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**REFINING OF URANIUM FROM LEACH PLANT ION
 EXCHANGE NITRATE ELUATES BY EXTRACTION
 WITH DIBUTYL BUTYLPHOSPHONATE**

by

V. M. McNAMARA, H. W. PARSONS & R. SIMARD

EXTRACTION METALLURGY DIVISION

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REFINING OF URANIUM FROM LEACH PLANT ION EXCHANGE
NITRATE ELUATES BY EXTRACTION WITH
DIBUTYL BUTYLPHOSPHONATE

by

V.M. McNamara*, H.W. Parsons*, and R. Simard**

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SUMMARY OF RESULTS

Continuous liquid-liquid extraction tests were done on current samples of ion exchange nitrate eluates with 25% v/v dibutyl butylphosphonate in kerosene. From eluates containing 15 to 28 g U₃O₈/l, recoveries of 99% or better were obtained in four countercurrent stages. The effects of nitrate and sulphate concentrations on extraction equilibria were determined. The extract, analyzing 75 to 95 g U₃O₈/l, was stripped effectively in four stages with 5 to 10% ammonium sulphate solutions at pH's ranging from 3.5 in the first stage to 6.0 in the last. The uranium was precipitated as ammonium diuranate by gaseous ammonia, and the barren strip was recycled to the circuit. The effect of temperature and dilution of the ammonia with nitrogen on the filtering rate of the ammonium diuranate (ADU) precipitate and the subsequent reduction and sintering properties were investigated. The product was of nuclear grade purity and was used to produce high density (>10.5) uranium dioxide pellets.

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INTRODUCTION

In the established flowsheet of most uranium acid leach plants, uranium is recovered from leach solutions by anion exchange resins which normally are eluted with acidified nitrate solutions. The eluate is then neutralized in two stages, the first to pH 3.5 with lime, and the second to pH 7.0 with ammonia, magnesia or caustic soda. The first-stage precipitate or iron cake is recycled to leaching while the final uranium yellow cake is shipped to a custom refinery for re-extraction with tributyl phosphate (TBP) and production of nuclear grade uranium trioxide or ammonium diuranate.

Previous studies⁽¹⁾⁽²⁾ have been made at the Mines Branch on the possibilities of refining the uranium by direct extraction with TBP from nitrate eluates, or by the sulphuric acid elution-amine extraction process. A third possibility was suggested by the availability of data⁽³⁾ on the properties of dialkyl alkylphosphonates as efficient extractants of uranyl nitrate from nitric acid solutions. It was considered likely that phosphonates would be more suitable than TBP, because of the higher extraction coefficients obtained at the lower nitric acid concentration used for ion exchange elution (0.3-0.4 N HNO₃).

Two kerosene-soluble phosphonates are available commercially in Canada;* they are dibutyl butylphosphonate (DBBP) and bis-2 ethylhexyl 2-ethylhexylphosphonate. These were first investigated in batch-scale

* Electric Reduction Co. of Canada, Toronto, Ontario.

tests in the laboratory, and, on the basis of the better performance of the former, it was then decided to carry out continuous bench-scale tests with nitrate eluates obtained from operating plants.

Batch equilibrium tests are continuing and will be reported separately. These tests had already defined certain operating conditions, mainly the uranium capacity of 25% DBBP and the necessity of using ammonium sulphate for effective stripping, but the continuous tests were necessary 1) to confirm extraction and stripping equilibria, 2) to determine the selectivity of the extractant as evidenced by the purity of the final product, 3) to determine the solubility and entrainment losses of phosphonate, and 4) to demonstrate the adaptability of such a process to current leach plant circuits.

For this work, drum quantities of eluate were obtained from Stanleigh Uranium Mines Limited and Consolidated Denison Mines Limited, of Elliot Lake, Ontario. A lesser quantity of nitrate eluate was obtained from operation of the ion exchange pilot plant (4 in. diameter columns) assembled at Ottawa by the Extraction Metallurgy Division of the Mines Branch. The feed to the ion exchange pilot plant was a solution produced by acid leaching of ore obtained from Milliken Lake Uranium Mines Ltd., Elliot Lake, Ontario.

PROCEDURE

Extraction and Stripping

The existing four-stage mixer-settler units used in earlier tests were used for this work. The reasons for the choice of this equipment over pulse columns are discussed in a previous report.⁽¹⁾

The flowsheet shown in Figure 1 was adopted for all tests. Each lot of eluate was first neutralized with lime to pH 3.0 to 3.5, filtered and reacidified with nitric acid to 0.4 N HNO₃. Twenty litres of 25% v/v DBBP solvent in Shell* kerosene (flash point, 140° F) were prepared and equilibrated with 0.7 N HNO₃ before being added to the circuit. The stripping solution was made up from dilute sulphuric acid neutralized to pH 7.0 with gaseous ammonia. For pH control in stripping, a 2.0% ammonia solution was also prepared.

