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SOLVENT EXTRACTION FOR THE RECOVERY OF URANIUM AND THORIUM FROM LEACH SOLUTIONS OF A URANIUM ORE (Ref. 7/57-7) FROM THE BANCROFT AREA, ONTARIO

by

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RADIOACTIVITY DIVISION

MINES BRANCH
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R. Simard*

SUMMARY OF RESULTS

Leach solutions obtained from the sulphuric acid leaching of flotation concentrates of an ore from the Bancroft area were treated successfully by amine solvent extraction to recover high grade uranium and thorium products. Recoveries of 99% for uranium and 95% to 99% for thorium were possible at a reagent cost of 12 cents per pound U_3O_8 and 8 to 10 cents per pound ThO_2.

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CONTENTS

(5 tables, 4 illus.)

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INTRODUCTION

The work was done in conjunction with pilot plant flotation and leaching of a 20-ton bulk sample of uranium ore (our Ref. No. 7/57-7) from the Bancroft area of Ontario, received at the Mines Branch laboratories, Ottawa, in August, 1957.

The ore was first concentrated by flotation in a 400 lb/hr circuit. The flotation concentrates plus slimes were leached in batches at a controlled pH of 1.5 to 1.8 with sulphuric acid and sodium chlorate. The leach solutions were then accumulated for ion exchange and solvent extraction studies. The flotation and leaching results appear in Radioactivity Division Special Reports SR-480/57(1) and SR-483/57(2) respectively. Recovery of uranium by anion exchange resin (IRA-400) was also studied and the results of this work will be reported at a later date.

This report covers the work done on both uranium and thorium recovery from these solutions by continuous solvent extraction.

GENERAL SUMMARY

1. Uranium Recovery

Some 1250 litres of leach solution was collected from the leaching of ore sample Ref. No. 7/57-7 (flotation concentrate plus slimes). This solution, assaying 2.3 to 2.7 gm U_3O_8/1 and 1.5 to 1.8 gm ThO_2/1, was put through a uranium solvent extraction circuit containing 5% tri-isooctyl amine and 2% n-decyl alcohol in kerosene. From the 4 counter-current mixer-settlers, final barren raffinates of 0.005 gm U_3O_8/1 or less were obtained. The loaded extract, assaying 5.5 to 5.8 gm U_3O_8/1, was stripped in a 3-stage counter-current mixer-settler with either 10% or 20% Na_2SO_4 solution to yield a high-grade strip of 11 to 19 gm U_3O_8/1. However, in the course of the operation it was soon realized that an accumulation in the strippers of some polymerized fatty acids, carried over from the flotation to the leach circuit and extracted by the 5% amine solvent, would cause serious mechanical difficulties (mainly coating and consequent sluggishness of the pH electrodes used for control, and eventual blockage of mixer-settler connections). For this reason, after five runs with sodium sulphate stripping, this was replaced by a 10% sodium carbonate strip which kept the gum from forming and carried it out as a soluble or colloidal sodium soap. The fatty acid responsible is believed to be mainly linolenic acid, present originally in the flotation reagent (Armour Neo Fat 42-12).

The high grade sulphate strip collected from each run was clarified and precipitated with NaOH to pH 7.0-7.2 and the barren filtrate was then recycled to the stripping section. A composite precipitate assayed 81% U_3O_8 and 0.03% ThO_2. Other impurities also met impurity specification limits. NaOH consumed for stripping and precipitation was 1.05-1.10 lb per lb U_3O_8.

The sodium carbonate strip, assaying 25-30 gm U_3O_8/1, was acidified with sulphuric acid and the uranium precipitated with MgO or NaOH. Due to the build-up of the fatty acid soap in the carbonate strip, it would not be advisable to precipitate the uranium directly with NaOH and recycle the carbonate to stripping. For this reason the acidification procedure was adopted. In this case, reagent consumption would be 3.3 lb Na_2CO_3, 2.1 lb H_2SO_4 and 0.36 lb NaOH per lb U_3O_8. If magnesia is used to replace caustic, MgO consumed will be 0.2 lb per lb U_3O_8, approximately.

