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SOLVENT EXTRACTION TREATMENT OF SOLUTION PRODUCED IN ACID LEACHING OF URANIUM ORE FROM THE KITTS PROPERTY OF BRITISH NEWFOUNDLAND EXPLORATION LIMITED, NEWFOUNDLAND

by

V. M. McNAMARA, R. SIMARD & W. A. GOW

RADIOACTIVITY DIVISION

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SUMMARY OF RESULTS

Acid leach solutions, with uranium contents in the range of 2.6 to 6.3 g U_3O_8/1, were successfully treated by solvent extraction. Two extractants were studied: 0.1N Alamine in kerosene and 0.1N tri-iso-octylamine in kerosene. Uranium recovery was 99.9+% in precipitates containing an average of 80 to 85% U_3O_8. All impurity specifications on the high-grade uranium product were met. Complete operational data are presented. Solvent losses and reagent consumptions were determined.

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CONTENTS

(27 pages, 2 figures, 11 tables)

INTRODUCTION

This work was done in conjunction with pilot plant leaching

of bulk samples of uranium ore (Samples 4/58-12 and 5/58- 4) from

the Kitts property of British Newfoundland Exploration Limited at

Makkovik, Labrador, Newfoundland.[1] The resultant uranium-bearing

solutions were treated for uranium recovery by ion exchange[2], direct

precipitation[3], and solvent extraction processes.

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 can be expected in the extraction of uranium from
   Brinex * leach liquor with 0.1N tri-iso-octylamine in
   kerosene, followed by stripping with 15% Na_2SO_4 solution
   at pH 4.5 to 5.0.

B. To similarly test the operating efficiency of 0.1N Alamine
   in kerosene as an extractant for uranium from the Brinex
   leach solutions, and to compare the results obtained with
   those resulting from tri-iso-octylamine extraction.

C. To obtain sufficient precipitate, from caustic neutralization
   of the sodium sulphate strip solutions, for complete product
   analyses and to enable determination of reagent consumptions.

Tri-iso-octylamine was supplied by Carbide Chemicals Company.

The commercial production of the amine has been discontinued by the

company.

Alamine is a product of General Mills Inc, Chemical Division, and

is a tri-fatty amine. It is now produced commercially.

The two amines were employed as approximately 0.1N solutions

in B.A.**jet fuel kerosene. In each case the solvent contained 2% n-decyl

alcohol as a modifier to prevent third phase formation in the settlers.

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*Brinex - British Newfoundland Explorations Limited.
** B.A. - British-American Oil Company

GENERAL SUMMARY

A series of 25 runs was completed, in the continuous counter-current solvent extraction pilot plant unit, in order to recover the uranium from Brinex (Samples 4/58-12 and 5/58-4) acid leach solutions. Twelve runs, treating 2030 litres of solution, involved the use of 0.109N tri-iso-octylamine as the extractant. Another thirteen runs, treating 1155 litres of solution, involved the use of 0.124N Alamine as the extractant. The uranium loadings on the tri-iso-octylamine extractant ranged from 5.2 to 6.3 g U_3O_8/l, while the uranium loadings on the Alamine ranged from 5.4 to 7.6 g U_3O_8/l. The range of uranium concentrations in the aqueous feed solution was from 2.6 to 6.3 g U_3O_8/l, which resulted in the previously mentioned wide range of operational loadings.

The loaded solvent was stripped in three counter-current stages with 15% Na_2SO_4 solution maintained at pH 4.5 to 5.0 with NaOH solution. The high-grade strip was then precipitated at pH 7.0 by means of further addition of caustic, to produce an uranium cake assaying 80 to 85% U_3O_8 and which met Eldorado refinery feed specifications.

Reagent consumptions will consist of the sodium hydroxide for pH control and precipitation, plus the solvent losses in the raffinate.

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

TABLE 1

Summary of Test Data and Reagent Consumptions

Run Nos.	393-403	404-405	406-410	411-415	416-418
Leach Solution					
U_3O_8 (g/l)	3.05	5.80	6.25	6.25	3.34
pH	1.75	1.80	1.75	1.50	1.55
Solvent					
Amine	TIOA**	TIOA**	Alamine	Alamine	Alamine
Normality	0.109	0.109	0.124	0.124	0.124
Strip Solution					
Na_2SO_4(g/l)	132	129	136	153	156
Flowrates (ml/min)					
Feed	.289	188	191	199	285
Solvent	152	173	175	175	171
Strip	52	52	58	53	63
U_3O_8 assays (g/l)					
Final barren	0.002	0.002	0.002	0.001	0.0008
Recycle solvent	0.031	0.037	0.032	0.033	0.044
Loaded extract	5.68	6.23	6.80	7.07	5.74
Strip solution	17.09	20.55	19.10	22.26	14.39
Uranium Recovery (%)	99.93	99.97	99.97	99.98	99.98
Reagent Consumption (lb/lb U_3O_8)					
a. NaOH					
Total	1.04	1.00	0.99	1.15	1.41
% for precip.	27	26	29	26	16
b. Amine					
Soluble	N.D.*	N.D.	N.D.	N.D.	
Entrainment	0.018	0.004	0.002	0.007	
c. Kerosene					
Entrainment	0.069	0.015	0.005	0.055	

