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SOLVENT EXTRACTION TREATMENT OF SOLUTION PRODUCED BY AIR OXIDATION PRESSURE LEACHING OF URANIUM-BEARING ORE FROM KLERKSDORP CONSOLIDATED GOLDFIELDS LIMITED, SOUTH AFRICA

by

V. M. McNAMARA & W. A. GOW

RADIOACTIVITY DIVISION

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SUMMARY OF RESULTS

Klerksdorp acid leach solutions, containing an average 1.75 g U_3O_8/l, were successfully treated by the solvent extraction process for uranium recovery. The efficiencies of two amine extractants were studied. The amines were Alamine (a tertiary amine) and Amberlite LA-1 (a secondary amine). The amine concentration was approximately 0.1N in kerosene. Uranium recoveries of greater than 99.9% were readily attainable.

The precipitates, containing an average 75 to 80% U_3O_8 (dry basis), were able to meet all the requirements of the refinery specification for impurities.

Complete operational data are presented. Make-up to replace solvent losses was shown to be a minor process cost.

Average sodium hydroxide consumption under operating conditions, (the principal reagent cost), was 1.09 lb/lb U_3O_8 for the Alamine circuit, compared to 1.30 lb/lb U_3O_8 for the Amberlite LA-1 circuit.

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INTRODUCTION

This study was undertaken in conjunction with pilot plant pressure leaching studies on a bulk sample of uranium ore (Sample 5/58-9) from Klerksdorp Consolidated Goldfields Limited, South Africa[1].

The uranium-bearing leach solutions were treated for uranium recovery by the solvent extraction process. At the same time, a comparative study was made of the efficiency of the tertiary amine Alamine, and the secondary amine Amberlite LA-1.

The main objectives in carrying out the solvent extraction test work described in this report were as follows:

(A) To obtain sufficient operating data to indicate the efficiency that may be expected in the extraction of uranium from Klerksdorp leach solutions with 0.1N (approx.) Alamine in kerosene, followed by stripping with 15% sodium sulphate solution at pH 4.5 to 5.0.

(B) Similarly, to test the operating efficiency of 0.1N (approx.) Amberlite LA-1 in kerosene as an extractant for uranium from the Klerksdorp leach solutions, and to compare the results obtained with those resulting from uranium extraction with Alamine.

(C) To obtain sufficient uranium concentrate by caustic neutralization of the sodium sulphate strip solutions for complete product analyses, and to enable determination of reagent consumptions.

Alamine is a product of General Mills Inc., Chemical Division, and is a tri-fatty amine. It is now produced commercially.

Amberlite LA-1 is a commercially available secondary amine, formerly known as Amine 9D-178. It is produced by Rohm and Haas Company, Philadelphia, Pa.

The kerosene employed was supplied by Imperial Oil Limited, and is representative of regular production from their Montreal East refinery ($\simeq 10\%$ aromatic content). In each case, the solvent contained 2% n-decyl alcohol as a modifier to prevent third phase formation in the settlers.

GENERAL SUMMARY

A series of 21 runs was completed in the continuous counter-current solvent extraction pilot plant unit[2][3] in order to recover the uranium from Klerksdorp (Sample 5/58-9) pressure leach solutions. Seven runs, in which 835 litres of solution was treated, involved the use of 0.105N Alamine as the extractant. A further fourteen runs, in which 1390 litres of solution was treated, involved the use of 0.095N Amberlite LA-1 as the extractant.

The uranium loadings on the Alamine extractant ranged from 5.3 to 6.5 g U_3O_8/l; the upper limit representing saturation. The uranium loadings on LA-1 ranged from 3.6 to 3.9 g U_3O_8/l with the higher value representing saturation. The average uranium concentration in the aqueous feed solution was 1.75 g U_3O_8/l.

The loaded solvent was stripped in three counter-current stages with 15% sodium sulphate solution maintained at pH 4.5 to 5.0 by the addition of sodium hydroxide solution. The high-grade strip solution was then precipitated at pH 7.0 by means of further addition of sodium hydroxide solution to produce a uranium cake assaying, on a dry basis, 80.2% U_3O_8 for the composite product from the Alamine circuit and 75.8% U_3O_8 for the composite product from the Amberlite LA-1 circuit. The precipitates met all the requirements of the refinery specification for impurities.

Reagent consumptions consisted of the sodium hydroxide for pH control and for precipitation, plus the solvent losses to the raffinate.

Table 1 summarizes the data obtained from the test work and the calculated reagent consumptions.

Sodium sulphate was produced by the reactions involved in the stripping of the amine sulphate. As a result, it was not necessary to adjust the sodium sulphate concentration in the strip solution before recycling.

No trouble was experienced in handling the uranium product. It can be made to settle readily with Separan and glue, and it filtered at the rate of 2 to 3 lb/sq ft/hr.

