge rate = 200 ml/min in all cases

** 30% TBP-Soltrrol was laboratory carbonate-washed and allowed to stand about 2 mo, Runs 1 through 9; laboratory carbonate-washed and used immediately, Runs 10 through 14, and Purex R-7 (aged 2 mo), Run 15.

The apparatus was jacketed, and controlled temperature water was circulated through the jacket.

Continuous Counter Current Contacts

A miniature pulse column of the type devised by Koski⁽⁷⁾ was used to simulate the 1BX column. A few runs were made, but were discontinued when it became apparent (as described later) that the organic phase residence time was too short to properly duplicate plant conditions.

Stability of Uranium(IV) in Solvent

One concern about the proposed flowsheet was the stability of uranium(IV) when extracted into solvent, since the solvent will have a finite residence time in the makeup tank (Tank J-2). Tests were conducted to determine the rate of oxidation of uranium(IV) under conditions which will prevail in Tank J-2. These data, presented in Table I, show that the residence time in Tank J-2 can be at least 7 hr and considerable longer if good quality solvent is used. Other conclusions which may be drawn from these data are as follows:

- About 30% of the uranium(IV) was oxidized almost immediately on contact with solvent.
- Oxidation of uranium(IV) was less rapid when it had been prepared by the DeLeone process than when it had been prepared with aluminum as the reductant. Residual reducing capacity in the aqueous phase probably accounts for the difference noted.
- Oxidation of uranium(IV) was less rapid when the solvent used had been washed recently with carbonate solution.
- Blanketing the mixture with nitrogen did not decrease the rate of oxidation of uranium(IV) as compared to an air blanket. Removing the aqueous phase after the uranium(IV) was extracted into the solvent did not reduce the uranium(IV) oxidation rate.

Reduction of Plutonium in the 1BX Column

The reduction of plutonium(IV) to plutonium(III) by uranium(IV) is rapid, and only a small excess of uranium(IV) is normally required to attain complete reduction of plutonium.⁽⁸⁾ The following batch contact experiments were performed to compare the effectiveness of uranium(IV) and ferrous sulfamate (FS) as partitioning agents under 1BX column operating conditions. Equal volumes of synthetic 1BX column feed solution (0.21M HNO_3 - 0.32M $\text{UO}_2(\text{NO}_3)_2$ - plutonium(IV) in 30% TBP-Soltrol) and 1BX extractant solution were contacted in the reciprocating contractor. Plutonium distribution ratios were determined after 2 and 10 min contact

time. All contacts were made at a temperature of 45°C which is considered to be the average plant operating temperature. Data obtained are shown in Table II.

TABLE II
1BX COLUMN BATCH CONTACTS

HNO_3	UNH	Initial Aqueous Phase		Plutonium E_a°	
		Reducing Agent	Holding Reductant	2 min	10 min
2.0M	0.12M	None	None	1.91	1.56
2.0M	0.12M	0.02M Ferrous Sulfamate	0.03M Sulfamic Acid	0.016	0.010
2.0M	0.12M	0.002M Ferrous Sulfamate	0.03M Sulfamic Acid	0.025	0.095
2.0M	0.12M	0.02M Uranium(IV)*	0.02M N_2H_4	0.009	0.009
2.0M	0.12M	0.006M Uranium(IV)	0.02M N_2H_4	0.008	0.012
2.0M	0.12M	0.002M Uranium(IV)	0.02M N_2H_4	0.023	0.014

* Uranium(IV) prepared by reduction of UNH with aluminum powder.

That uranium(IV) was at least as effective as iron(II) (as a plutonium reductant) is apparent from the low plutonium distribution ratios obtained. When iron(II) and uranium(IV) were present at the same concentration, lower plutonium distribution ratios were obtained with uranium(IV) as reductant. In the proposed flowsheet for the Purex plant, initial uranium(IV) concentration in the 1BX column solvent is 0.0013M. In the experiments summarized in Table II, the lowest initial uranium(IV) concentration in the solvent was 0.0012 (assuming a uranium(IV) E_a° of 1.4). At this concentration the plutonium distribution ratio was comparable to that obtained with ferrous sulfamate and low enough to ensure adequate separation of plutonium from uranium. It should be noted that the tests shown in Table II were made under conditions expected in the bottom of the 1BX column where salting strength in the aqueous phase due to uranyl nitrate and nitric acid is relatively high. Higher in the column, the aqueous phase contains less nitric acid and uranyl nitrate. Plutonium (either (III) or (IV)) extraction should be even less there.