*N.D. - not detected
**TIOA- tri-iso-octylamine

Sodium sulphate is produced from the stripping of the amine sulphate. Consequently it was not necessary to make-up with Na_2SO_4 to maintain the concentration in the strip solution before recycling.

No trouble was experienced in handling the uranium product. It can be settled readily, with Separan and glue, and 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 pregnant leach liquor, after pH adjustment, was fed through a flowmeter to stage 1 of 4 stages of counter-current mixer-settlers.

The solvent flow was pumped from the recycle surge-tank, by an Adjust-O-Feeder, to a dilute acid scrubber (5% H_2SO_4), and then to stage 4 of the extractor. The purpose of the scrubber was to sulphate the solvent and so prevent any major rise in the pH of the aqueous flow at stage 4.

For Na_2SO_4 stripping, three stages of countercurrent mixer-settlers were used. In order to control pH, the electrodes were placed in the semi-settled zone of the double baffle. An automatic pH recorder-controller was used for stage 1, where 10 to 15% caustic was added. Control pH was 4.8. Manual control was used in stages 2 and 3, with additions of 1 to 5% caustic to maintain the pH at 4.5 to 5.0.
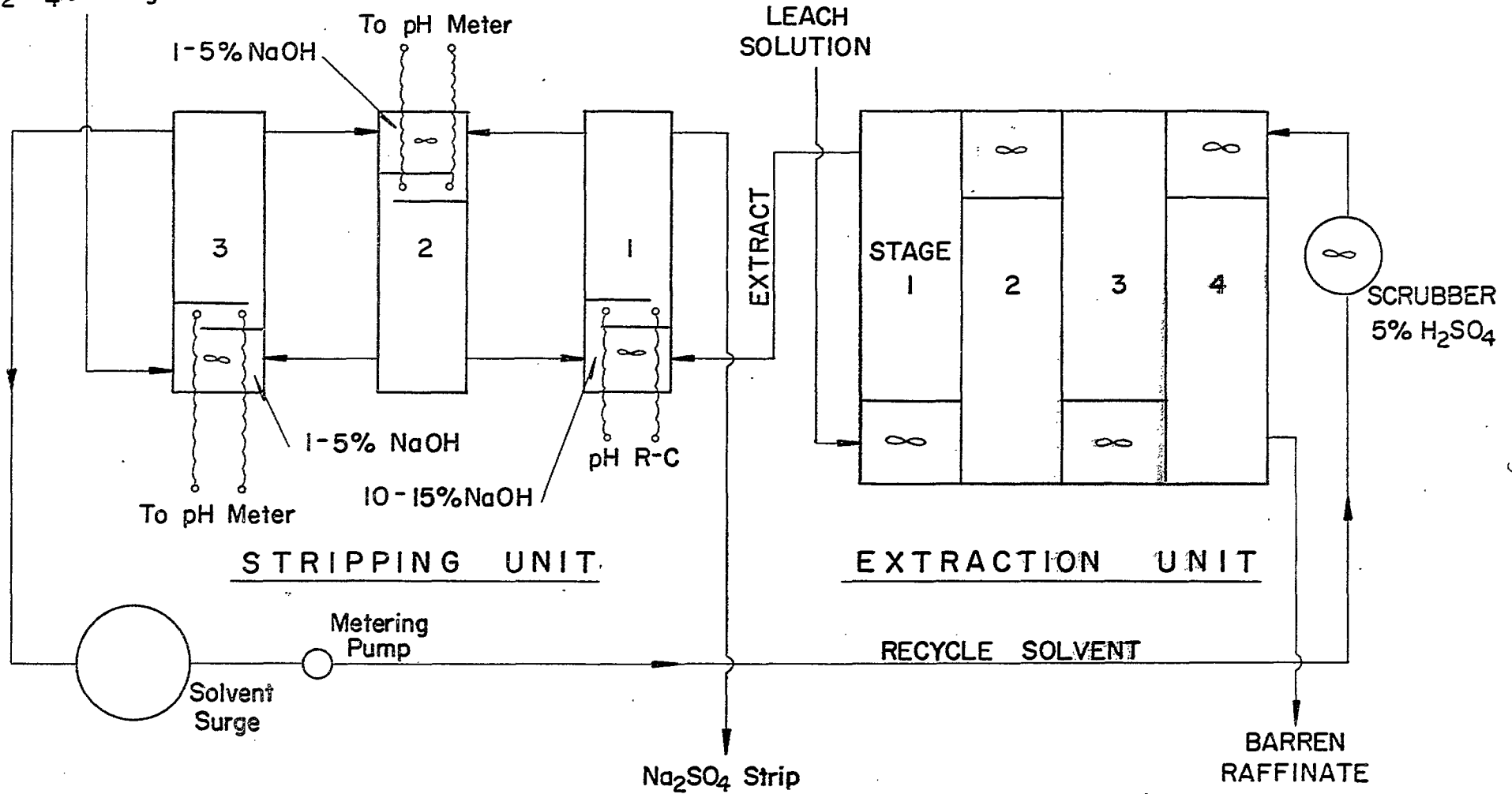
FIGURE 1

