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RECOVERY OF URANIUM FROM REXSPAR (BIRCH ISLAND, B.C.) LEACH SOLUTION BY SOLVENT EXTRACTION WITH ALAMINE.

by

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RADIOACTIVITY DIVISION

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SUMMARY OF RESULTS

Rexspar autooxidation pressure leach solution, assaying 0.48 g U_3O_8/l, was successfully treated by solvent extraction, using 0.1N Alamine in kerosene, to recover high-grade uranium product. Uranium recovery was 99.99% in a precipitate containing 86.2% U_3O_8. The fluoride content of the precipitate was slightly over the specified limit but all other impurity specifications were met. Reagent consumption was 1.34 lb NaOH/lb U_3O_8 recovered. Total extractant loss amounted to 0.02 lb Alamine and 0.30 lb kerosene/lb U_3O_8 recovered.

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INTRODUCTION

In the early part of 1958, the Mines Branch carried out an extensive investigation on ore from the Birch Island, British Columbia property of Rexspar Uranium and Metals Mining Co.,Ltd. The purpose of the work was to study the amenability of the ore to autooxidation pressure leaching,and of the resultant uranium-bearing solutions to the amine solvent extraction process, using tri-iso-octylamine, for uranium recovery [1] [2].

Subsequent to this work, the supplier of tri-iso-octylamine, Carbide Chemicals Ltd., announced that the manufacturing of this reagent had been discontinued. Consequently, A.H. Ross and Associates, consultants for Rexspar, requested that the organic extractant Alamine be tested as an alternative to the tri-iso-octylamine. Alamine is a product of General Mills Inc., Chemical Division, and is a tri-fatty amine.

A further quantity of Rexspar ore was pressure-leached (Appendix 1) and the resulting solution was sufficient for six test runs in the solvent extraction circuit. The ore sample used for leaching was the same ore used in the earlier work (Sample No. 11/57-7)[1].

[1] and [2]- See References on page 7

DESCRIPTION OF TEST WORK

The description of the circuit and the general operating procedure have been dealt with in detail in our previous Investigation Report (2). The only variation from previous procedure was the use of 15% w/v sodium sulphate strip solution instead of 20% w/v strip solution.

A series of six continuous runs was completed in the laboratory solvent extraction unit in this investigation. A total of 1260 litres of leach solution containing, on the average, 0.48 g U_3O_8/l was treated. The extractant was 5% v/v Alamine in the kerosene (BA jet fuel) with 2% n-decyl alcohol added to prevent third-phase formation. A four-stage countercurrent mixer-settler was used as in the previous tests on tri-iso-octylamine.

The loaded solvent, assaying 5 to 6 g U_3O_8/l, was stripped in three counter-current stages with 15% Na_2SO_4 solution which was maintained at pH 4.5 to 5.0 by the addition of dilute NaOH solution.

The high-grade strip was precipitated at pH 7.0 to 7.2 by means of a further addition of caustic, to produce the final uranium product.

RESULTS AND DISCUSSION

The run data are summarized in Table 1. Saturation loadings of better than 6 g U_3O_8/l on the solvent were possible. The six runs are considered to be representative of operating conditions, with loadings of 5 to 6 g U_3O_8/l on the solvent. The runs were of sufficient duration to enable one complete cycle of the organic solvent (0.1 N amine).

Titrations of amine normality in the solvent, at the end of each run, show that there was no loss of Alamine resulting from solubility in the barren raffinate. Other studies have confirmed this observation.

It may be noted that, under the conditions of the six runs, the concentration of Na_2SO_4 in the recycled barren strip solution remained essentially unchanged.

Table 1 gives the values for NaOH consumption for each run. The overall average reagent consumption is shown in Table 2.