DESCRIPTION OF CIRCUIT

Figure 1 represents the flowsheet of the continuous solvent extraction unit. Clarified leach solution from the pressure leaching was adjusted to pH 1.6 by sulphuric acid addition and fed, through a flow-

TABLE 1

Summary of Test Data and Reagent Consumptions

Conditions	Saturation	Operating		Saturation		Operating	
Run Nos.	432	431 and 436	433-435	439-441	449-450	442-446	447, 448, 451
Aqueous Feed Solution							
U_3O_8 (g/l)	1.79	1.80	1.66	1.56	1.81	1.76	1.74
pH	1.60	1.60	1.60	1.60	1.60	1.60	1.60
Solvent							
Amine	Alamine	Alamine	Alamine	LA-1	LA-1	LA-1	LA-1
Normality	0.105	0.104	0.105	0.093	0.095	0.095	0.095
Strip Solution							
Na_2SO_4 (g/l)	164	164	165	166	151	167	159
Flowrates (ml/min)							
Aqueous Feed	375	343	279	289	326	218	306
Solvent	91	91	92	91	149	107	149
Strip Solution	27	27	27	28	49	26	48
U_3O_8 assays (g/l)							
Final Barren	0.24	0.013	0.001	>0.3	0.23	0.004	0.002
Recycle Solvent	0.018	0.015	0.038	0.036	0.73	0.058	0.038
Loaded Extract	6.37	6.30	5.67	3.78	3.66	3.76	3.59
Strip Solution	21.02	18.60	20.49	12.51	9.48	14.54	10.29
Uranium Recovery (%)	86.6	99.3	99.9	<80	87.3	99.8	99.9
Reagent Consumption (lb/lb U_3O_8)							
a. NaOH							
Total	1.00	1.08	1.10	1.20	1.21	1.29	1.31
% for Precip.	28	25	26	27	25	24	24
b. Amine							
Soluble		N.D.*			N.D.*		
Entrainment		0.004			0.007		
c. Kerosene							
Entrainment		0.014			0.012		

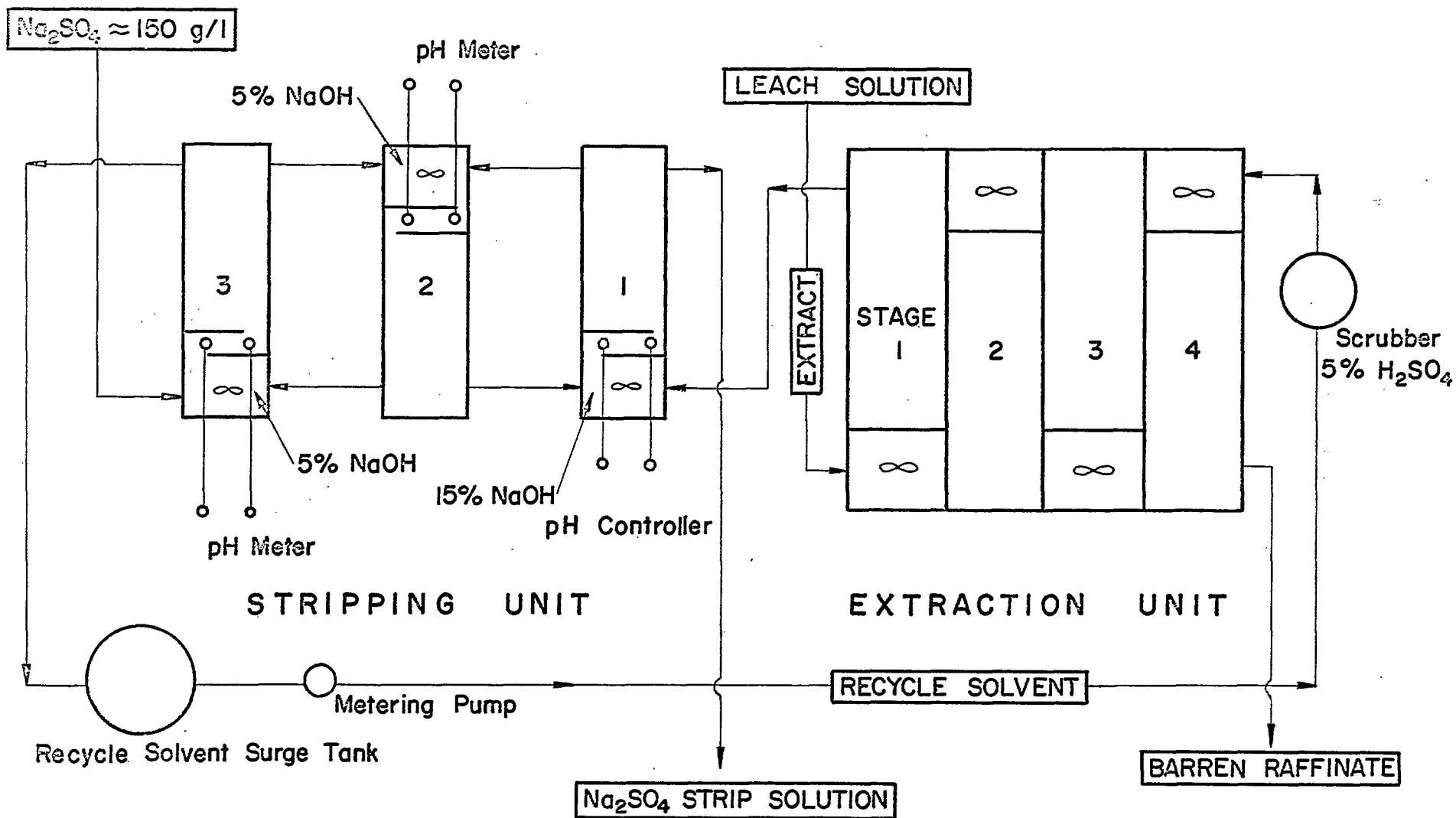* Not detectable by titration of recycling organic phase