FLOWSHEET OF EXTRACTION AND SULPHATE STRIP CIRCUITS

OPERATING PROCEDURE

The limiting factor in the solvent extraction circuit for uranium recovery from pregnant leach solutions is the retention time of the solvent in the stripping unit mixers. About 1 3/4 to 2 minutes retention time should be allowed, as a minimum, in order to ensure complete stripping. From previous data an operating uranium loading of about 6 g U_3O_8/l of extractant could be assumed. Therefore, the leach solution flow rate and the Na_2SO_4 strip solution flow rate were set accordingly. The uranium concentration in the strip solution should not go much above 20 g/l if precipitation in the number 1 stripping cell is to be avoided.

Table 2 gives the solution retention time 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 analyses. 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.

TABLE 2

Solution Retention Time in Each Cell
(min)

Run Nos.	393 - 403		404 - 405		406 - 410		411 - 415		416 - 418	
	M*	S*	M	S	M	S	M	S	M	S
Leach Solution	1.75	5.50	2.75	8.50	2.50	8.50	2.50	8	1.75	5.50
Solvent Extraction	2.5	7.50	2	6.75	2	6.50	2	6.50	2	6.75
Stripping	2	4	1.75	3.50	1.75	3.50	1.75	3.50	1.75	3.50
Strip Solution	6	12	6	12	5.25	10.25	5.75	11.25	4.75	9.50

* M=Mixer
S =Settler

Total NaOH consumption was based on the volumes required to control the pH of the strip circuit, and to neutralize the strip solution to produce the uranium precipitate. Kerosene loss in the raffinate was determined by direct infra-red analysis of a carbon tetrachloride extract of raffinate samples. The loss of amine was computed from the analysis for NH_3 in the barren raffinate. The decrease, if any, in the amine normality, as the test work progresses, will give a basis for computing soluble losses of the amine. This study indicated that there was no detectable soluble amine loss.

NaOH solution was used to neutralize the high-grade strip solution to a pH of 7.0. To aid settling of the uranium precipitate, a solution of Separan (0.1%) was added slowly to a total of 2 ml per litre of high-grade strip, followed by a 1% solution of glue to a total of 8 ml per litre of strip solution, with gentle stirring. The precipitate settled to 20% of the total volume and the supernatant solution was decanted onto the filter, followed by filtration of the precipitate slurry.

The precipitate was washed twice with water; each wash equivalent to 30 ml of wash per litre of strip solution.

RESULTS AND DISCUSSION

Leach Solution Assays

Composite samples of pregnant solution obtained from the pilot plant leaching of both low and high-grade ore were assayed for the significant elements and results are shown in Table 3. These solutions, less the volumes required for ion exchange and direct precipitation studies, were used as feed to the solvent extraction unit.

TABLE 3

Leach Solution Assays
(g/l)

Leach Run No.	85	86	87
U_3O_8	3.17	3.43	5.95
ThO_2	0.01	0.003	0.002
Fe^3	0.30	0.60	0.8
Fe^2	4.80	5.0	4.6
TiO_2	0.07	0.07	0.04
Mo	0.0001	0.0002	0.0002
P_2O_5	0.087	0.29	0.16
V_2O_5	0.05	0.03	0.038
F	0.07	0.06	0.05
SO_4	21.5	27.3	26.1
Free H_2SO_4	1.05	3.1	0.85

Continuous Tests

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

It was observed that the two amines were equally efficient in all respects when used as the extractant for uranium from Brinex leach solutions.

Extraction

Maximum extract loadings for Alamine show values about 14% higher than those for TIOA. However, the Alamine normality was 14% higher than TIOA. (Table 1). In each case the amine diluent was B.A. jet fuel kerosene, with 2% n-decyl alcohol added to prevent third phase formation.

