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**SOLVENT EXTRACTION PURIFICATION OF URANIUM-233
WITH DI-SEC-BUTYL PHENYLPHOSPHONATE**

J. F. PHILLIPS, JR.

JUNE 1967

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SOLVENT EXTRACTION PURIFICATION OF URANIUM-233
WITH DI-SEC-BUTYL PHENYLPHOSPHONATE

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SOLVENT EXTRACTION PURIFICATION OF URANIUM-233
WITH DI-SEC-BUTYL PHENYLPHOSPHONATE

ABSTRACT

A solvent extraction process for separating uranium from thorium, plutonium, polonium, iron, and cobalt has been developed. The extractant is di-sec-butyl phenylphosphonate dissolved in carbon tetrachloride.

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SOLVENT EXTRACTION PURIFICATION OF URANIUM-233
WITH DI-SEC-BUTYL PHENYLPHOSPHONATE

J. F. Phillips, Jr.

INTRODUCTION

A procedure for solvent extraction purification of ^{233}U , using di-sec-butyl phenylphosphonate (DSBPP) as the extractant, was tested on a laboratory scale. To accommodate the acid feed, and because extensive experience in the use of carbon tetrachloride as a diluent in similar processes was available, a solvent flowsheet was sought which uses DSBPP diluted with carbon tetrachloride as the extractant and uranium nitrate-nitric acid solution as the feed.

^{233}U , produced by neutron irradiation of thorium, contains a small amount of ^{232}U . The decay daughters of ^{232}U (^{228}Th , ^{224}Ra , ^{220}Rn , ^{216}Po , ^{212}Pb , ^{212}Bi , ^{212}Po , and ^{208}Pb) represent a significant beta-gamma emission from product ^{233}U recovered from irradiated thorium after the product has aged long enough for these daughters to grow.⁽¹⁾ Consequently, unless these daughters are removed from aged ^{233}U , a significant beta-gamma exposure can be involved in handling the ^{233}U . All of the radioactive daughters have very short half-lives (< 4 days) except ^{228}Th ($t_{1/2} = 1.9$ years). Hence, if ^{228}Th is removed, radioactivity from the remaining daughters will soon decay to negligible levels. Other impurities in product ^{233}U of interest in a purification process are stainless steel corrosion products and traces of plutonium.

In studies at Oak Ridge National Laboratory, DSBPP, diluted with an aromatic hydrocarbon, showed promise

as an extractant for solvent extraction separation of uranium from thorium.⁽²⁻⁴⁾ Uranium extraction was 1.2 to 1.8 times higher, and thorium-uranium separation factors were ten times higher than with tri-n-butyl phosphate-aliphatic hydrocarbon diluent combinations. Subsequently, Ryan and Lowrie⁽⁵⁾ devised and tested, on an engineering scale, a DSBPP solvent extraction flowsheet which used an acid deficient, aluminum nitrate salted feed and an aromatic diluent. However, the ^{233}U product, as produced by the Hanford Project Purex Plant, was expected to be three molar in nitric acid and, hence, not suitable for an acid-deficient flowsheet without feed adjustment.

SUMMARY

A process for solvent extraction-purification of ^{233}U was developed and demonstrated on a laboratory scale. A chemical flowsheet for this process, which uses DSBPP in carbon tetrachloride as the extractant, is presented and described. The three basic steps involved are:

1. Extraction of uranium from a nitric acid aluminum-nitrate salted feed.
2. Scrubbing with aluminum nitrate-nitric acid-ferrous sulfamate solution.
3. Stripping with dilute nitric acid.

Uranium recovery was greater than 99.9% and the thorium-uranium decontamination factor was ≥ 110 .

EXPERIMENTAL PROCEDURE

SOLVENT SOURCE AND PREPARATION

The DSBPP used, was supplied by Stauffer Chemical Company. The extractant was prepared by diluting the DSBPP in its purchased form, with an appropriate volume of C. P. carbon tetrachloride (Mallinckrodt Chemical Company). Except where noted, the extractant was washed successively with equal volumes of 3% sodium carbonate, and one molar nitric acid and water.