FIGURE I

FLOWSHEET OF EXTRACTION AND SULPHATE STRIP CIRCUITS

meter, to stage 1 of 4 stages of counter-current mixer-settlers.

The solvent was pumped from the recycle surge-tank, by an Adjust-O-Feeder metering pump, to a dilute acid scrubber (5% H_2SO_4), and then to the 4 stage extractor. The purpose of the scrubber was to sulphate the amine (which is substantially in the free base form after stripping) and so prevent any major rise in pH of the aqueous at stage 4.

For sodium sulphate stripping, three stages of counter-current mixer-settlers were used. In order to control the pH, glass electrode-calomel half-cell combinations were placed in the semi-settled zones of the double baffle. An automatic pH recorder-controller was used for stage 1, where 15% sodium hydroxide was added, and the pH was controlled at a value of 4.8. Manual control was used in stages 2 and 3, with additions of 5% sodium hydroxide to maintain the pH at 4.5 to 5.0.

OPERATING PROCEDURE

The controlling factor in a solvent extraction circuit for uranium recovery from leach solutions is the retention time required for adequate stripping of the solvent in the stripping unit mixers. About 2 minutes retention time should be allowed, as a minimum, in order to ensure complete stripping.

From previous data, a satisfactory operating uranium loading for Alamine of about 6 g U_3O_8/l of solvent extract could be assumed. The uranium concentration in the strip solution should not be allowed to go much above 20 g U_3O_8/l if precipitation in the number 1 stripping cell is to be avoided. Therefore, the aqueous feed flow rate and the strip solution flow rate were set accordingly.

Table 2 gives the average solution retention times in each cell for both the extraction and stripping units.

The circuit (Figure 1), including the surge tank, contained about 18 litres of solvent. The duration of the run was sufficient in all cases to allow at least three solvent cycles per run.

Near the end of each run, when the system was in equilibrium, representative samples were taken for uranium analysis. These consisted of samples of the aqueous layer in all four stages of the extractor, loaded extract from stage 1 of the extractor, recycle solvent from the solvent surge tank, and final strip solution from the collecting carboy. A second sample of the recycle solvent was taken for amine normality determination. A leach solution sample was also obtained for each run.

Values for total sodium hydroxide consumption were based on the volumes required to control the pH of the strip circuit, and to neutralize the strip solution to produce the uranium precipitate.

Values for the entrainment loss of amine were determined by a colorimetric method for the amine in the barren raffinate. Kerosene loss in the barren raffinate was determined by direct infra-red analysis of a carbon tetrachloride extract of the raffinate sample.

The decrease, if any, in the amine normality as the test work progressed would give a basis for computing soluble losses of the amine. This study indicated that there was no detectable soluble amine loss.

Sodium hydroxide solution (15% w/v) was used to neutralize the high-grade strip solution to a pH of 7.0. To aid settling of the uranium

TABLE 2

Summary of Solution Retention Time and Solvent Entrainment Losses to Barren Raffinate

Run Nos.	Phase	Extraction				Stripping	
		Retention Time (min)		Entrainment Loss (lb/lb U_3O_8)		Retention Time (min)	
		Mixer	Settler	Amine	Kerosene	Mixer	Settler
431 and 433-436	Organic	4.0	12.6	-	-	3.3	6.6
	Aqueous	1.5-1.8	4.7-5.7	0.004	0.014	11.1	22.2
442-446	Organic	3.4	11.7	-	-	2.8	5.6
	Aqueous	2.3	7.3	0.007	0.012	11.5	23.1
447, 448, 451	Organic	2.4	7.7	-	-	2.0	4.0
	Aqueous	1.6	5.2	0.007	0.011	6.3	12.5

precipitate, a solution of Separan (0.1%) was added slowly with gentle stirring, to provide a total of 2 ml per litre of high-grade strip, followed by a 1% solution of glue to provide a total of 8 ml per litre of strip solution. The precipitate settled to 20% of the original volume and at this point the supernatant solution was decanted onto the filter followed by the precipitate slurry.