Two distinctly different concentrations of uranium leach solution were fed to the solvent extraction unit. One series of leach solutions averaged about 3 g $U_3O_8/1$. The other series of leach solutions averaged 6 g $U_3O_8/1$. Both grades of solution were amenable to treatment in both the tri-iso-octylamine and the Alamine extraction circuits. Extract uranium loadings were somewhat higher when treating the higher grade solution.

In the first 17 pilot plant runs, the leach solution pH was 1.75 to 1.80 (runs 393 to 410). In subsequent runs the leach solution pH was lowered to 1.50-1.55. This change in pH did not affect operating efficiency in the extractors. The change was necessitated by the presence of excessive, white precipitate which built

TABLE 4

OPERATING DATA

Run No.	392	393	394	395	396	397-8	399-400	401	402	403	404	405	406	407	408	409	410
Operating time (hrs)	4.5	7	7	7	7	13	12	14	14	14	14	14	13	7	7	7	7
Leach Solution																	
pH	1.75	1.75	1.75	1.85	1.85	1.85	1.65	1.50	1.70	1.65	1.80	1.80	1.75	1.75	1.75	1.75	1.75
U_3O_8 (g/l)	2.70	2.56	2.92	3.25	2.89	2.98	3.13	3.33	3.34	4.60	5.70	5.93	6.25	6.25	6.25	6.25	6.25
Solvent																	
Amine	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	TIOA	Alamine	Alamine	Alamine	Alamine	Alamine
Normality	0.108	0.109	-	0.108	0.107	0.107	-	-	0.114	0.105	0.109	0.109	0.124	0.123	0.123	0.124	0.123
Strip Solution																	
Na_2SO_4 (g/l)	145	145	135	130	130	130	125	130	130	130	130	128	128	133	133	142	142
Flowrates (ml/min)																	
Feed	275	270	335	280	280	275	300	300	275	287	188	187	190	190	195	188	195
Solvent	145	145	140	155	162	155	155	155	150	160	168	177	171	174	175	177	178
Strip *	90	53	53	57	56	53	48	48	51	54	53	52	64	55	57	56	58
Acid Scrub	30	30	30	32	30	30	25	25	25	26	26	24	27	26	26	28	26
U_3O_8 assays (g/l)																	
Aqueous, Stage 1	-	1.14	2.70	1.37	1.08	1.76	2.13	2.93	3.35	2.78	5.65	6.35	2.28	2.28	1.87	2.08	2.05
" " 2	-	0.060	2.00	0.084	0.061	0.17	0.23	1.78	3.10	0.22	4.18	4.43	0.18	0.20	0.22	0.33	0.064
" " 3	-	0.002	1.32	0.003	0.004	0.006	0.011	0.21	2.39	0.006	0.40	0.60	0.002	0.015	0.006	0.019	0.024
Barren " 4	0.003	0.0009	0.053	0.001	0.001	0.002	0.002	0.032	0.087	0.008	0.002	0.002	0.001	0.002	0.0006	0.003	0.002
Recycle solvent	0.025	0.024	0.033	0.037	0.037	0.034	0.022	0.023	0.036	0.027	0.044	0.029	0.017	0.039	0.021	0.041	0.044
Loaded extract	5.34	5.17	5.94	5.33	5.68	5.49	5.82	5.88	6.11	5.86	6.30	6.16	6.83	6.92	6.72	6.81	6.97
Strip	8.81	14.31	15.60	16.26	15.55	15.87	18.33	18.35	22.46	16.90	19.45	21.65	16.40	19.74	20.29	20.29	21.49
U_3O_8 in strip solution** (total g)	207	318	351	382	370	667	665	738	808	760	867	942	820	454	477	467	520
Precipitation																	
Strip volume (l)	23.5	22.2	22.5	23.5	23.8	42.0	36.3	40.2	36.0	45.0	44.6	43.5	50.0	23.0	23.5	23.0	24.2
Precipitate																	
Wet (g)	-	750	1000	-	1167	-	2182	-	2247	2453	3249	2757	-	1500	1560	1420	-
Dry (g)	-	365	400	408	401	795	797	903	886	874	951	1017	-	520	550	575	560
Reagent Consumption																	
NaOH: Stripping (g)	130	253	260	280	270	570	488	560	600	556	680	656	576	323	334	335	365
Precipitation (g)	54	92	94	107	99	199	192	208	199	206	231	245	262	124	117	138	150
Total (g)	184	345	354	387	369	769	680	768	799	762	911	901	838	447	451	473	515
NaOH: (lb/lb U_3O_8)	0.89	1.09	1.02	1.01	1.00	1.14	1.02	1.03	0.99	1.00	1.05	0.96	1.02	0.98	0.95	1.01	0.99