Stability of Uranium(IV) in the 1BX Column

Residence time of the organic phase in the 1BX column is estimated as about 15 min. The data given in Table II and Table III indicate that, in the absence of oxidants other than plutonium(IV) in the organic phase,

TABLE III
1BS COLUMN BATCH CONTACTS

Procedure: Equal volumes, contacted at 45 C, using reciprocating sieve plate agitation (40 strokes/min). Phases pre-equilibrated (without plutonium) and preheated before adding Pu(III). (Stock Pu(III) prepared by reduction with aluminum metal, contacted with 0.2M D2EHPA-Soltrol prior to each use to remove any Pu(IV) present).

Results:

<u>Initial Aqueous Phase Composition</u>			<u>Total Pu, Distribution Ratio</u>		
<u>HNO₃</u>	<u>N₂H₄</u>	<u>Pu(III)</u>	<u>10 min</u>	<u>30 min</u>	<u>90 min</u>
2.0M	0.02M	0.0032*	0.40	**	1.6
2.0M	0.02M	0.015	0.11	0.58	**
2.0M	0.02M	0.033	0.068	0.19	0.36
2.0M	0.02M	0.10	0.038	0.10	0.22
2.0M	0.02M	0.14	0.062	0.11	0.21
4.0M	0.02M	0.016	0.33	1.15	6.25

* Expressed as fraction of nominal Purex IBP concentration.

** Insufficient Samples

uranium(IV) is adequately stable under 1BX conditions to insure its presence throughout the 1BX column. In the current Purex process flowsheet, the 1BXF contains a small concentration of nitrous acid (Figure 1). Nitrous acid will oxidize uranium(IV) rapidly. For this reason, it is recommended that sufficient hydrazine be added to the 1BXF feed tank to assure the feed is free of nitrous acid.

Plutonium Reflux in the 1BS Column

In the present Purex plant flowsheet, plutonium is maintained in the trivalent state in the 1BS column by ferrous ion in the the aqueous phase. Concentration of ferrous ion is about 0.015M. In the proposed uranium(IV) flowsheet, the concentration of reductant (uranium(IV)) will be much lower (about 0.001M at the top and as low as 10^{-5} M at the bottom). If plutonium is re-oxidized and re-extracted under these conditions, reflux will occur. Excessive reflux could overload the reducing capacity in the 1BX column and allow plutonium to go overhead in the uranium stream. Batch contact experiments were made to determine the rate of re-oxidation of plutonium under 1BS column conditions. These are summarized in Table III. They show that even in the absence of any uranium(IV), oxidation and re-extraction of plutonium is relatively slow. Residence time of the aqueous phase

in the 1BS column is estimated at 20 min. These experiments were made at plutonium concentrations less than anticipated under process conditions. However, the data indicate that the rate at which plutonium(III) is oxidized in the two-phase system has a less than first order dependency on the plutonium concentration in the aqueous phase. Hence, even lower plutonium distribution ratios would be expected under actual operating conditions. It is estimated that with plutonium E_a° values of 0.1 or less, plutonium reflux in the 1BS column will not be significantly greater than that which occurs in the present process (ca 10%) with ferrous sulfamate as the reducing and holding agent.

Uranium Contamination in Product Plutonium

With the proposed flowsheet, the aqueous stream entering the top of the 1BS column should contain about 0.001M uranium(IV). To permit estimation of the amount of uranium which could leave the 1BS column in the product plutonium stream, distribution ratios for uranium(IV) under 1BS column conditions were determined (Table IV). Uranium(IV) distribution ratios remained essentially constant over an equilibrium aqueous phase concentration range of 0.0003 to 0.015M. Oxidation of uranium(IV) to uranium(VI) is relatively slow, less than 50% in 2 hr. Since the 1BS column conditions do not change appreciably after most of the uranium(VI) is removed in the top stages, the uranium(IV) distribution ratios in Table IV can be considered applicable to most of the 1BS column. If there are four theoretical stages in the column, the maximum uranium concentration expected in the product plutonium stream is about 5×10^{-5} M when the