2. Thorium Recovery

Some 600 litres of barren raffinate collected from the uranium extraction circuit was treated similarly in a 4-stage mixer-settler with 5% 9D-178 Rohm and Haas amine and subsequently with 5% di-tridecyl amine from Canadian Carbide Chemicals. No alcohol was used as an additive. From a feed of 1.50 to 1.8 gm ThO_2/1, good recoveries of 90 to 99%, depending on the loading, were obtained. For high recoveries (e.g. 99%), operating loadings of 3 to 3.5 gm ThO_2/1 will be possible with 9D-178, and 3.5 to 4.0 gm ThO_2/1 with di-tridecyl amine. The loaded extract was stripped co-currently with 10% Na_2CO_3 to produce a high grade strip of 15 to 20 gm ThO_2/1. After clarification to remove the insoluble impurities (mainly ferric hydroxide), thorium was precipitated by adding solid NaOH to pH 12-12.2. The barren carbonate was recycled to stripping. NaOH consumption was 1.80 lb when using 9D-178 amine as extractant, and 1.5 lb per lb ThO_2 when using di-tridecyl amine. The thorium product, assaying 70% ThO_2, can be upgraded to 97-98% by redissolving the wet cake in HCl and precipitating with oxalic acid. The main impurities in the calcined oxalate were 0.1% Ti and 0.06% Zr.

DESCRIPTION OF CIRCUITS AND OPERATING PROCEDURE

Both the uranium and thorium extractors are identical 4-stage countercurrent mixer-settlers and are described in detail in Radioactivity Division reports SR-416/56 and SR-468/57.(3, 4)

<u>1. The uranium circuit,</u> as shown in Figure 1, comprises the 4-stage extractor in series with a 3-stage stripper. An acid scrubber to sulphate the solvent before contacting the leach solution consists of a 3"D x 18" column with countercurrent flow of 5% H_2SO_4 and solvent. A rotating disk at or near the interface acts as contactor. The spent acid is collected, made up to 5% v/v strength, and recycled to the head tank. The main purpose of the scrubber is to prevent high pH in the 4th stage, where the entering solvent would pick up free sulphate.

For sulphate stripping, the 10% or 20% Na_2SO_4 solution was fed countercurrently to the loaded solvent and maintained at a constant pH (4.5 to 5.0) by automatic addition of 10% NaOH in stage 1, and by manual control of 2% NaOH flowing to the mixers of stages 2 and 3.

For carbonate stripping, 10% Na_2CO_3 was metered to give a maximum uranium concentration, which is limited to 30-35 gm/1 at pH 7.5 to 8.0. Below this pH, uranium will precipitate out and cause emulsion problems.

The stripped solvent is returned continuously to the extractor.