PREPARATION OF THORIUM-234 TRACER

Thorium decontamination was followed with the aid of ^{234}Th tracer. This thorium isotope was separated from aged ^{238}U by passing a $2\text{M UO}_2\text{Cl}_2$ - 6M HCl solution through a 12 in. x 1 in. diameter bed of 100-200 mesh Dowex-X 8 cation exchange resin. The column was washed with 6M HCl and the strongly absorbed thorium was then eluted with 0.3M oxalic acid. To facilitate elution, the ion exchange column was jacketed and heated with boiling acetone.

ANALYSIS

Radioisotope concentrations were obtained by alpha or gamma counting. Uranium, where greater than 0.1 g/liter was determined by X-ray fluorescence; when less than this value, it was determined with a fluorimeter. Nitric acid was determined by sodium hydroxide titration. Data presented for mixer-settler runs are for steady-state conditions.

MIXER-SETTLER DESCRIPTION

A 12 stage mixer-settler was used to test chemical flowsheet conditions.

Operation of such contactors has been described by Schulz⁽⁶⁾ and others.⁽⁷⁾ The mixer-settler unit was operated with seven extraction and five scrub stages. All 12 stages were operated in stripping runs.

DETERMINATION OF DISTRIBUTION RATIOS

Distribution ratios were obtained by mixing equal volumes of aqueous and carbonate-washed organic for ten minutes at room temperature ($25 \pm 1^\circ\text{C}$). Phases were separated, and the aqueous phase was centrifuged and submitted for analysis. Uranium concentration in the organic phase was found by difference. Uranium distribution at 50°C was measured by shaking the samples in closed vials for ten minutes in a water bath, quickly separating phases and analyzing the aqueous phase for uranium.

RESULTS

BATCH CONTACT STUDIES

Uranium and nitric acid extraction data are presented in Tables I through XI. For ease of comparison, the data are also presented graphically in Figures 1 through 6.

TABLE I. Uranium Distribution into 5% DSBPP- CCl_4 from 0.5M Nitric Acid

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
4.66	4.12	0.54
8.20	7.27	0.93
21.16	19.23	1.93
32.36	27.98	4.38
48.29	42.56	5.73
65.44	55.51	9.93
81.90	70.35	11.55
97.78	85.17	12.61

See Figure 1 for graphical presentation.

TABLE II. Uranium Distribution into 5% DSBPP- CCl_4 from 1.0M Nitric Acid

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
4.79	3.40	1.39
8.50	6.01	2.49
12.53	9.66	2.87
21.26	16.45	4.81
33.06	25.58	7.48
48.22	38.48	9.74
65.21	52.01	13.20
82.19	67.81	14.38
98.11	82.39	15.72

See Figure 1 for graphical presentation.

TABLE VI. Uranium Distribution into 10% DSBPP- CCl_4 from 1.5M Nitric Acid

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
5.56	1.37	4.19
9.66	2.45	7.21
14.32	4.20	10.12
21.38	7.27	14.11
32.82	13.03	19.79
48.66	23.18	25.48
65.37	35.79	29.58
82.39	49.10	33.29
98.44	61.71	36.73

See Figure 2 for graphical presentation.

TABLE III. Uranium Distribution into 5% DSBPP- CCl_4 from 1.5M Nitric Acid

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
5.56	3.00	2.56
9.66	5.40	4.26
14.32	9.52	4.80
21.38	14.40	6.98
32.82	23.36	9.46
48.66	36.85	11.81
65.37	50.13	15.24
82.39	66.06	16.33
98.44	81.67	16.77

See Figure 1 for graphical presentation.

TABLE VII. Uranium Distribution into 5% DSBPP- CCl_4 from 1.0M Nitric Acid-0.5M Aluminum Nitrate

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
5.16	1.83	3.33
8.00	3.37	4.63
15.48	6.07	9.41
21.27	8.0	13.27
32.75	17.2	15.55
48.39	32.0	16.39
65.34	47.3	18.04
82.16	61.9	20.26
98.11	76.4	21.71

See Figure 3 for graphical presentation.

TABLE IV. Uranium Distribution into 10% DSBPP- CCl_4 from 0.5M Nitric Acid

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
4.66	3.28	1.38
8.20	5.24	2.96
21.16	14.28	6.88
32.36	21.12	11.24
48.29	32.55	15.74
65.44	43.19	22.25
81.90	55.63	26.27
97.78	68.45	29.33

See Figure 2 for graphical presentation.