For each run the flowrates were held constant for the time required for a complete solvent cycle (6 hr). The aqueous phase from each settler and the extract and the recycle solvent were sampled before shutdown. A composite of the barren eluate for the whole series of runs was also collected and the phosphonate determined by an alkalimetric molybdate procedure after conversion to orthophosphate ion by a wet combustion procedure.

The pH in stripping was maintained by an automatic pH controller, feeding the weak ammonia solution to the first-stage mixer. In the

* Shell Oil Co., of Canada Ltd.

VOLUMES (ml)

	Extractor	Stripper
Mixer	800	400
Settler	2400	1200

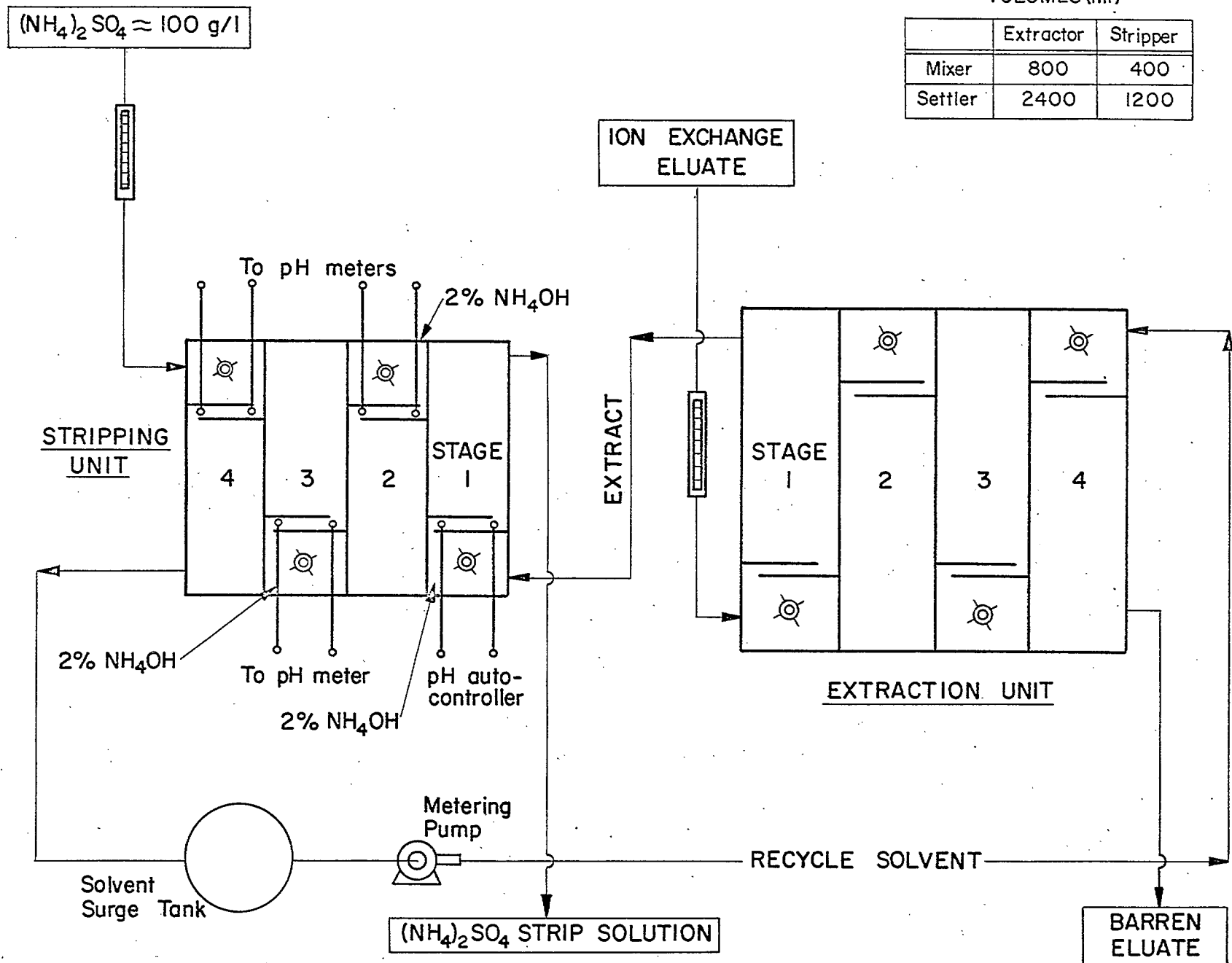


FIGURE 1

FLWSHEET OF EXTRACTION AND SULPHATE STRIP CIRCUITS

subsequent mixer-settler stages the pH was controlled manually. The strip solution collected from each run was sent to precipitation and the barren solution was then recycled to stripping in the subsequent test, after being adjusted to pH 7.0.