* Total Flow: Na_2SO_4 + NaOH solutions

** Based on strip solution assay

TABLE 4(Continued)

OPERATING DATA

Run No.	411	412	413	414	415	416	417	418
Operating time (hrs)	7	7	7	7	5	6	5	6
Leach Solution								
pH	1.50	1.50	1.50	1.50	1.50	1.55	1.55	1.55
U_3O_8 (g/l)	6.25	6.25	6.25	6.25	6.25	3.34	3.34	3.34
Solvent								
Amine	Alamine	Alamine	Alamine	Alamine	Alamine	Alamine	Alamine	Alamine
Normality	0.125	0.123	0.123	0.125	0.124	0.124	0.125	0.124
Strip Solution								
Na_2SO_4 (g/l)	145	150	153	158	158	154	157	158
Flowrates (ml/min)								
Feed	192	200	198	198	205	264	265	326
Solvent	169	176	178	177	176	170	167	176
Strip *	60	55	48	40	60	58	77	54
Acid scrub	31	28	27	24	26	27	33	29
U_3O_8 assays (g/l)								
Aqueous stage 1	2.74	1.64	1.19	1.24	2.11	0.23	0.24	0.42
" " 2	0.24	0.15	0.042	0.057	0.087	0.013	0.011	0.015
" " 3	0.008	0.004	0.004	0.0009	0.001	0.0009	0.005	0.0007
Barren " 4	0.002	0.001	0.0004	0.0007	0.001	0.0008	0.0009	0.0006
Recycle solvent	0.023	0.023	0.130	0.031	0.076	0.030	0.056	0.045
Loaded extract	7.56	7.09	6.67	6.76	7.25	5.38	5.72	6.12
Strip	19.81	20.98	23.60	27.77	19.16	13.32	11.98	17.88
U_3O_8 in strip solution ** (total g)	495	483	472	472	525	280	276	347
Precipitation								
Strip volume (1)	25.0	23.0	20.0	17.0	18.0	21.0	23.0	19.4
Precipitate								
Wet (g)	-	1660	1810	2049	1505	1159	1265	1404
Dry (g)	580	600	644	-	495	442	405	464
Reagent Consumption								
NaOH: Stripping (g)	389	406	410	395	325	368	316	380
Precipitation (g)	166	144	133	139	84	65	62	73
Total (g)	555	550	543	534	409	433	378	453
NaOH: (lb/lb U_3O_8)	1.12	1.15	1.15	1.13	1.18	1.55	1.37	1.31

* Total Flow: Na_2SO_4+NaOH solutions

** Based on strip solution assay

up at the interface in the extractors during runs 406 to 410. At the end of run 410 all precipitated material was drawn from the extractors, centrifuged, filtered, and dried at 100°C. There were 45 grams of material recovered which included a small quantity of slimes carried into the circuit by the leach solution. Table 5 presents the results of chemical analysis of this material. The assays requested were chosen on the results of a preliminary spectrographic analysis.

With the leach solution pH reduced to 1.50 - 1.55 the precipitate no longer formed in the extractor unit.

TABLE 5

Extractor Sludge (Runs 406-410)

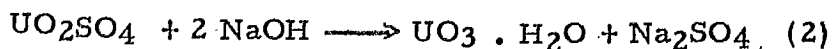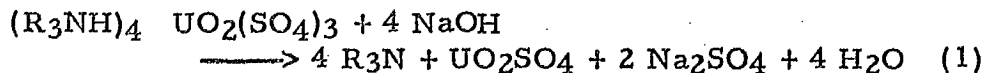| Assay	Percent
Loss on Ignition	21.9
P_2O_5	20.7
Fe	19.5
SO_4	15.8
SiO_2	6.5
Heavy Metals	4.21
Ti	2.55
CaO + MgO	2.40
Al	2.34
As	1.50
U_3O_8	0.55

Stripping

Careful control of the pH in the three stripping stages is required to ensure satisfactory sulphate stripping. If the pH falls much below 4.5, inefficient stripping results. If the pH increases to above 5.5, then precipitation of uranium in the cells may occur. Furthermore, a pH higher than 5.5 will cause emulsification and subsequent co-current movement of aqueous and organic. The addition of dilute NaOH (1 to 5%) to stripping cells 2 and 3 enabled efficient manual control of the pH in these cells.

Control of Na_2SO_4 Concentration in the Stripping Solution

In the operation of the solvent extraction circuit, when using a sodium sulphate strip, it is desireable to set conditions so that the Na_2SO_4 concentration of the recycled strip solution remains fairly constant. This is done by adjusting, where possible, those operating conditions which tend to change the sulphate strength of the stripping medium.