TABLE VIII. Uranium Distribution into 5% DSBPP- CCl_4 from 1.5M Nitric Acid-0.5M Aluminum Nitrate

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
5.16	1.6	3.56
8.00	3.1	4.9
15.16	5.0	10.16
21.27	10.0	11.27
32.75	14.5	18.25
48.34	46.0	19.34
82.16	61.4	20.76
98.11	75.5	22.61

See Figure 3 for graphical presentation.

TABLE V. Uranium Distribution into 10% DSBPP- CCl_4 from 1.0M Nitric Acid

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
4.79	1.77	3.02
8.50	3.26	5.24
12.53	5.20	7.33
21.26	9.93	11.33
33.06	16.38	16.68
65.21	36.94	28.27
82.19	52.71	29.48
98.11	65.64	32.47

See Figure 2 for graphical presentation.

TABLE IX. Uranium Distribution into 10% DSBPP- CCl_4 from 1.0M Nitric Acid-0.5M Aluminum Nitrate

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
21.27	3.31	17.96
32.75	6.94	25.81
48.39	14.6	33.79
65.34	25.8	39.54
82.16	42.3	39.86
98.11	59.0	39.11

See Figure 4 for graphical presentation.

TABLE X. Uranium Distribution into 10% DSBPP-CCl₄ from 1.5M Nitric Acid-0.5M Aluminum Nitrate

Uranium Feed Concentration, g/liter	Uranium Aqueous Concentration, g/liter	Uranium Organic Concentration, g/liter
21.27	2.94	18.33
32.75	5.80	26.95
48.39	13.0	35.39
65.34	27.8	37.54
82.16	42.5	39.66
98.11	56.9	41.21

See Figure 4 for graphical presentation.

TABLE XI. Nitric Acid Distribution into DSBPP-CCl₄

Aqueous HNO ₃ Conc., M	Organic HNO ₃ Conc., M	HNO ₃ E _a
5% DSBPP-CCl ₄		
3.81	0.13	0.034
2.86	0.10	0.035
1.95	0.057	0.029
0.97	0.016	0.016
0.46	0.005	0.011
15% DSBPP-CCl ₄		
3.58	0.40	0.11
2.67	0.29	0.11
1.78	0.18	0.10
0.93	0.054	0.058
0.47	0.014	0.030

See Figure 5 for graphical presentation.

TABLE XII. Uranium Stripping Data at 25 °C and 50 °C with 0.01M Nitric Acid

Initial Aqueous Uranium Concentration, g/liter	Final Aqueous Uranium Concentration, g/liter	Calculated Organic Uranium Concentration, g/liter
5% DSBPP - 25 °C		
23.32	22.60	0.72
58.31	54.30	4.01
116.7	104.7	12.0
363.1	335.6	27.5
15% DSBPP - 25 °C		
23.32	20.00	3.32
58.31	42.80	15.51
116.7	80.9	35.8
232.1	175.0	57.1
349.9	295.0	54.9
459.3	405.0	54.3
15% DSBPP - 50 °C		
17.8	16.0	1.8
27.2	22.0	4.3
43.8	34.0	9.8
58.2	46.2	12.0
64.9	47.0	17.9
73.3	49.0	24.3

See Figure 6 for graphical presentation.

FIGURE 1. *Uranium Distribution Between Nitric Acid and 5% DSBPP*

FIGURE 2. *Uranium Distribution Between Nitric Acid and 10% DSBPP*

FIGURE 3. Uranium Distribution Between 5% DSBPP and Nitric Acid--
0.5M Aluminum Nitrate

FIGURE 4. Uranium Distribution Between 10% DSBPP and Nitric Acid--
0.5M Aluminum Nitrate

FIGURE 5. Nitric Acid Extraction with 5% and 15% DSBPP

FIGURE 6. Extraction of Uranium from 0.01M Nitric Acid with DSBPP-CCl₄

Salient features of these data are as follows:

- While not as effective as aluminum nitrate, nitric acid appears to adequately salt uranyl nitrate into DSBPP.
- Nitric acid extraction by DSBPP is relatively low.
- Stripping uranium from 15% DSBPP is difficult with 0.010M HNO_3 .
- Increased temperature does not increase the ease of stripping.

With these distribution data, a tentative flowsheet was constructed by G. L. Richardson of Battelle-Northwest Laboratory in which the feed and scrub streams were salted only with nitric acid. However, on the basis of miniature mixer-settler runs, described below, some modifications were necessary to reduce uranium waste losses.