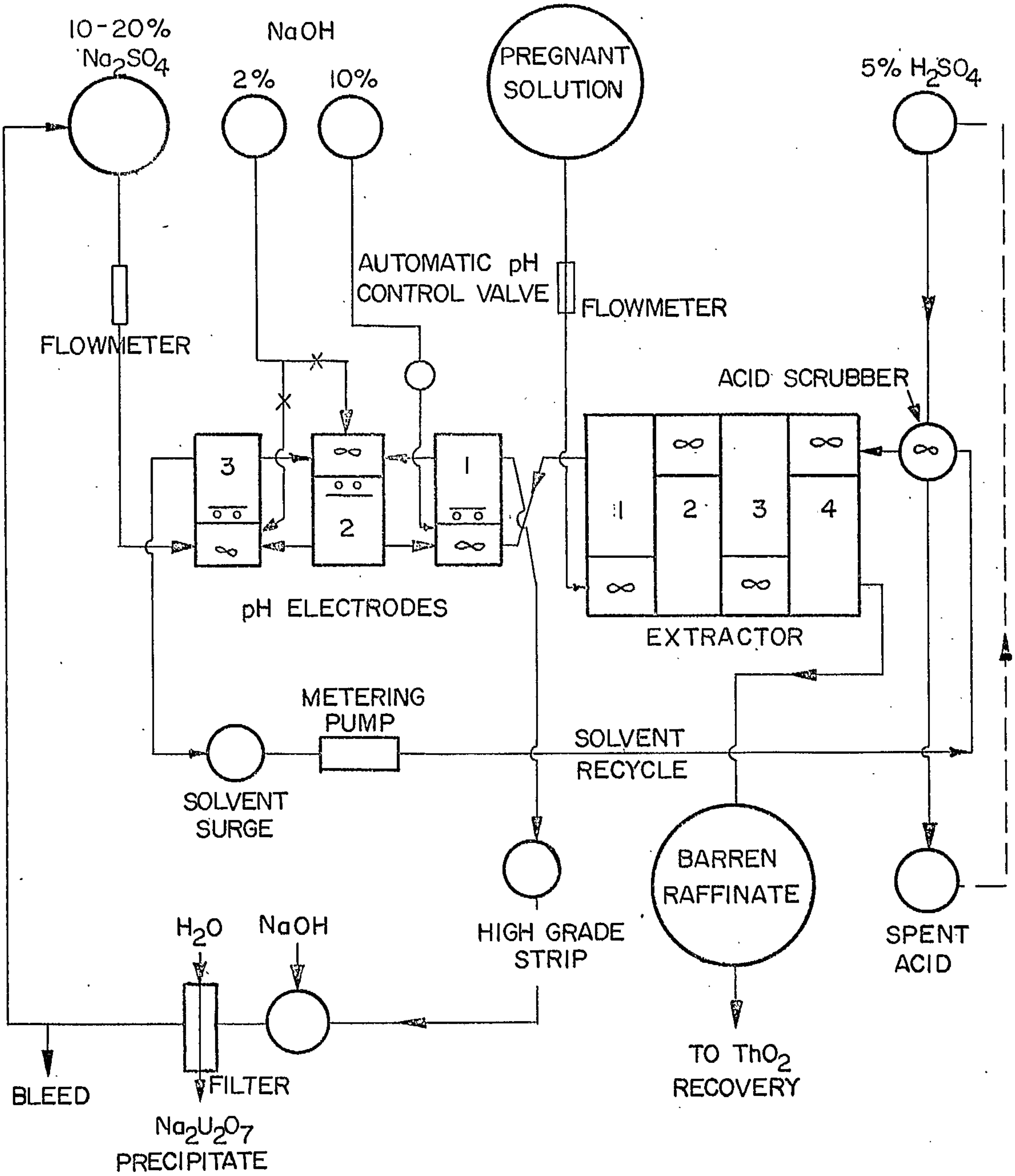

FIGURE 1

URANIUM SOLVENT EXTRACTION FLOWSHEET
(WITH Na_2SO_4 STRIPPING)

through a surge tank and an adjustable metering pump. The circuit holds a total of 15 to 18 litres of solvent, and at the flowrates used in the present tests (200-400 ml solvent/min) one complete cycle would require 50 to 100 minutes. Contact times in the various stages of extraction and stripping are as follows:

Extraction	Aqueous Mixing Time	=1-1.5 min/stage
	Organic Mixing Time	= 3 min/stage
Stripping	Aqueous Mixing Time	= 2 min/stage
	Organic Mixing Time	= 2-3 min/stage

Settling area provided per stage of extraction was 2 to 3 sq ft per gpm, but previous experience has shown that 1 to 1.2 sq. ft per gpm was the lower limit for a similar system where the organic phase is continuous. This is achieved by keeping the organic to aqueous ratio in the mixer at 2 to 1 or better. The phase ratio can be controlled in the 4 stages by adjusting the raffinate leg at the 4th stage to give the required interface level at 2 organic to 1 aqueous.

The high-grade strip collected from each run was precipitated in one batch. From the sulphate strip, uranium was precipitated by adding 10% or 20% NaOH to pH 7.0-7.2. The slurry can then be settled with a combination of separan and glue to give a filterable product. Two water washes were used to give a total of 4 displacements. The filtrate was recycled to the strip head tank. To maintain the sulphate strength, the concentration of NaOH used for precipitation can be adjusted accordingly.

The carbonate high-grade strip was precipitated by first acidifying to pH 2 to 2.5, clarifying, and neutralizing to pH 7-7.2 with either MgO or NaOH.

2. The thorium circuit, as shown in Figure 2, uses a similar extractor but the stripping was simplified to a two-stage co-current arrangement. Three amine extractants were considered for study: (1) Armeen 2-12, a secondary laurylamine from Armour;(2) 9D-178, a secondary dodecenyl amine from Rohm and Haas;and (3) DTDA (ditridecyl amine) from Canadian Carbide Chemicals. Batch extraction tests on these three solvents made up to 0.1M in kerosene were first carried out. The continuous runs were done on two of these: 9D-178 and ditridecyl.