TABLE 1

Operating Data

Run Number	419	420	421	423	424	425
Pregnant Solution Assays						
U_3O_8 (g/l)	0.50	0.50	0.50	0.45	0.40	0.52
SO_4 (g/l)	11.6	11.6	11.6	15.8	16.2	19.9
pH	1.6	1.6	1.6	1.6	1.6	1.6
Amine Normality	0.1066	0.1066	0.1066	0.1066	0.1066	0.1066
Na_2SO_4 in Strip Solution (g/l)	145	145	145	145	145	143
Operating Time (hours)	7	7	4 3/4	6 1/2	7 1/4	8
Flow Rates (ml/min)						
Pregnant Solution	456	497	502	524	596	528
Solvent	50	48	49	45	45	48
Na_2SO_4 Strip Solution	15	14	18	14	15	15
Scrub (5% H_2SO_4)	12	14	14	15	13	14
Raffinate Assays (U_3O_8, g/l)						
Stage 1	-	-	0.089	0.075	0.053	0.100
Stage 2	-	-	0.002	0.001	0.002	0.003
Stage 3	-	-	0.001	0.0003	0.0005	0.0002
Comp. Barren Raffinate (U_3O_8, g/l)	0.0004	0.0006	0.0004	0.0002	0.0004	0.0002
U_3O_8 Recovery (%)	99.99	99.99	99.99	99.99	99.99	99.99
U_3O_8 (g/l)						
Solvent Extract	5.29	6.14	5.72	5.63	4.98	5.66
Solvent Recycle	-	-	0.017	0.016	0.014	0.037
Na_2SO_4 Strip	-	-	12.88	16.32	14.10	13.11
Total Uranium (g)			231.8	86.5	91.6	91.8
Precipitation						
Strip Volume (l)			18.0	5.3	6.5	7.0
Precipitate Wt (wet/dry, g)			656/304	--/89	335/112	304/115
NaOH Consumption (lb/lb U_3O_8)						
Stripping			1.069	0.971	1.103	1.090
Neutralization			0.302	0.220	0.284	0.316
Total			1.371	1.191	1.387	1.406

Note: High-grade strip solution from runs 419, 420 and 421 were combined for precipitation.

Samples of the composite barren raffinate were analyzed for amine and for kerosene, in order that entrainment losses could be calculated. The raffinate contained 120 ppm kerosene and 9.8 ppm amine. These assays enabled the calculation of values for organic reagent consumption (Table 2).

TABLE 2

Reagent Consumption

(1) Average NaOH Consumption ($lb/lb\ U_3O_8$)

For pH control in stripping unit	= 1.058
Neutralization requirement	= 0.281
Total	= 1.339

(2) Average Organic Reagent Consumption ($lb/lb\ U_3O_8$)

Amine (Alamine)	= 0.02
Kerosene	= 0.30

Table 3 presents a record of the important specification assays for the composite precipitate from the six runs. Only the fluorine assay is somewhat higher than the specification limit which is set at 0.10 part F per 100 parts U3O8. The assay indicates 0.104 part F per 100 parts U3O8 in the composite final product.

The composite barren raffinate assayed 15.1 g $SO_4^{=}$/l and 0.017 g/Cl^-/l.

TABLE 3

Composite Precipitate Assays (%)
(Chemical Laboratory Sample Number - 2750)

U_3O_8	86.22
ThO_2	<0.003
F	0.09
Cl	0.007
Ti	<0.01
SO_4	3.5
$(RE)_2O_3$	<0.004
Mo	0.002
P_2O_5	0.006
B	<0.001
Acid Insoluble	0.16
CaO	<0.02
Moisture	0.18

The conclusion drawn from this study is that Alamine is a satisfactory organic extractant for the uranium contained in Rexspar leach liquors.

REFERENCES

1. V.F. Harrison - Autooxidation Pressure Leach Investigation of Uranium-bearing Ore from Rexspar Uranium and Metals Mining Co. Ltd., Birch Island, Kamloops Mining Division, B.C., Reference No. 11/57-7. Mines Branch Investigation Report IR 58-85, Department of Mines and Technical Surveys, Ottawa, Canada, May 13, 1958.

2. V.M. McNamara - Recovery of Uranium from Rexspar (Birch Island, B.C.) Leach Solution by Solvent Extraction with Tri-iso-octylamine, Reference No. 11/57-7. Mines Branch Investigation Report IR 58-86, Department of Mines and Technical Surveys, Ottawa, Canada, May 13, 1958.

APPENDIX 1

LEACHING

Supplementary to the test work described in Mines Branch Investigation Report IR 58-85, leaching of Rexspar ore (Sample No. 11/57-7), was carried out at the Mines Branch to supply a quantity of pregnant solution for the Alamine solvent extraction investigations. Nine 230-lb batches of ground ore (75-80% minus 200 mesh) were leached under a pressure of 200 psig, with an air flow rate of 17.3 scfm/sq ft pulp surface area, for 6 hours retention time at 300°F temperature. The leach pulps from each batch were filtered on an 18 in. dia. x 12 in. drum filter using one, 1/4% acid displacement wash. The filtrate was sampled and submitted to the solvent extraction section where it was diluted with enough water, mildly acidified with H_2SO_4, to give a solution-to-ore ratio of 2.3 to 1.0. The solvent extraction feed assayed about 0.5 g U_3O_8/l.