TABLE IV
URANIUM(IV) DISTRIBUTION RATIOS AT 45 C

<u>HNO₃ in</u> <u>Aqueous Phase</u>	<u>Contact</u> <u>Time</u>	<u>Uranium(IV)</u> <u>Aqueous</u>	<u>Uranium (IV)</u> <u>Organic</u>	<u>Uranium(IV)</u> <u>E_a°</u>	<u>% Initial</u> <u>Uranium(IV)</u> <u>Remaining</u>
2.0 <u>M</u>	10 min	0.0162	0.0224	1.38 <u>M</u>	91.5 <u>M</u>
2.0 <u>M</u>	40 min	0.0148	0.0202	1.36 <u>M</u>	82.5 <u>M</u>
2.0 <u>M</u>	120 min	0.0097	0.0140	1.44 <u>M</u>	56.2 <u>M</u>
4.0 <u>M</u>	10 min	0.015	0.039	2.60 <u>M</u>	102.2 <u>M</u>
4.0 <u>M</u>	10 min	0.0056	0.016	2.86 <u>M</u>	102.2 <u>M</u>
4.0 <u>M</u>	10 min	0.001	0.003	3.00 <u>M</u>	94.7 <u>M</u>
4.0 <u>M</u>	10 min	0.0003	0.0006	2.00 <u>M</u>	85.7 <u>M</u>

nitric acid concentration is 4M . If addition of the 1BXP-HNO_3 stream was discontinued and consequently the 1BS aqueous stream was 2M HNO_3 , the maximum uranium concentration in the product plutonium stream would be about $1.5 \times 10^{-4}\text{M}$. These values are approximations only and neglect the more complete extraction of any uranium oxidized to uranium(VI).

Continuous Counter Current Contacts

The results of several experiments in which simulation of the extraction section of the 1B column (1BX) with a miniature pulsed column was attempted are given in Table V. In three runs, uranium(IV)-hydrazine was used as partitioning agent, and in three others, ferrous sulfamate-sulfamic acid was used. High plutonium losses (about 10%) occurred in all runs. The losses were about the same in all cases except Run 3. Emulsion formation caused unstable column performance in this run. Since the losses were independent of such variables as 1BX flow, mole ratio of

TABLE V
1BX COLUMN DEMONSTRATION RUNS IN MINIATURE PULSE COLUMN

	Run 1*	Run 2	Run 3	Run 4	Run 5	Run 6
Flows, ml/min						
1BXF	1.27	1.0	1.0	2.02	1.55	1.58
1BX	0.052	0.060	0.039	0.137	0.474	0.388
Uranium(IV)	0.076	-----	0.058	-----	-----	0.574
1BXF Composition, M						
Plutonium†	†	†	†	†	†	†
Uranium	0.336	0.347	0.32	0.311	0.328	0.32
HNO ₃	0.25	0.331	0.337	0.097	0.22	0.181
1BX Composition, M						
Fe (NH ₂ SO ₃) ₂	-----	0.03	-----	0.03	0.03	-----
N ₂ H ₄	0.02	-----	0.05	-----	-----	0.05
HNO ₃	0.30	0.50	0.30	0.50	1.0	1.0
Uranium(IV) Composition, M (in 30% TBP-Soltrol)						
Uranium(IV)	0.023	-----	0.031	-----	-----	0.029
Total Uranium	0.068	-----	0.078	-----	-----	0.076
Plutonium Material Balance, %**	28.7	93.2	170	90	99.4	103
Plutonium Loss to 1BU, % **	8.4	9.5	23.2††	10.7	9.4	9.1

* Runs 1, 2, and 3 made in 0.23 in diam column, Runs 4, 5, and 6 in 0.268 in diam column.

† Classified

** Poor material balance in Run 1 due to plutonium loss to aqueous in pulse leg, in Run 3 due to back-transfer of plutonium out of aqueous in pulse leg.

†† Column very severely emulsified, even when pulse amplitude increased, and pulse frequency lowered.

plutonium to reducing agent, and the reducing agent, it was concluded that the rate of transfer of plutonium to the organic phase was limiting. Residence time of the organic phase in the dispersed portion of the column was only 10 to 15 sec. To increase the organic residence time appreciably would have required an impractical lengthening of the column. Consequently, no further work was done with the miniature pulsed column.

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