The precipitate was washed twice, each wash consisting of 30 ml of water per litre of strip solution.

RESULTS AND DISCUSSION

Leach Solution Assays

Composite samples of leach solution, obtained from the pilot plant leaching of Klerksdorp ore, were assayed for the significant elements. The results are shown in Table 3. These solutions were used as feed to the solvent extraction unit. The following conclusions are drawn from the assay results:

a) The leach solution should be amenable to the recovery of uranium by the solvent extraction process. (The present work confirms this to be the case).

b) The leach solution should be amenable to recovery of uranium by the ion exchange process. A leach solution pH of 1.7 to 1.8 would be optimum. There is a possibility that, if ion exchange were used, the product specification on fluorine might be exceeded.

c) The leach solution assays substantiate the visual observation[1] that there was no appreciable corrosion of the stainless steel pressure tower during the leaching of Klerksdorp ore.

TABLE 3

Klerksdorp Composite Leach Solution Assays

	Alamine Circuit Feed Solution	LA-1 Circuit Feed Solution	Overall Feed Solution Composite
Run Nos.	431-436	439-451	431-451
Assay Ref. No.	RE3375	RE3772	RE3799
pH	1.6	1.6	1.6
Assay	g/l	g/l	g/l
U_3O_8	1.75	1.74	1.76
Fe total	1.50	2.17	-
Fe^{3+}	0.75	1.03	-
Cl+Br+I	0.01	0.005	-
F	0.33	0.31	-
Mn	0.29	0.28	-
SiO_2	0.72	0.70	-
Free H_2SO_4	3.28	2.85	-
$Na_2S_4O_6$	<0.01	<0.01	-
ThO_2	-	-	0.03
$(RE)_2O_3$*	-	-	2.17
Ti	-	-	<0.01
V_2O_5	-	-	<0.01
SO_4	-	-	25.5
As+P_2O_5	-	-	0.03
Al	-	-	1.52
Ce	-	-	0.46
Zr	-	-	<0.0001
Mo	-	-	<0.0025
Heavy metals**	-	-	0.32
Ni	-	-	0.16
Cr	-	-	0.005
Co	-	-	0.084

*$(RE)_2O_3$ - rare earths oxides
**Heavy metals are Pb, Bi, As, Cu, Sb

Continuous Tests

The operating data obtained from the solvent extraction pilot plant are contained in Table 4.

It was found that both Alamine and Amberlite LA-1 are effective for the extraction of uranium from the Klerksdorp leach solutions. Neither amine presented any difficulties due to poor phase separation or to losses of amine due to solubility in the raffinate (requiring make-up of recycle solvent). In each case the diluent was kerosene from Imperial Oil Ltd. The Alamine required the addition of 2% n-decyl alcohol to improve phase separation. The Amberlite LA-1 did not require the addition of a modifier.

Extraction

The maximum uranium loading for Alamine was 1.6 times that for Amberlite LA-1. However, the Alamine normality was 1.1 times the Amberlite LA-1 normality. The operating uranium loading for Alamine is, therefore, approximately 1.5 times the equivalent uranium loading obtained on the Amberlite LA-1, which is a considerable advantage.

When Amberlite LA-1 was used as the extractant, the sodium hydroxide consumption increased by 19%.

Stripping

Careful control of the pH in the three stripping stages was required to ensure satisfactory sulphate stripping. If the pH fell much below 4.5, inefficient stripping resulted. If the pH increased above 5.5, then precipitation of uranium in the cells occurred. Furthermore, a pH