Precipitation

The apparatus used for continuous precipitation was described in a previous report.⁽²⁾ Briefly, it consists of a 2000 ml beaker, provided with a heating mantle, pH electrodes and mechanical stirrer, to which are fed the strip solution and ammonia gas through separate flowmeters. The slurry overflows to a 1500 ml surge tank and thence to the filter. Where ammonia was diluted with nitrogen, the gases were metered separately before joining in a common tube leading to the bottom of the precipitation beaker.

Optimum conditions, as previously determined for continuous precipitation of ammonium diuranate (ADU) from amine sulphate strip solutions,⁽²⁾ were used in the present tests, namely:

pH:	7-7.5
Temperature:	30-60°C
Retention time:	15-20 minutes
Washing:	By reslurrying

In this investigation, batch precipitation was compared with the continuous method. Also, the effects of temperature and gas dilution on the filtration, subsequent reduction and sintering of the ADU precipitate were studied.

The final ADU precipitates were submitted to the Research and Development Division of Eldorado Mining and Refining Ltd., Ottawa, for reduction and sintering tests.

RESULTS AND OBSERVATIONS

Extraction and Stripping

A series of nine runs was completed with eluates from the three sources. The results are presented in Table 1, and equilibrium data for three typical runs are shown graphically in Figures 2, 3 and 4.

Extraction Equilibria:

As the uranyl nitrate capacity of 25% DBBP was already known to be of the order of 90 g U_3O_8/l , the flowrates were adjusted accordingly. The effects of sulphate and nitrate concentrations in the eluate on uranium distribution (Co/Ca) were not clearly demonstrated but, within the range of concentrations covering possible variations in plant practice, it was established that barren eluates containing less than 0.1 g U_3O_8/l were readily obtained in four countercurrent stages from a feed acidified to 0.4 N HNO_3 .

Stripping Equilibria:

It was found that by adjusting the pH of the incoming solution to 6.0 and the pH of the first stage to 3.5, the pH in the 2nd, 3rd and 4th stages was automatically maintained in a range just below the point of uranium precipitation, where the stripping efficiency is highest. Under this condition, a minimum of three stages were required at high sulphate and low nitrate concentrations (Figure 3). Where the barren strip solution was recycled, a build-up of nitrate to 0.35 N NO_3 occurred which increased the required number of stages to four when 8% $(NH_4)_2SO_4$ solution was used and to five with 4% $(NH_4)_2SO_4$ (Figure 4). In all

cases there is a well defined operating range which limits the uranium concentration to 45 g U_3O_8/l in the strip solution and to 0.2 g U_3O_8/l in the recycle solvent. This last value does not affect extraction efficiency, since it is still much below the equilibrium point of the fourth stage of extraction.

Phase Separation and Stage Efficiency:

Physically, the operation of the circuit was very satisfactory at the flowrates used. Although no quantitative data were obtained on settling rates, the use of 3 sq ft of settling area/gal/min of combined aqueous and organic phases was more than was necessary when operating with a continuous organic phase. Eluate retention time in each stage was approximately one minute, which gave almost 100% stage efficiency on the basis of the batch equilibrium data obtained previously with the same solutions.

Solvent Losses:

A composite barren eluate sample of the nine runs was analysed for solvent content and showed 0.10 g DBBP equivalent per litre (100 ppm). This represents a loss of 0.006 lb DBBP/lb U_3O_8 for an eluate containing 15 g U_3O_8/l . Soluble loss of DBBP has been reported previously⁽⁴⁾ for a 2.5% solution in kerosene when in contact with acidic solutions; a value of 25 ppm was given. In the present investigation, where a 25% solution was used, the loss of 100 ppm does not seem unreasonably high.

TABLE I

Continuous Countercurrent Extraction and Stripping Tests.
(25% DBBP)