The sulphate, present in the stripping solution prior to precipitation, is recycled as sodium sulphate after uranium precipitation. (See chemical reactions 1 and 2). This tends to increase the sulphate concentration of the recycle and is a condition which cannot be adjusted without affecting the efficiency of the stripping circuit.

$(R_3NH)_4\ UO_2(SO_4)_3 + 4\ NaOH$
$$\longrightarrow 4\ R_3N + UO_2SO_4 + 2\ Na_2SO_4 + 4\ H_2O \quad (1)$$

$$UO_2SO_4 + 2\ NaOH \longrightarrow UO_3 . H_2O + Na_2SO_4 \quad (2)$$

The sodium sulphate concentration of the recycled strip 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 volume, due to the chemical reaction (1) above; (c) dilution, amounting to about 1% of the strip, due to the water contained in the dilute solutions of Separan and glue used as a flocculant 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.

Table 6 indicates the changes that were made in the strength of the NaOH solutions used in pH control and precipitation as the work progressed. Average values for the recycled strip solution concentration are also shown. Initial Na_2SO_4 concentration was 15% in the strip solution. This strength can be maintained without make-up.

Because of dilution, the quantity of Na_2SO_4 solution formed was rather great. It was found convenient to allow complete settling of the precipitate and then decant the supernatant liquor for recycling to the strippers. The filtrate from slurry filtration was discarded (20% of total precipitation feed).

TABLE 6

Solution Concentrations
(g/l)

Run Nos.	NaOH to			Na_2SO_4 Recycle Strip
	No.1 Stripper	No.2 and 3 Stripper	Precipitation	
392-3	100	20	100	145
394	"	10	"	135
395	"	10	200	130
396	"	10	"	130
397-8	150	10	"	130
399-406	"	15	"	128
407-10	"	50	"	136
411-15	"	"	"	153
416-18	"	"	"	158

As a typical example, the solution balance for run 415 can be shown as follows:

Na_2SO_4 solution drawn from the head tank for the run was
14.6 litres of 15.8% N_2SO_4.

Na_2SO_4 solution formed during the run amounted to
3.4 litres of 23.5% (calc.) Na_2SO_4.

Na_2SO_4 solution collected during the run amounted to
18.0 litres of 17.3% (calc.) Na_2SO_4.

Na_2SO_4 solution discarded after filtration amounted to
4.0 litres of the 17.3% solution.

The discard was 22% of the total volume. The remainder was returned to the head tank. A small increase in sulphate concentration is indicated.

Assays obtained on the initial and final strip solution (Table 7) show that there was no build-up of fluoride but there was an appreciable chloride increase. The seven-fold increase in chloride occurred in spite of the 20% (approx) bleed of precipitation filtrate. The Cl and F

ions did not build up in the recycling organic phase to any extent.

TABLE 7

Fluorine and Chlorine Assays on Recycled Solutions
(g/1)

| | Strip Solution | | Organic Recycle |
	Initial	Final	Final
F	0.02	0.004	0.05
Cl	0.20	1.48	0.029
Na_2SO_4	-	153	-

Solvent Losses

Solvent losses of tri-iso-octylamine and Alamine due to solubility in the aqueous phase were negligible. No drop in amine normality was recorded within the extent of this work (Table 4).

Losses of kerosene and amine which are due to entrainment in the aqueous are shown in Table 8. It is apparent that the increase in retention time of the aqueous feed in the settler from 5.5 to 8.5 minutes was sufficient to reduce several fold the solvent entrainment losses.

The assay method for kerosene involves a carbon tetrachloride extraction of the kerosene from the sample, followed by infra-red analysis of the extract. The assay method for amine involves a Kjeldahl digestion of the sample, followed by a colorimetric finish for NH_3.

TABLE 8

Data: Composite Barren Raffinates

Run No.	392-402	403-405	406-410	411-418
Assay Reference No.	1771	2260	2261	3415
Cl (g/1)	0.45	0.35	0.42	0.35
SO_4 (g/1)	21.0	26.0	22.7	27.0
Volume throughput (1)	1474	556	471	684
Av. flow rate (ml/min)	288	221	191	231
Av. Retention Time in Settler (min)	5 1/2	7 1/4	8 1/2	7
Amine in barren (ppm)	54	18	10	33
(lb/lb U_3O_8)	0.018	0.004	0.002	0.007
Kerosene in barren (ppm)	210	70	31	270
(lb/lb U_3O_8)	0.069	0.015	0.005	0.055
Extractant	TIOA	TIOA	Alamine	Alamine

NaOH Consumption

Average values for the reagent consumption are shown in Table 9. The caustic consumption in runs 416 to 418 was rather high. As observed from Table 4 data, the uranium loadings on the Alamine for runs 416 to 418 were well below saturation. Therefore NaOH was consumed in the stripping unit to remove an appreciable amount of SO_4 ion from the amine.