The strippers consist of two 1-litre beakers with vertical baffles and 6-blade 1 1/2"D turbines. The final settler is a 2-litre lucite box.

Since the maximum thorium concentration possible in the strip can not exceed 20 gm ThO_2/1 due to the tendency for thorium to hydrolise below pH 8.5, direct acidification of the clarified strip would consume 5 lb or more of Na_2CO_3 per lb ThO_2. Although this procedure was investigated, the alternative procedure was considered more practical, that is, to conserve the carbonate by precipitating with solid NaOH and recycling the barren strip. To control Na_2SO_4 build-up below the saturation point in 10% Na_2CO_3 (140 gm/1 at 20° C), excess sulphate is removed by cooling to 10° C, at or near the saturation point for 10% Na_2CO_3. The Na_2SO_4 concentration increases at a rate of 30 to 40 gm/1 per cycle and would reach saturation in less than 5 cycles. An alternative to the cooling step would be to bleed 20 to 25% of the barren strip and make up with water at an expense of 1.2 to 1.5 lb Na_2CO_3 per lb ThO_2.

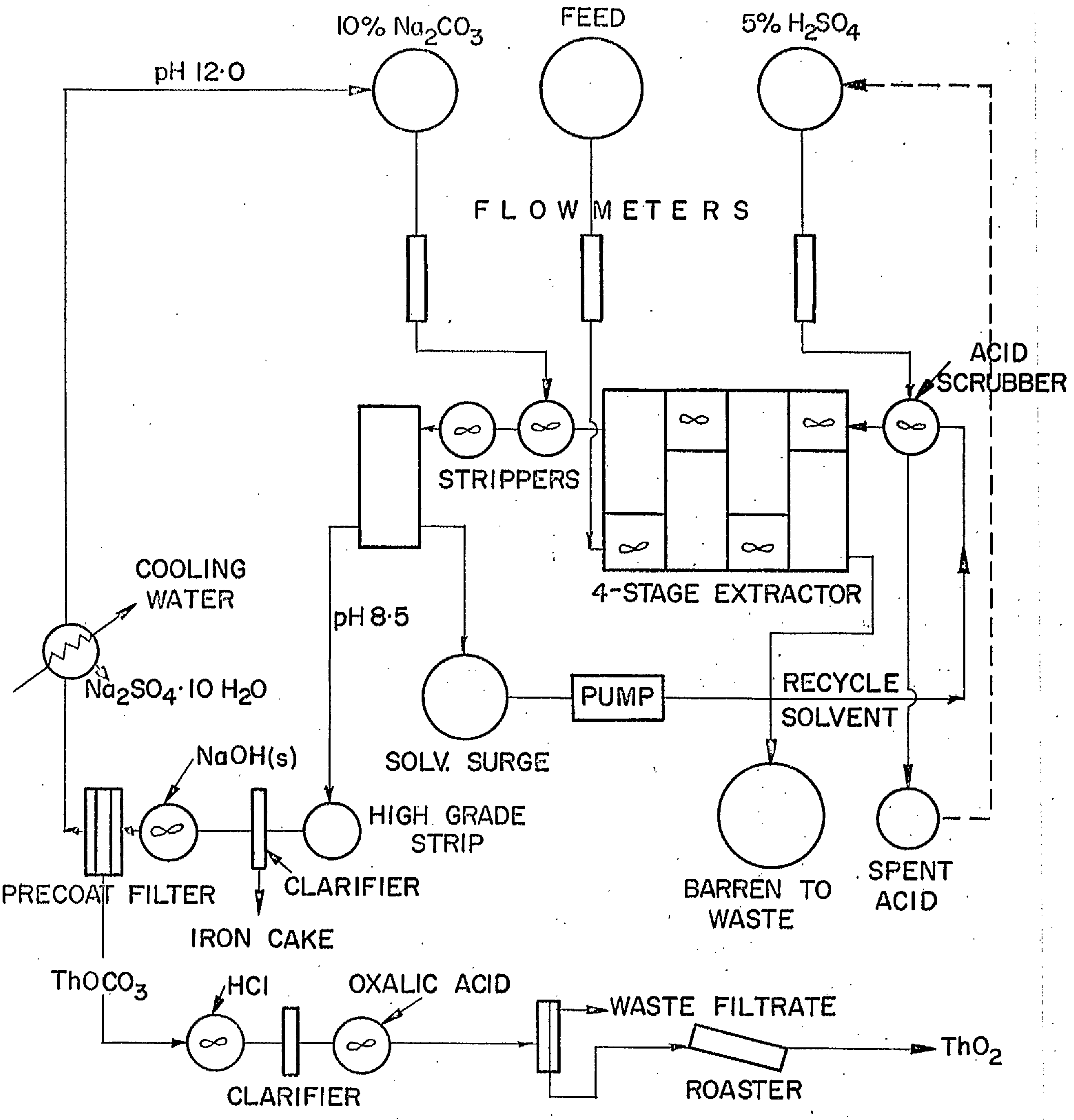

FIGURE 2

THORIUM SOLVENT EXTRACTION FLOWSHEET

The thorium oxycarbonate product obtained from the clarified strip was dried and assayed. To establish maximum purity possible by the oxalic precipitation step, the wet precipitate from two runs was repulped in water and acidified with HCl to pH 1.1-1.2. The insolubles, mainly filter-aid from the filter, were discarded and the solution was precipitated with 10% oxalic acid solution using 5% excess over stoichiometric. The thorium oxalate was dried and calcined at 750°C for 1 hour.

ANALYSES OF LEACH SOLUTIONS

Leach solution assays on the two lots of stored solution are given in Table 1 A.

Table 1 A

Analyses of Leach Solutions

	Assay Lab. R.D.	pH	U_3O_8	ThO_2	Fe^{+3} (gm/l)	Fe^{+2}	RE_2O_3	SO_4