RUN NO:	469	470	471	472	473	474	475	476	477
<u>Ion Exchange Eluate</u>		<u>Stanleigh</u>			<u>Milliken Lake</u>		<u>Consolidated Denison</u>		
U ₃ O ₈ , g/l		18.19			15.33		14.51		
HNO ₃ Normality		0.40			0.40		0.40		
Tot. NO ₃ "		1.70			0.60		1.20		
SO ₄ , "		0.64			0.125		0.405		
<u>Stripping Solution</u>	<u>FRESH</u>	<u>RECYCLE</u>	<u>RECYCLE</u>	<u>RECYCLE</u>	<u>FRESH</u>	<u>FRESH</u>	<u>RECYCLE</u>	<u>RECYCLE</u>	<u>RECYCLE</u>
(NH ₄) ₂ SO ₄ (g/l)	193	183	152	164	193	97	83	79	40
NO ₃ (g/l)	nil	10.6	23.6	26.2	nil	nil	12.5	22.3	20.0
pH	7.0	5.2	6.0	6.1	6.1	6.0	6.1	6.2	6.2
<u>Duration of Run, hours</u>	6.3	6.2	6.1	6.0	6.0	6.0	5.6	5.8	6.2
<u>Flowrates, ml/min</u>									
Ion Exchange Eluate	241	270	278	273	330	283	298	296	296
Recycle Solvent	62	59	54	50	54	55	50	49	49
Stripping Solution	122	117	121	120	103	109	99	98	109
<u>U₃O₈ Analyses, g/l</u>									
Eluate: Stage 1	3.17	4.93	11.13	12.29	13.27	3.85	8.40	11.49	10.95
2	0.25	0.42	2.87	2.43	8.78	0.49	1.72	4.19	4.34
3	0.015	0.026	0.29	0.14	4.09	0.041	0.14	0.58	0.96
4	0.002	0.003	0.008	0.005	0.30	0.008	0.012	0.046	0.10
Strip: Stage 1	37.24	42.86	43.90	37.33	45.72	36.00	41.27	43.48	41.94
2	16.19	6.46	27.29	25.00	34.36	4.24	7.83	10.78	25.24
3	0.46	0.24	1.08	0.16	0.11	0.14	0.31	0.74	6.78
4	0.043	0.058	0.029	0.054	0.042	0.051	0.060	0.096	1.69
Composite Barren Eluate	0.005	0.002	0.006	0.006	0.21	0.017	0.010	0.027	0.064
Extract	74.76	83.85	92.61	94.31	92.01	64.76	80.42	86.73	80.81
Composite Strip	38.95	43.81	42.26	41.28	48.72	33.33	38.96	41.11	39.69
Recycle Solvent	0.14	0.20	0.19	0.25	0.23	0.20	0.20	0.24	0.55
<u>Control pH in Stripping</u>									
Stage 1	3.0	3.5	3.5	3.5	3.5	3.4	3.6	3.5	3.6
2	4.0	4.3	4.2	4.2	4.2	4.1	4.1	3.9	4.0
3	4.4	5.1	4.9	5.1	5.5	4.5	4.5	4.5	4.3
4	7.2	7.2	5.3	5.9	6.4	5.0	5.1	5.5	5.3
<u>Volumes, litres</u>									
Barren Eluate	87	96	98	100	125	98	95	100	105
Strip Solution	41	40.5	41	43	35.5	35.5	31.5	30	36
<u>Composite Barren Eluate</u>									
ThO ₂ , g/l		0.10			0.10		0.07		
(RE) ₂ O ₃ , "		<0.01			<0.01		0.01		
Fe, "		0.06			0.05		0.05		
<u>Ammonia Consumption</u>									
lb NH ₃ /lb U ₃ O ₈	0.008	0.011	0.007	0.018	0.005	0.009	0.013	0.008	0.021