TABLE 4

Operating Data

Run Number	430	431	432*	433	434	435	436	438	439*	440*
Aqueous Feed Solution										
U_3O_8 (g/l)	1.42	1.83	1.79	1.74	1.61	1.63	1.76	1.70	1.38	1.62
pH	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
Operating Time (hrs)	6	6	6	6	6	7	6	6	6	6
Solvent										
Type	Alamine	Alamine	Alamine	Alamine	Alamine	Alamine	Alamine	LA-1	LA-1	LA-1
Normality	0.104	0.102	0.105	-	0.106	0.105	0.106	0.096	0.094	0.092
Strip Solution										
Na_2SO_4 (g/l)	160	158	164	166	166	163	170	158	166	166
Flowrates (ml/min)										
Feed Solution	375	364	375	271	286	279	322	333	342	290
Solvent	91	91	91	91	92	92	91	91	91	91
Strip Solution **	26.8	26.7	26.8	26.2	27.7	27.7	27.9	29.1	29.6	28.9
Scrub	17.5	28.6	27.2	24.2	26.3	27.1	28.3	23.1	25.6	23.3
U_3O_8 Assays (g/l)										
Aqueous: Stage 1	1.24	1.60	2.06	0.32	0.19	0.08	0.19	1.85	1.43	1.40
Stage 2	0.81	0.78	1.85	0.04	0.016	0.022	0.008	1.85	1.43	1.40
Stage 3	0.05	0.07	1.55	0.005	0.002	0.002	0.001	>0.8	>0.8	>0.5
Final Barren Stage 4	0.009	0.024	0.240	0.002	0.0005	0.0006	0.001	>0.6	>0.4	>0.4
Recycle Solvent	0.016	0.012	0.018	0.014	0.069	0.031	0.018	0.17	0.16	0.18
Loaded Extract	6.28	6.57	6.37	6.02	5.66	5.33	6.02	3.95	3.85	3.75
Strip Solution	12.03	18.70	21.02	22.77	21.07	17.64	18.49	9.64	12.45	11.75
Uranium Recovery (%)	99.4	98.7	86.6	99.9	99.9	99.9	99.9	<65	<71	<75
Precipitation										
Strip Volume (l)	9.66	9.61	9.65	9.42	9.98	11.63	10.04	10.48	10.65	10.42
Weight of Cake (g)										
Wet	515	475	-	663	608	643	535	570	425	499
Dry	170	230	262	263	263	235	220	150	165	170
U_3O_8; Total Weight (g)*	116.3	179.7	203.0	214.4	210.3	205.2	185.7	101.0	132.6	122.5
NaOH Consumption (lb/lb U_3O_8)										
Stripping	1.37	0.87	0.72	0.73	0.81	0.90	0.74	1.06	0.87	0.89
Neutralization	0.37	0.26	0.28	0.29	0.28	0.29	0.28	0.34	0.32	0.34
Total	1.74	1.13	1.00	1.02	1.09	1.19	1.02	1.40	1.19	1.23

* Runs continued until solvent saturated.
** Total Flow: Na_2SO_4 + NaOH solutions.
*** Based on strip solution assay.

Note: Runs 430 and 438 were conditioning runs. Results
are excluded from study since they are not
considered representative.

TABLE 4 (Continued)

Operating Data

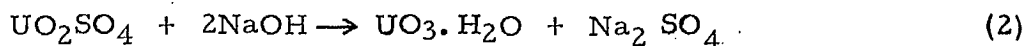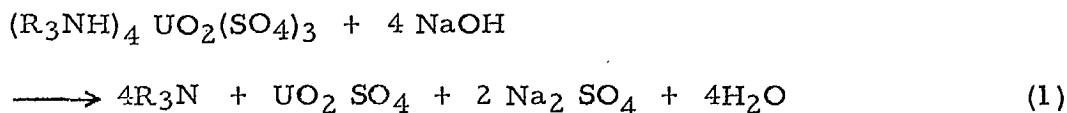| Run Number	441*	442	443	444	445	446	447	448	449*	450*	451
Aqueous Feed Solution											
U₃O₈ (g/l)	1.69	1.83	1.71	1.76	1.74	1.74	1.70	1.81	1.81	1.81	1.70
pH	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
Operating Time (hrs)	6	6	6	6	6	6	6	6	6	6	6
Solvent											
Type	LA-1	LA-1	LA-1	LA-1	LA-1	LA-1	LA-1	LA-1	LA-1	LA-1	LA-1
Normality	-	0.095	0.096	0.096	0.095	-	0.095	-	-	0.096	0.094
Strip Solution											
Na₂SO₄ (g/l)	-	164	164	166	166	173	160	158	150	152	-
Flowrates (ml/min)											
Feed Solution	236	230	211	210	222	219	281	303	333	318	333
Solvent	91	106	106	107	107	109	150	150	150	148	148
Strip Solution **	25.3	26.8	24.6	26.3	25.9	26.9	45.9	49.6	49.9	48.4	48.6
Scrub	24.7	22.5	21.7	23.6	25.3	26.4	33.3	31.4	33.6	35.0	33.4
U₃O₈ Assays (g/l)											
Aqueous: Stage 1	1.75	0.34	1.80	1.68	1.54	1.31	0.62	1.35	1.78	1.80	0.56
Stage 2	1.73	0.053	1.63	1.03	0.90	0.56	0.09	0.36	1.68	1.61	0.089
Stage 3	1.20	0.019	>0.3	0.03	0.036	0.03	0.003	0.020	1.42	>0.9	0.008
Final Barren Stage 4	0.11	0.004	0.011	0.002	0.002	0.003	0.0005	0.001	0.13	0.32	0.003
Recycle Solvent	0.074	0.069	0.047	0.110	0.025	0.041	0.40	0.27	0.62	0.84	0.48
Loaded Extract	3.73	3.78	3.79	3.78	3.75	3.68	3.56	3.56	3.62	3.69	3.65
Strip Solution	13.32	13.84	15.70	15.99	13.99	13.20	10.86	9.55	9.36	9.60	10.45
Uranium Recovery (%)	93.5	99.8	99.4	99.9	99.9	99.8	99.9	99.9	92.8	82.3	99.8
Precipitation											
Strip Volume (l)	9.11	9.65	8.87	9.27	9.33	9.67	16.54	17.87	17.95	17.43	17.48
Weight of Cake (g)											
Wet	315	654	579	564	474	390	609	589	624	519	714
Dry	145	199	179	199	164	173	239	244	244	224	269
U₃O₈; Total Weight (g)*	121.4	133.5	139.3	148.2	130.6	127.6	179.6	170.7	168.0	167.4	182.7
NaOH Consumption (lb/lb U₃O₈)											
Stripping	0.88	0.95	0.97	0.95	0.98	1.01	0.97	0.99	0.98	0.83	1.03
Neutralization	0.31	0.34	0.32	0.31	0.30	0.30	0.35	0.31	0.29	0.33	0.29
Total	1.19	1.29	1.29	1.26	1.28	1.31	1.32	1.30	1.27	1.16	1.32