MINIATURE MIXER-SETTLER RUNS

Extraction and strip column operations were initially tested with unwashed DSBPP. Impurities in the extractant, thought to be similar to di-(2-ethylhexyl)phosphoric acid, caused high uranium losses to the strip-column waste. Therefore, miniature mixer-settler runs to measure uranium losses were made with carbonate washed DSBPP. However, decontamination factors were measured with unwashed DSBPP.

Extraction Column Operation

Shown in Table XIII are results of extraction column miniature mixer-settler runs.

Extraction Column Decontamination Factors

Extraction column decontamination factors for potential contaminants were measured, using the feed stream

composition of Run 32. Thorium decontamination in Run 39 was measured with carbonate washed DSBPP; all other decontamination factors were measured with unwashed DSBPP, as shown in Table XIV.

TABLE XIII. Extraction Column Operation

Extractant (1AX): Carbonate washed 10% DSBPP diluted with carbon tetrachloride
 Scrub (1AS) : 2.0M HNO_3 , 0.05M $\text{Fe}(\text{NH}_2\text{SO}_3)_2$, 0.25M $\text{Al}(\text{NO}_3)_3$
 Flows : Extraction/Feed/Scrub = 1.0/0.09/0.20

Run Number	Feed Composition	Uranium Loss
24B	1.26M $\text{UO}_2(\text{NO}_3)_2$, 3.0M HNO_3	0.37%
31	1.26M $\text{UO}_2(\text{NO}_3)_2$, 3.0M HNO_3 , 0.10M $\text{Al}(\text{NO}_3)_3$	0.021%
32	1.26M $\text{UO}_2(\text{NO}_3)_2$, 3.0M HNO_3 , 0.25M $\text{Al}(\text{NO}_3)_3$	0.001%

TABLE XIV. Extraction Column Decontamination Factors

Run Number	Element	Decontamination Factor
11	Thorium	≥ 110
12	Plutonium	1210
13	Polonium	236
14	Cobalt	≥ 147
15	Iron	≥ 1175
39	Thorium	≥ 125

Strip Column Operation

Additional mixer-settler runs, described below, were designed to test the strip column operation. Using the product from Run 32, the strip column was run with the following results:

Run Number: 33
 Feed (1AP): 10% DSBPP Diluted with Carbon Tetrachloride, 27 g/liter uranium
 Extractant (1CX): 0.01M HNO_3
 Flows: (1AP)/(1CX) = 1.0
 Uranium Waste Loss: 0.08%

Extraction - Strip Column Operation with Used DSBPP

One final run was made using the flow ratios and feed, together with the scrub and strip solution compositions of Runs 32 and 33. The DSBPP previously had been used for one extraction-strip cycle, allowed to stand for ten days, and then carbonate-washed. Results were as follows:

Uranium loss to the 1AW stream:

0.004%

Uranium loss to the 1CW stream:

0.08%

DISCUSSION AND CONCLUSION

Based on the mixer-settler runs, the chemical flowsheet shown in Figure 7 is proposed for dedaughterizing ^{233}U . Uranium loss data of Table XIII shows

that $\text{Al}(\text{NO}_3)_3$ in the feed is necessary to prevent high uranium losses. While the flowsheet is adequate with a 0.10M $\text{Al}(\text{NO}_3)_3$ feed, extraction column uranium losses are further reduced by approximately 20 if 0.25M $\text{Al}(\text{NO}_3)_3$ feed is used.

Decontamination factors for all elements tested exceeded 100. Although these decontamination factors were measured with unwashed DSBPP, we feel they are valid for carbonate-washed DSBPP as well. In fact, the suspected impurity in unwashed DSBPP is apparently a better extractant than DSBPP. Hence, the decontamination factors which would be obtained with carbonate-washed DSBPP may be actually higher than those quoted in Table XIV.

FIGURE 7. Proposed Flowsheet for Dedughterizing ^{233}U

Uranium losses in the strip column were substantially higher than extraction column waste losses. Nevertheless, if the 1AP/1CX flow ratio is 1.0 or greater, uranium losses will be less than 0.1%.

One final run made with aged DSBPP showed that the extractant may be re-used if it is carbonate-washed prior to solvent extraction.

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