1st lot (7-342-45)	2120	2.1	2.71	1.41	1.25	0.2	1.10	17.7
2nd lot (7-346-50)	2295	1.8	2.39	1.57	2.1	0.8	1.34	24.0

Table 1 B

Semi-quantitative Spectrographic Analysis of Total Solids (34 gm/l) from 1st Lot

Element	%	Element	%
Fe	6	Y	0.4
Th	8	Mn	0.5
U	3	Cu	0.06
Al	2.5	Ti	0.03
Mg	2	B	0.01
Ca	1.5	Pb	0.08
Si	2	Zr	0.02
Ce	2.5	Be	0.002
La	1.0		
Yb	0.1		
Gd	0.05		
Dy	0.05		

RESULTS AND DISCUSSION

1. Uranium Recovery

1.1 Extraction and Stripping

A total of 1250 litres leach solution was treated in a series of 10 continuous runs. Operating data are given in Table 2. In the first five runs, where sodium sulphate was used for stripping, it was necessary to cut down the throughput of feed solution and operate at a lower loading in the extract, due to high recycle solvent values (0.3 gm U_3O_8/l). This was mainly due to poor pH control in the strippers, where a viscous gum was constantly building up around the electrodes and at the interface. As shown in Figure 3, the theoretical equilibrium for 5% TiOA vs 10% Na_2SO_4 at pH 4.5 was never attained. Since it was found that an alkaline strip would not present the same difficulties, the last part of the operation was carried out with 10% Na_2CO_3. It was then possible to boost the throughput and load to 5 gm/l or better. The strip loading was maintained near 30 gm U_3O_8/l without difficulty.

1.2 Precipitation

The collected high grade sulphate strip, when precipitated at pH 7-7.2 with gradual addition of 20% NaOH, gave a filterable product. The composite precipitate assays for runs 7-342 to 7-344 are listed in Table 3. The sodium carbonate strip was acidified to pH 2.5, when most of the soluble fatty acid floated to the surface and was filtered out. The clarified solution was then precipitated to pH 7.0 with NaOH or MgO. Product assays are also listed in Table 3.