* Runs continued until solvent saturated.
** Total Flow: Na₂SO₄ + NaOH solutions.
*** Based on strip solution assay.

greater than 5.5 would cause emulsification and subsequent co-current movement of aqueous and organic phases. Manual addition of dilute sodium hydroxide (5%) to stripping cells 2 and 3 enabled efficient control of the pH in these cells.

Control of Sodium Sulphate Concentration in Stripping Solution

In the operation of the solvent extraction circuit, it is desirable to set conditions so that the sodium sulphate concentration of the recycled strip solution remains reasonably constant. This is accomplished by adjusting, where possible, those operating conditions which tend to change the sulphate strength of the strip solution.

The sulphate in the extract prior to stripping is recycled as sodium sulphate after uranium precipitation. (See chemical reactions 1 and 2). This tends to increase the sulphate concentration of the recycle strip solution and is a factor which cannot be controlled directly.

$$(R_3NH)_4 \ UO_2(SO_4)_3 \ + \ 4 \ NaOH$$

$$\longrightarrow 4R_3N \ + \ UO_2 \ SO_4 \ + \ 2 \ Na_2 \ SO_4 \ + \ 4H_2O \qquad (1)$$

$$UO_2SO_4 \ + \ 2NaOH \longrightarrow \underline{UO_3.H_2O} \ + \ Na_2 \ SO_4 \qquad (2)$$

On the other hand, the sodium sulphate concentration of the recycled strip solution is reduced by (a) dilution of the recycle by the addition of the water contained in the sodium hydroxide solution used to control the pH in the stripping circuit and in the precipitation step; (b) dilution, amounting to about 1% of the strip solution volume, due to the chemical reaction (1) above; (c) dilution, amounting to about 1% of the

strip solution volume, due to the water contained in the dilute solutions

of Separan and glue used as flocculants in the precipitation step. Of

these factors, the dilution due to (a) may be varied in order to counteract

the increase in sodium sulphate concentration discussed in the previous

paragraph.

In the present study, the sodium sulphate concentration was

maintained reasonably steady by employing the following solutions for

pH control:

pH control in stripping cell No. 1	15% NaOH
pH control in stripping cells Nos. 2 and 3	5% NaOH
neutralization of high-grade strip	15% NaOH.

The quantity of sodium sulphate solution formed was rather

large. It was found convenient to allow complete settling of the precipitate

and then to decant the supernatant liquor for recycling to the stripping unit.

The filtrate from the slurry filtration (20% of total precipitation feed) was

discarded and this constituted a bleed for removing excess sodium

sulphate from the system.

Assays obtained on the strip solution from runs 436 and 451

(Table 5) show that there was no build-up during the investigation of either

chloride or fluoride. Furthermore, there was no serious increase in the

concentrations of fluoride and chloride ions in the recycling organic phase.

Initial organic phase and initial aqueous strip solution contained no

halogens. The concentration of fluoride (0.3 g F/l) in the leach solution

was rather high.

TABLE 5

Chloride and Fluoride Assays on Recycled Solutions
(g/l)

Run No.	Organic Recycle Solution		Strip Solution	
	436	451	436	451
Cl$^-$	0.010	0.005	0.073	0.034
F$^-$	0.04	0.06	0.01	0.007
Na$_2$SO$_4$	-	-	-	155

Solvent Losses

Solvent losses of Alamine and Amberlite LA-1 due to solubility in the aqueous phase are negligible. No decrease in amine normality was recorded within the extent of this work (Tables 1 and 4).

Losses of kerosene and amine, due to entrainment in the barren raffinate, are shown in Tables 1 and 2.

Sodium Hydroxide Consumption

Average values for the reagent consumption, for saturation loading conditions and for operating loading conditions, were shown in Table 1.