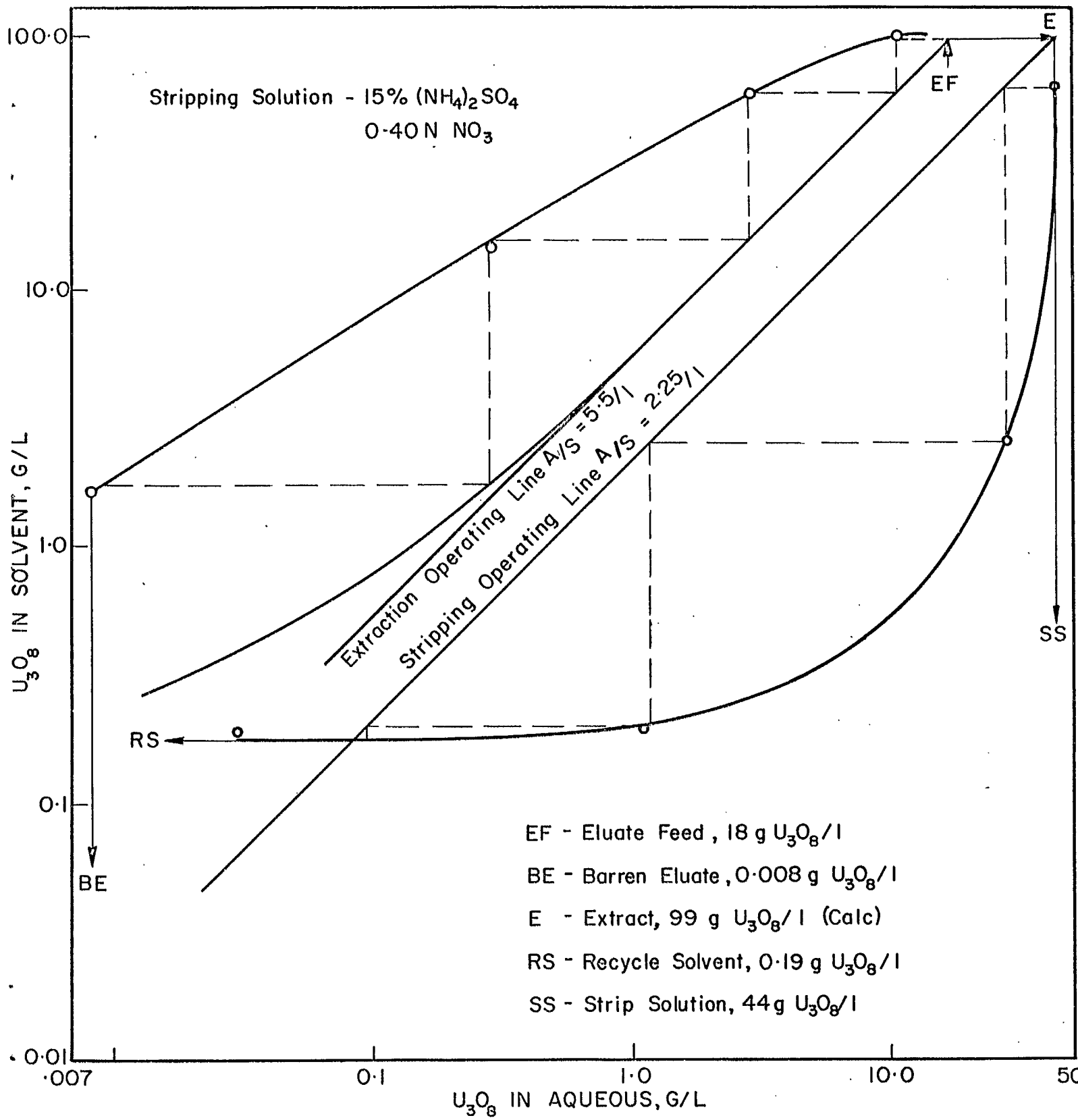


FIGURE 2

OPERATING EQUILIBRIA

25% Dibutyl Butylphosphonate vs Stanleigh Ion Exchange Eluate(Run 471)