Table 2

Recovery of U_3O_8 with 5% TIOA (+2% Decyl Alcohol)

Continuous Extraction and Stripping

Operating Data and Results

Run No.	7-342	7-343	7-344	7-345	7-346	7-347	7-348	7-349	7-350	
Conditions										
Stripping Solution										
Flowrates, ml/min										
Pregnant	300	300	200	200	200	220	275	385	400	
Solvent	150	150	120	105	100	110	110	155	175	
Strip	60	55	40	35	28	22	22	27	30	
Acid Scrub(5% H_2SO_4)	35	35	30	25	25	25	28	28	20	
Normality of TIOA, final	0.103	0.1017	0.1000	-	-	0.091	0.091	0.090	0.089	
Operating Time, hr	5	5	6	4 1/2	4 1/2	3	8	7	23	
Assays: (U_3O_8 gm/l)										
Barren Stage 1	no sple	2.65	2.12	no sple	no sple	no sple	1.64	2.20	2.20	
2	"	2.31	1.00	"	"	"	0.18	2.00	2.05	
3	"	1.09	0.046	"	"	"	0.006	0.33	0.52	
4	0.005	0.056	0.004	0.007	0.002	0.001	0.001	0.012	0.016	
Extract	5.77	5.80	5.1	5.0	4.69	4.90	5.13	5.49	5.70	
Recycle Solvent	0.49	0.30	0.32	0.46	0.36	0.001	0.001	0.0004	0.004	
Pregnant Strip	11.30	14.40	17.02	15.0	19.00	24.0	30.0	30.33	27.6	
Volume Strip, litre	18.7	16.5	13.3	9.7	7.5	4.0	10.0	11.0	41.0	
pH Strip	4.6	4.4	4.5	4.65	4.6	9.0	8.5	8.30		
Reagents Consumed, lb/lb U_3O_8									10 litres	10 litres
NaOH for pH control	0.95	0.76	0.69	0.84	0.80	-	-	-	-	-
" for precipitation	0.20	0.27	0.36	0.24	0.37	0.3	-	0.37	-	0.36
" Total	1.15	1.03	1.05	1.08	1.17	0.3	nil	0.37	nil	0.36
Na_2CO_3 for stripping	nil	nil	nil	nil	nil	3.30	3.30	3.30	3.60	3.60
H_2SO_4 for acidification	"	"	"	"	"	2.60	2.10	2.10	1.95	1.95
MgO for precipitation	"	"	"	"	"		0.18	nil	0.24	nil
Precipitate										
Dry wt, gm	270	250	236	148	168	117	305	400	309	300
U_3O_8 %			81.1			-	-	81.3	-	-