The overall average sodium hydroxide consumption for conditions that would prevail in plant operation are as follows:

Solvent	Sodium Hydroxide Consumption (lb/lb U$_3$O$_8$)
Alamine	1.09
Amberlite LA-1	1.30

Grade of Product

Table 6 presents the results of comprehensive analyses of the composite precipitates from both the Alamine circuit and the Amberlite LA-1 circuit. All Canadian refinery specification requirements are met.

It is known that tertiary amines are more selective than secondary amines. The precipitate analyses demonstrate this fact rather well. Several ions are much more strongly adsorbed by the secondary amine Amberlite LA-1. Examples are fluoride, thorium, the rare earths and phosphorus. The only anomaly is presented by zirconium, whch is more strongly adsorbed by the tertiary amine at equivalent pH values.

REFERENCES

1. Harrison, V. F., McCreedy, H. H., Honeywell, W. R., and Gow, W. A., "Ore Treatment Investigations of Uranium-Bearing Ore from Klerksdorp Consolidated Goldfields Ltd., South Africa" Mines Branch Investigation Report IR 58-205, Department of Mines and Technical Surveys, Ottawa, Canada, November 26, 1958, (Industrial Confidential).

2. Simard, R., "Solvent Extraction Studies, Progress Report September 1955 to January 1956", Radioactivity Division Special Report No. SR-416/56, Department of Mines and Technical Surveys, Ottawa, Canada, April 6, 1956.

3. Bellingham, A., and Simard, R., "Recovery of Uranium from Rexspar Leach Solution by Solvent Extraction with Tri-iso-octylamine", Radioactivity Division Special Report No. SR-468/57, Department of Mines and Technical Surveys, Ottawa, Canada, March 29, 1957, (Industrial Confidential).

TABLE 6

Klerksdorp Composite Precipitate Assays
(%)

Extractant	Alamine	LA-1
Run Nos.	431-436	439-451
Assay Ref. No.	RE3863	RE3864
U_3O_8	77.98	73.74
Cl	0.005	0.005
F	0.0015	0.024
Fe	<0.05	0.078
H_2O	2.72	2.70
ThO_2	0.01	0.60
SO_4	7.11	9.06
$(RE)_2O_3$*	<0.006	0.03
SiO_2	0.05	0.06
Acid insoluble	0.09	0.11
Mo	<0.001	<0.001
As	<0.01	<0.01
P_2O_5	0.003	0.022
V_2O_5	<0.01	<0.002
CeO_2	<0.004	0.006
Ti	<0.01	<0.01
Mn	<0.02	<0.01
Ni	<0.004	<0.004
Cr	<0.001	<0.001
Co	<0.005	<0.005
Al	0.0048	0.0096
Sr	<0.002	<0.002
ZrO_2	0.045	<0.002
B	<0.001	<0.001

*$(RE)_2O_3$ - rare earths oxides

APPENDIX 1

Determination of Amine Normality

This determination was done on a sample taken from the solvent surge-tank at the end of each run. The steps were:-

1. Measure 30 ml of solvent sample into a 100-ml separatory funnel.

2. Add 30 ml of 10% hydrochloric acid. Shake well and allow layers to separate.

3. Discard bottom (aqueous) layer.

4. Filter top layer through dry paper to remove entrained aqueous solution. The dry filter paper absorbs traces of acid.

5. Pipette 10 ml of filtered organic solvent into a beaker. Add 100 ml of water.

6. Titrate with standardized 0.1N sodium hydroxide solution to pH 7.0.

$$\text{Amine Normality} = \frac{\text{Vol NaOH } \times \text{ Normality NaOH}}{\text{Vol sample (10 ml)}}$$

APPENDIX 2

Extraction Equilibrium Data

Batch shake-out tests were performed in order to establish the uranium distribution between Klerksdorp leach solution and two amines*considered as possible extractants. The data was used for a preliminary comparison of amine uranium capacities and distribution coefficients. The leach solution used for these tests was obtained from a preliminary small-scale batch pressure leach of Klerksdorp ore. The solution was at pH 1.6 and assayed 1.17 g U_3O_8/l.

A 250-ml volume of 5% v/v amine in Imperial "Esso" kerosene (plus 2% n-decyl alcohol with the Alamine) was scrubbed with a 100-ml volume of 5% sulphuric acid and then contacted for 5 minutes with 5 successive volumes of leach solution (1 x 125 ml; 4 x 250 ml). The five raffinates and the final extract were assayed for U_3O_8 and the results are presented in Table 7.

Equilibrium curves plotted from the data on Table 7 are shown in Figure 2. The equilibrium data for Alamine show that it is more suitable than Amberlite LA-1. The saturation uranium loading of Alamine was considerably higher (1.3 times) than that of Amberlite LA-1, as confirmed by the continuous extraction runs. The efficiency of Alamine in the low concentration range is also superior.

Several stripping tests at pH 5.0, on the loaded amines, indicated that the efficiencies with which they were stripped in this system were comparable.