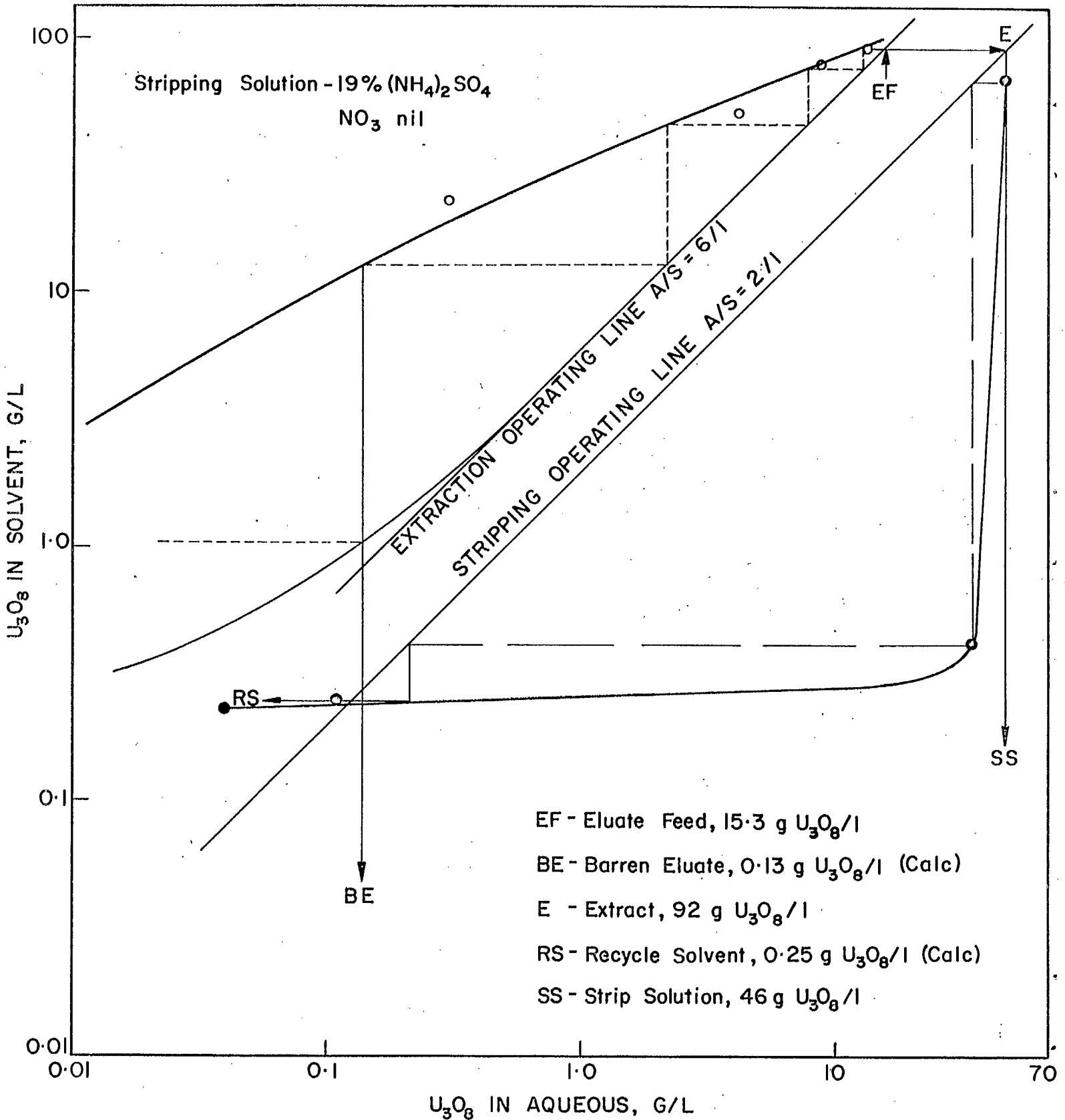


FIGURE 3

OPERATING EQUILIBRIA

25 % Dibutyl Butylphosphonate vs Milliken Lake Eluate (Run 473)