TABLE 9

Average NaOH Consumption
(lb/lb U_3O_8)

Runs	Amine	NaOH Consumption		Uranium Loading as Approximate % of Saturation
		Total	Percent for Precipitation	
393-402 404-405	TIOA	1.02	26	100
406-410 411-415	Alamine	1.07	27	100
416-418	"	1.41	16	80

Grade of Product

Table 10 presents a record of the precipitate analyses on three composite products. All specification limits are met and each of the composite precipitates is entirely satisfactory.

TABLE 10

Composite Precipitate Assays
(%)

Run Nos.	393-402	403-405	406-418
Assay Lab. No.	1601	3386	2679
U_3O_8	85.33	83.83	80.74
Halogens	0.026	0.017	0.077
SO_4	3.06	3.18	6.8
$(RE)_2O_3$*	<0.005	-	<0.003
ThO_2	0.002	-	<0.001
V_2O_5	0.011	-	<0.001
CO_2(evol)	0.12	-	0.10
F	0.0015	0.014	0.01
Mo	0.0046	-	0.0027
P_2O_5	0.007	-	<0.01
Cu	0.002	-	-
B	<0.001	-	<0.001
CaO	<0.01	-	-
As	0.01	-	-
Fe	-	<0.1	0.058
Ti	-	<0.05	0.06
Moisture	1.4	2.0	1.8

*$(RE)_2O_3$ - rare earths oxides

REFERENCES

1. H.H. McCreedy and W.A. Gow,"Pilot Plant Leach Tests on Ore from the Kitts Property of British Newfoundland Exploration Limited, Newfoundland,"Mines Branch Investigation Report IR 58-139, Department of Mines and Technical Surveys, Ottawa. August 8, 1958. (Industrial Confidential).

2. A.J. Gilmore and W.A. Gow, "Ion Exchange Treatment of Solution Produced in Acid Leaching of Uranium Ore from the Kitts Property of British Newfoundland Exploration Limited, Newfoundland,"Mines Branch Investigation Report IR 58-163, Department of Mines and Technical Surveys, Ottawa. August 28, 1958. (Industrial Confidential).

3. V.F. Harrison and W.A. Gow, "Direct Precipitation Tests on Solution Produced in Acid Leaching of Uranium Ore from the Kitts Property of British Newfoundland Exploration Limited, Newfoundland," Mines Branch Investigation Report IR 58-177, Department of Mines and Technical Surveys, Ottawa. October 21, 1958. (Industrial Confidential).

VMMc/RS/WAG/dm

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% HCl. Shake well and allow layers to separate.

3. Discard bottom (HCl) layer.

4. Filter top layer through dry paper to remove entrained HCl. 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 NaOH to pH 7.0.

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

APPENDIX 2

EXTRACTION EQUILIBRIUM DATA FOR VARIOUS AMINES

Batch shake-out tests were done to establish the uranium

distribution between Brinex leach solution and four amines

considered as possible extractants. The data can be used for a

preliminary evaluation of these amines and comparison of their uranium

capacities and distribution coefficients.

The tests were done on solution at pH 1.5 assaying as follows $(g/1)$

U_3O_8	Fe^{+3}	Fe^{+2}	SO_4	Cl	F	ThO_2
4.59	<0.1	5.0	23.4	0.35	0.11	0.007

A 500-ml volume of 5% v/v amine in kerosene (plus 2% n-decyl

alcohol where shown) was scrubbed with 5% sulphuric acid and then

contacted for two to three minutes with four successive volumes of

250-ml of leach solution. The four raffinates and the final extract

were assayed for U_3O_8 and results are given in Table 11.

TABLE 11

Batch Shake-out Tests

Amine Used (5%v/v in kerosene)	Stage No.	U_3O_8 in Raffinate (g/1)	U_3O_8 in Extract (g/1)	$E°a$
Tri-iso-octyl amine +2% n-decyl alcohol from Carbide and Carbon Chemicals Co.	1 2 3 4	0.005 0.035 0.820 3.15	2.25 4.30 6.06 6.66	450 125 7 2
Tri-fatty amine 7K-206 from General Mills	1 2 3 4	0.007 0.030 1.610 -	2.25 4.57 6.07 -	320 150 3.7 -
LA-1 (9D-178) Secondary amine from Rohm and Haas Limited	1 2 3 4	0.009 0.250 3.16 4.28	2.25 4.45 5.17 5.32	250 18 1.7 1.2
LA-2 Secondary amine from Rohm and Haas Limited	1 2 3 4	0.026 0.180 1.37 3.38	2.28 4.48 6.07 6.68	90 25 4.4 2.0

Equilibrium curves were plotted from the above data as shown in

Figure 2. The equilibria for TIOA and tri-fatty amine 7K-206 are

comparable as confirmed by the continuous extraction runs. The Rohm

and Haas amine LA-1 is equally efficient in the low loading zone but

saturation values are somewhat less. The LA-2 equilibrium is

depressed in the low concentrations but shows good uranium capacity.