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*Alamine and Amberlite LA-1

The overall suitability of the two amines for the uranium extraction process was established in the present report, which includes a study of the soluble and entrainment losses.

TABLE 7

Extraction Equilibria

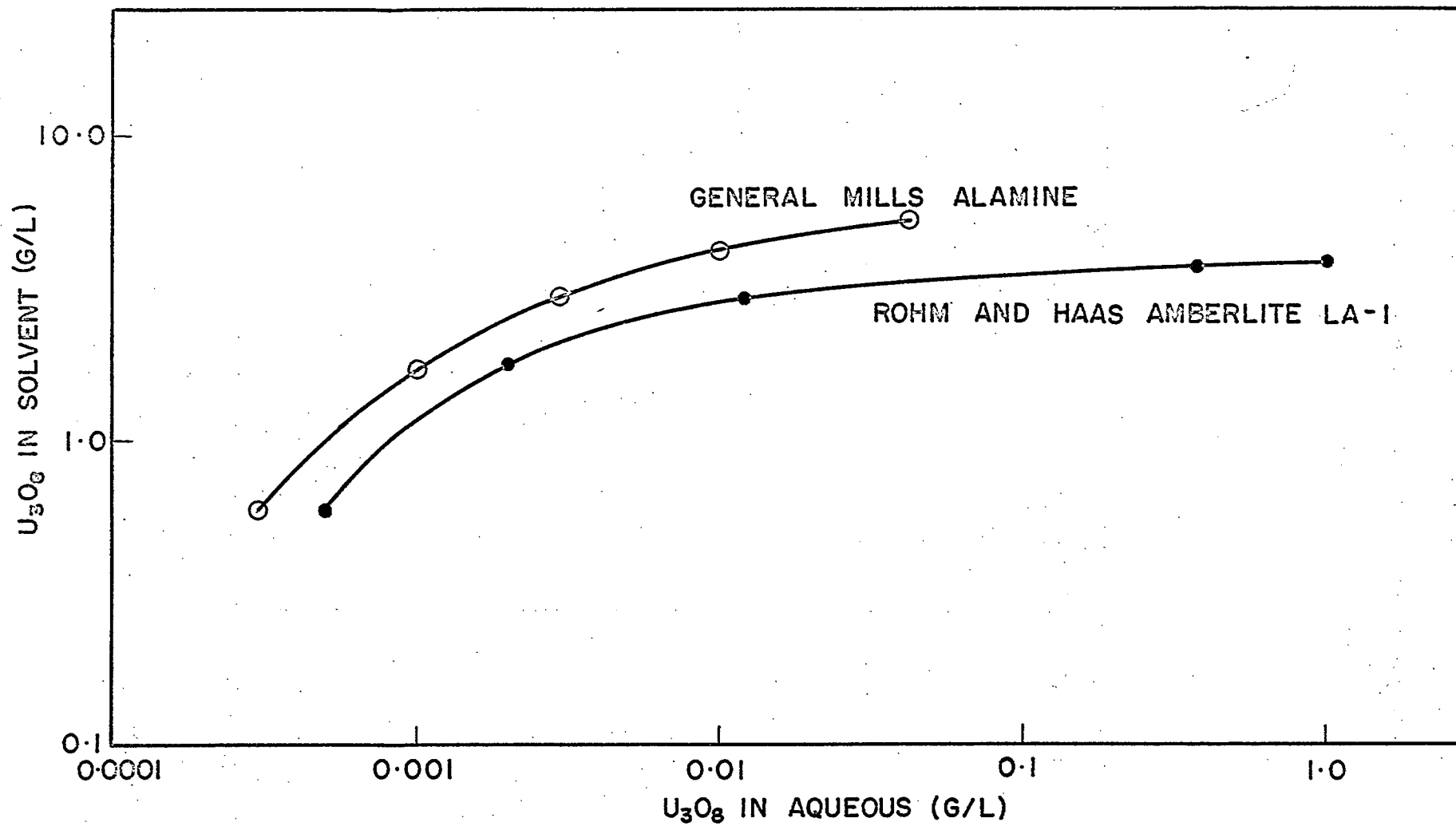| Extractant	Stage No.	$U_3O_8(g/l)$ in Raffinate (assay)	$U_3O_8(g/l)$ in Extract (calculated)	E_a^o
Alamine	1	0.0003	0.59	1950
	2	0.001	1.75	1754
	3	0.003	2.92	974
	4	0.010	4.08	408
	5	0.043	5.21	121
	Extract		5.15(assay)	
Amberlite LA-1 (9D-178)	1	0.0005	0.59	1170
	2	0.002	1.75	877
	3	0.012	2.91	243
	4	0.38	3.70	9.7
	5	1.03	3.84	3.7
	Extract		3.93(assay)	

VMM/WAG/eew

FIGURE 2

EQUILIBRIUM CURVES

Klerksdorp Leach Solution vs 5% Amine in Kerosene