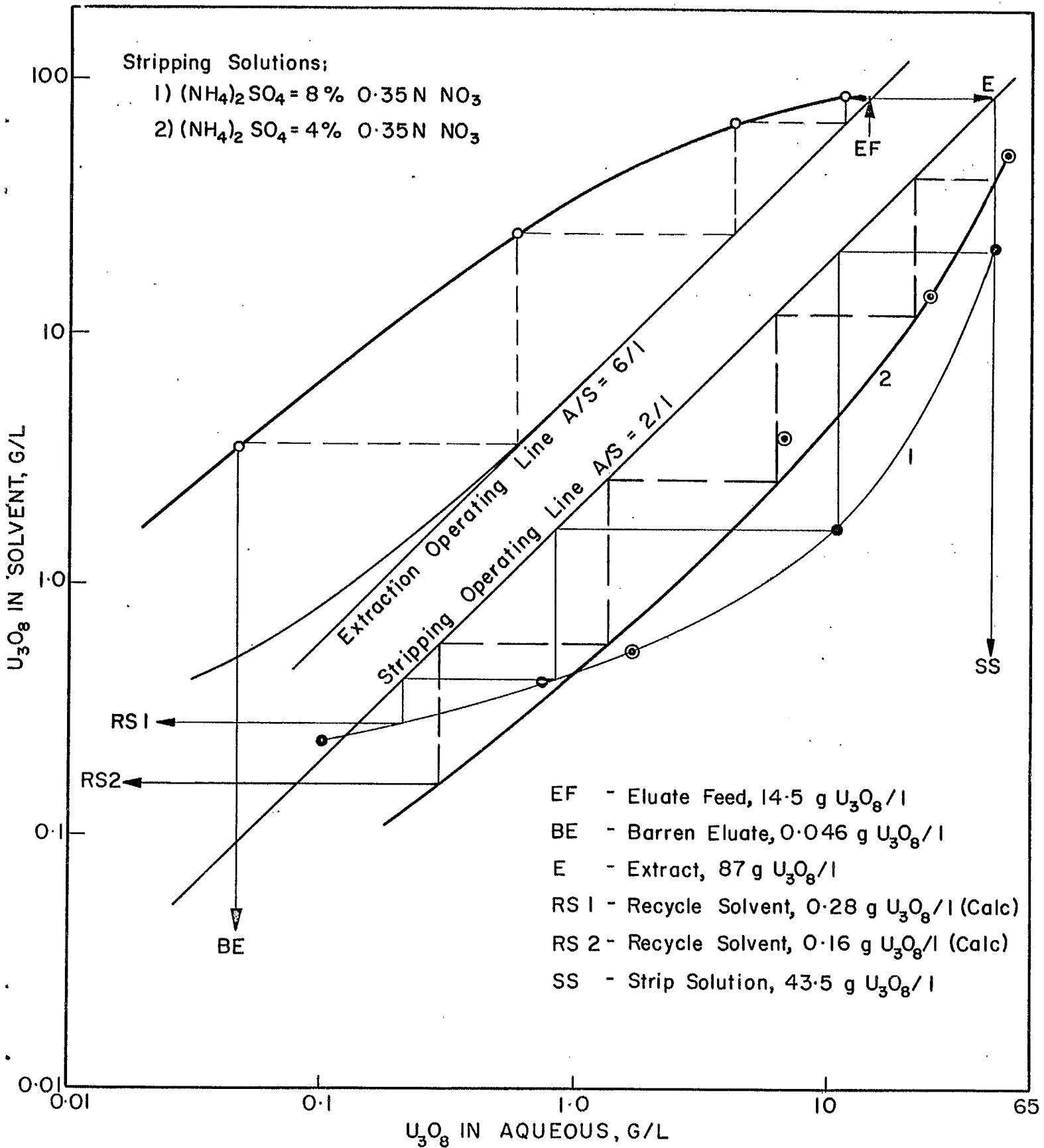


FIGURE 4

OPERATING EQUILIBRIA

25% Dibutyl Butylphosphonate vs Consolidated Denison Eluate(Run 476-7)

Precipitation

A series of batch tests was done on Stanleigh strip solution, for comparison with the continuous procedure. The effects of temperature, pH and rate of precipitation on the sulphate content of the ADU were studied. The results are presented in Table 2.

TABLE 2

Batch Precipitation of ADU from Stanleigh Strip Solution
(Strip Solution: Initial pH, 3.3; U₃O₈, 37.14 g/l)

Test No.	Precipitation Conditions			ADU Precipitate Analysis		
	Temp., °C.	Final pH	Time of ammonia add.(min)	U ₃ O ₈ , %	NH ₃ , %	SO ₄ , %
1	24	8.5	60	72.35	4.19	16.8
2	24	7.0	35	73.72	4.08	17.19
3	40	7.0	190	74.07	4.34	15.87
4	40	7.0	35	72.30	4.84	16.41
5	40	8.5	65	66.12	4.45	16.41
6	70	7.0	35	75.0	4.48	14.07
7	70	7.7	220	76.87	3.75	14.13

The UO₂ pellet produced from Test 6 ADU had a sintered density of 10.52.

In continuous precipitation tests, the effects were examined of temperature and of the dilution of ammonia with nitrogen on the sulphate content of the ADU and on the density of the UO_2 sintered pellets.

The results are shown in Table 3.

The precipitate obtained from batch precipitation had a much higher sulphate content, which is chemically bound, probably as an ammonium uranyl sulphate double salt. Variations in the rate of ammonia additions did not affect the sulphate content, and precipitation at higher temperatures reduced it only slightly. Batch-precipitated ADU filtered much more rapidly than did the ADU precipitated continuously with 100% ammonia gas.

In continuous tests in which 100% ammonia gas was used, the chemical composition of the ADU was not affected by temperature. By diluting the ammonia with nitrogen, a coarser ADU was obtained which filtered well without the aid of flocculants. From tests 13 to 19 it will be seen that reproducible results can be obtained from continuous precipitation under the following conditions:

Retention time: 10-15 minutes
pH: 7.0-7.5
Temperature: 60° C
Ammonia dilution: 33% NH_3 by vol. in air or nitrogen

Immediate filtration
Washing by reslurrying.

TABLE 3

Continuous Precipitation of ADU with Gaseous Ammonia

Test No.	Source of Eluate	Strip Solution				Precipitation Conditions			ADU Precipitate Analysis						UO ₂ Pellets	
		Vol., litres	pH	U ₃ O ₈ , g/l	SO ₄ , g/l	NO ₃ , g/l	Temp., °C	Gaseous Ammonia, % by vol.	pH* of Barren Strip	U ₃ O ₈ , %	NH ₃ , %	SO ₄ , %	ThO ₂ , ppm	RE ₂ O ₃ , ppm	B, ppm	Sintered Density
1	Stanleigh	15		42.67			40	100	7.8	86.0	2.88	3.90				10.52
2	"	14		"			60	"	8.8	85.7	2.99	5.40	28	26	2.0	10.13
3	"	12		"			75	"	8.5	83.6	3.40	4.23				
4	"	30	3.0	35.71	115.5	18.1	70	"	8.6	80.4	2.98	5.80	56	13	0.3	
5	"	8		42.65			"	33	7.8	86.0	2.80	2.46				
6	"	17		"			"	"	7.9	84.3	2.42	3.15				
7	"	10		"			"	"	7.8	87.7	2.62	1.47				
8	Milliken Lake	8	3.70	48.4	134.1	24.0	60	66	7.85	85.9	2.84	3.15				10.53
9	"	7	"	"	"	"	"	50	- -	85.4	2.46	5.40	49	<50		10.50
10	"	5	"	"	"	"	"	33	7.80	79.7	3.71	8.58	56	<50	4.0	10.53
11	"	9	"	"	"	"	"	25	7.75	87.7	2.88	3.45	53	<50		
12**	"	2	"	"	"	"	"	50	7.55	79.1	3.98	13.08**	36	17	<0.1	
13***	Cons. Denison	16	3.9	38.56	- -		60	33	7.85	85.1	2.86	1.83	46	< 5	<0.1	10.61
14	"	16	"	"	- -		"	"	7.85	87.1	2.36	1.08				10.58
15	"	30	3.6	41.94	- -		"	"	7.90	86.7	2.80	1.68				10.61
16	"	10	"	40.38	45.21	39.4	"	"	7.75	86.3	2.70	1.08				10.76
17	"	10	"	"	"		"	"	7.70	90.0	2.27	1.29				10.74
18	"	10	"	"	"		"	"	7.70	85.7	2.98	- -				10.62
19	"	10.5	3.40	36.79	- -		30	"	7.45	86.0	2.74	1.80				10.62