Also, some stripping tests at pH 4.75 indicated that LA-2 will not

strip as readily. On this basis the first three amines would be equally

suitable and final choice would depend on commercial availability,

soluble loss and price. These factors have been established in the
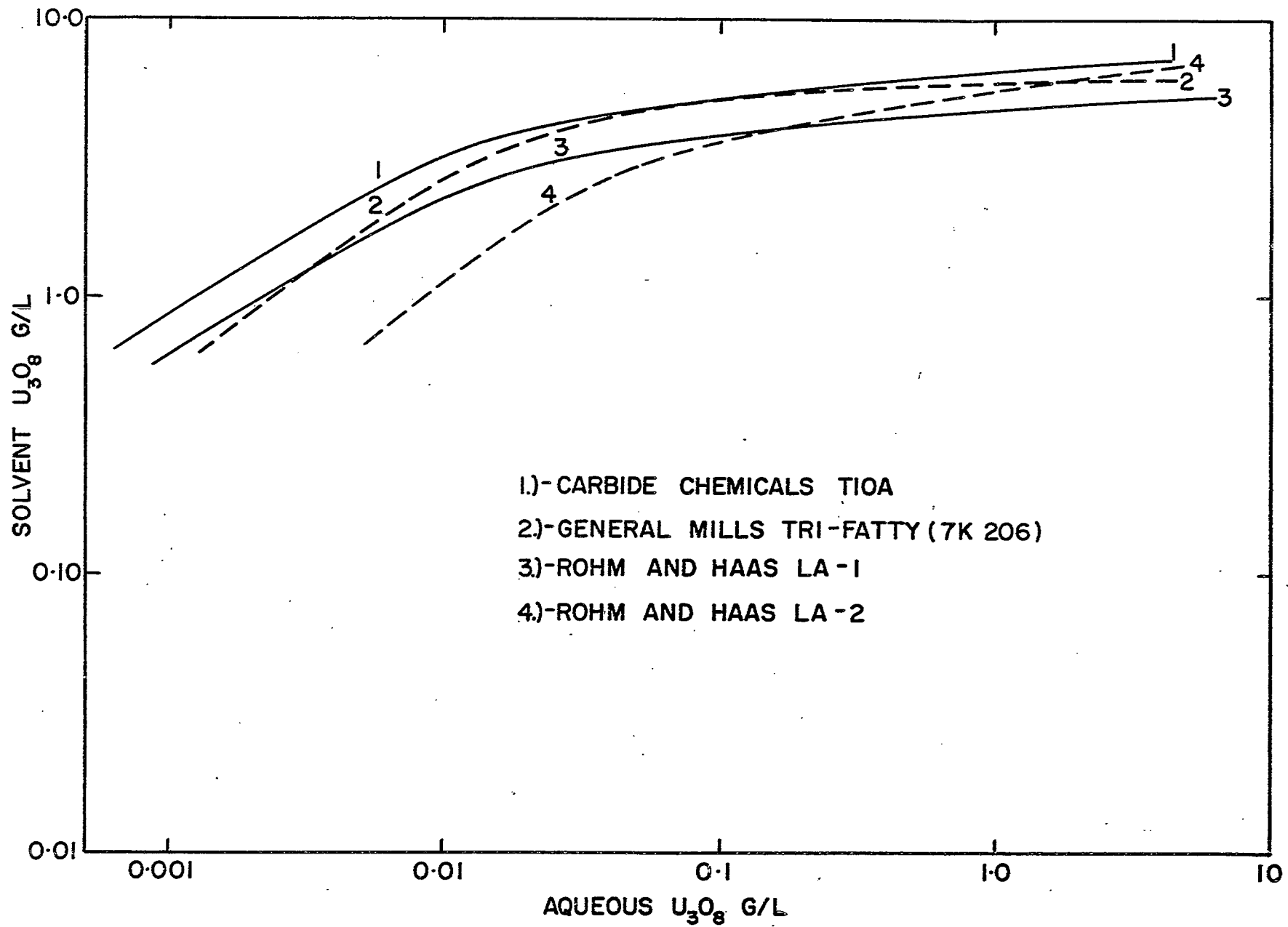
present report for TIOA and tri-fatty amine 7K-206.

FIGURE 2

EQUILIBRIUM DATA

BRINEX LEACH SOLUTION VS 5% AMINE IN KEROSENE