Results of the leach tests, shown in Table 4, agree with those in the previous report on Rexspar ore. In Test 31, extraction of uranium was 88.2% indicated by head and washed solids residue assays of 0.11 and 0.013% U_3O_8, respectively. The high uranium soluble losses in filtering were attributed to the use of a single wash on the filter. Earlier tests proved soluble losses to be negligible.[1]

TABLE 4

Pressure Leach Test Results

Test No.	Leach Feed Assay (% U_3O_8)	Final Washed Solids Residue Assay (% U_3O_8)	U_3O_8 Extraction* (%)	Final Leach Slurry pH	Filter Residue Assay (% U_3O_8)	Soluble Loss (% U_3O_8)	W.S. Leach Liquor		Undiluted Filter Filtrate	
							pH	Assay (g U_3O_8/l)	pH	Assay (g U_3O_8/l)
25	0.13	0.024	81.6	1.78	0.060	0.036	1.80	1.78	1.73	-
26	0.15	0.026	82.7	1.65	0.063	0.037	1.65	1.84	1.86	-
27	0.13	0.021	83.8	1.48	0.048	0.027	1.48	1.68	1.80	-
28	0.12	0.020	83.4	1.70	0.046	0.026	1.69	-	1.81	1.26
29	0.12	0.021	82.6	1.70	0.051	0.030	1.69	1.60	1.89	1.13
30	0.11	0.015	86.4	1.65	0.050	0.035	1.65	1.41	1.80	1.13
31	0.11	0.013	88.2	1.65	0.026	0.013	1.71	1.45	1.90	1.22
32	0.11	0.020	81.8	1.50	0.032	0.012	-	1.46	1.95	1.14
33	0.10	0.016	84.0	1.65	0.048	0.032	-	1.18	1.95	0.98

* Percent extraction was based on U_3O_8 assays of the leach feed and final washed solids.

APPENDIX 2

Batch Test Evaluation of Rohm and Haas Amine LA 1 as Extractant for Rexspar Leach Solution

At the request of the company and its consultants, the commercially available secondary amine LA-1 (formerly 9D-178), supplied by Rohm and Haas Co., was also used, in a series of shake-out tests, to establish the equilibrium curve and the possible uranium loading. The test was done with the same leach solution as used in the continuous runs described in the main body of this report. A 300-ml volume of 5% v/v amine LA-1 plus 2% primary decyl alcohol in kerosene was contacted with ten successive 450-ml volumes of leach solution. A similar test was completed with 5% Alamine taken from the circuit, after a carbonate scrub. Results are shown in Table 5.

TABLE 5

Results of Cross-Current Shake-Out Tests

Stage No.	U_3O_8 Assays (g/l)			
	LA-1		Alamine	
	Raffinate	Extract (calc)	Raffinate	Extract (calc)
0	0.39	0.29	0.39	0.00
1	0.002	0.87	0.0007	0.585
2	0.002	1.455	0.002	1.167
3	0.004	2.035	0.003	1.748
4	0.008	2.607	0.004	2.328
5	0.016	3.168	0.005	2.906
6	0.038	3.696	0.009	3.478
7	0.12	4.101	0.014	4.043
8	0.28	4.266	0.021	4.597
9	0.36	4.311	0.037	5.127
10	0.37	4.33	0.078	5.595
10		4.20 (assayed)		5.42 (assayed)

Equilibrium data, as shown, indicates a somewhat similar distribution coefficient for the two extractants, for loadings of 1 g U_3O_8/l or less. Saturation loading for LA-1 was 4.2-4.3 g U_3O_8/l; saturation for Alamine was not reached in 10 stages but continuous test data indicate a value of 6.0 g U_3O_8/l. Stripping tests, on 250 ml of the extracts obtained from the 10-stage loading, were done to determine NaOH consumption. These tests indicated that the caustic requirement was 0.9 lb NaOH/lb U_3O_8 for LA-1, and 0.80 lb NaOH/lb U_3O_8 for Alamine.

The above results show a slight advantage in the use of Alamine over Rohm and Haas LA-1. Since there was insufficient leach solution for continuous extraction tests, it was impossible to determine other factors such as soluble loss and selectivity.

VMMc/VFH/WAG/dm