* Controlled pH during precipitation, 7.0-7.5. ** Batch precipitation. *** Tests 13 to 18: reproducibility tests with immediate filtration and washing by reslurrying.

Purity of Product

In Table 4, the chemical and spectrographic analyses of the precipitates obtained by this process are compared with those of a refined ADU produced by Eldorado Mining and Refining Ltd.,⁽⁵⁾ Port Hope, Ontario. The values are comparable, but their significance in terms of nuclear grade purity can only be ascertained by actual neutron absorption tests as performed at Chalk River.

TABLE 4

Chemical and Spectrographic Analyses
of Composite Ammonium Diuranate Precipitates

Element	Source of Treated Eluate			Port Hope Refinery Specification of ADU
	Stanleigh	Milliken Lake	Cons. Denison	
<u>Chemical</u>				
U ₃ O ₈ , %	85.7	85.4	85.0	86
NH ₃ , %	3.0	2.5	2.66	-
SO ₄ , %	5.0	5.4	1.83	-
ThO ₂ , ppm	56	16	46	50
RE ₂ O ₃ ppm	13	17	< 5	-
B, ppm	0.3	< 0.1	< 0.1	0.2
<u>Spectrographic (ppm, U basis)</u>				
Ag	-	-	< 0.1	1.0
Cd	< 0.5	< 0.5	< 0.1	0.2
Co	20	20	10	-
Cr	< 5	< 5	10	10
Cu	8	8	7	10
Fe	< 20	< 20	17	35
Mn	< 10	< 10	< 1	5
Mo	< 10	< 10	6	1
Ni	< 15	< 15	2	15
P	< 40	< 40	10	-
Si	40	40	15	20
V	< 12	< 12	< 10	30
Dy	< 0.06	< 0.06	0.2	0.1
Gd	< 0.06	< 0.06	0.21	0.05

CONCLUSIONS

A refined uranium product has been obtained directly from current ion exchange nitrate eluates by extraction with dibutyl butylphosphonate in a continuous countercurrent mixer-settler unit. The ADU product obtained from the strip solution by continuous ammonia precipitation was of high purity and was used to produce uranium dioxide sintered pellets of densities greater than 10.5.

The process can be adapted to uranium leach plant flowsheets in which nitrate elution is used. Since the nitric acid concentration used in the liquid-liquid extraction is the same as is normally adopted for barren eluates recycled to ion exchange, nitric acid consumption will only be increased by the amount extracted by the DBBP solvent. In addition, the ammonia used for neutralizing this acid in stripping will represent an increase over that consumed in the precipitation of ADU. Solvent losses will also represent a small increase in reagent cost. All these items are shown in Table 5, which represents reagent costs over and above those of producing unrefined uranium "yellow cake" in the leach plant. The relatively high concentration of uranium in the solvent indicates that the circuit and the inventory of solvent will not be large.

TABLE 5

Costs of Reagents for Refining of Uranium from Ion Exchange
Nitrate Eluates by the DBBP Process

Reagent Used	Reagent		Cost \$/lb U ₃ O ₈
	Price, \$/lb	Consumption, lb/lb U ₃ O ₈	
HNO ₃ (100%)	0.07	0.037*	0.0026
NH ₃	0.045	0.01**	0.0004
(NH ₄) ₂ SO ₄	0.02	1.25***	0.0250
DBBP	1.70	0.006	<u>0.0102</u>
TOTAL			\$0.0382

* Calculated from the equivalent ammonia consumed.

** Averaged from Table 1.

*** Based on the use of 50 g (NH₄)₂ SO₄/l, in a strip solution containing 40 g U₃O₈/l. The barren strip was not recycled.

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