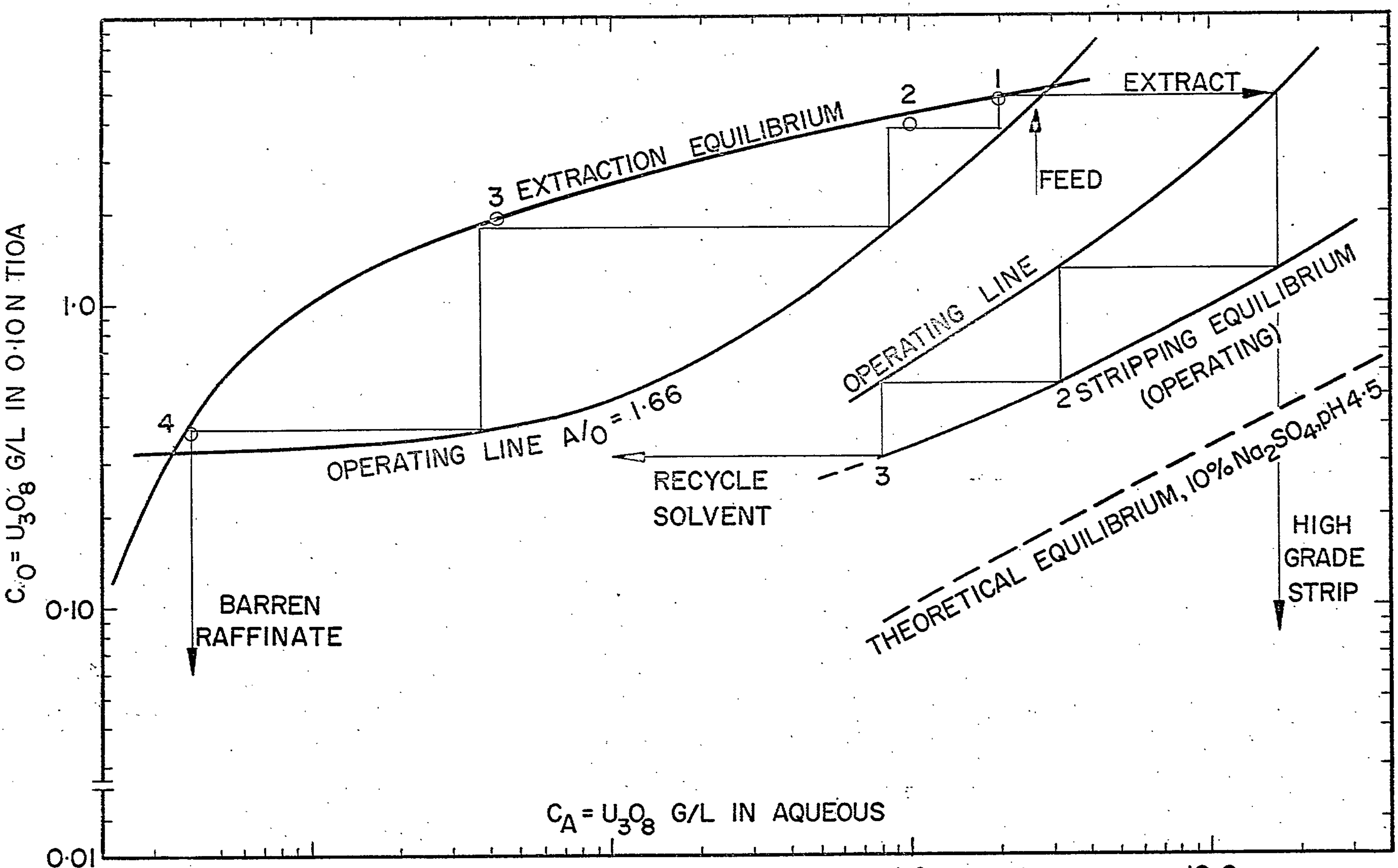

FIGURE 3

URANIUM OPERATING EQUILIBRIA

RUN 7-344

Table 3

Uranium Product Assays

	From Na_2SO_4 Strip with NaOH precipitation	From Na_2CO_3 Strip with acidification + NaOH
Run No.	7-342-343-344	7-349
Lab No.	RD-2445	RD-3381, RD-3933
U_3O_8	81.14 %	81.3 %
ThO_2	0.03	0.03
RE_2O_3	0.036	0.07
Fe	0.032	<0.01
Mo	0.001	0.002
TiO_2	<0.020	
V_2O_5	<0.010	<0.02
NH_3	0.007	0.004
CaO	<0.020	<0.01
Cu	<0.001	0.0002
SO_4	4.20	4.50
CO_2	0.36	0.140
Cl + Br + I	0.084	0.034
F	0.020	0.04
As + P_2O_5	<0.005	<0.005
B	0.001	0.001
H_2O	2.86	1.59

1.3 Reagent Consumption

With sulphate stripping, the only reagent consumed was 1.05 to 1.10 lb NaOH per lb U_3O_8. With carbonate stripping, the Na_2CO_3 consumed, on the basis of 30 gm U_3O_8/1 of strip, will be 3.3 lb/lb U_3O_8. It may be possible to boost the U_3O_8 content of the strip to 35 gm/1, thus lowering the carbonate consumption to 2.9 lb/lb U_3O_8. Other reagents will include sulphuric acid for acidification, and MgO or NaOH for precipitation, as shown in Table 2.

1.4 Solvent Losses

Steady state solubility loss of TIOA, based on the drop in normality through the last four runs (7-347 to 7-350) where 5% TIOA v/v is equivalent to 0.1N, will be as follows:

Normality difference = 0.0025 N
Equivalent % difference = 0.125%
Contained TIOA in circuit = 800 gm
Weight loss = $\frac{0.125 \times 800}{5.00}$ = 20 gm
Leach Solution treated = 900 litres
U_3O_8 recovered 900 x 2.4 = 2160 gm
Steady State Solubility Loss = 0.009 lb/lb U_3O_8

Entrainment losses cannot be determined too accurately on the present scale, but indicated losses were 0.2 gm per litre of raffinate or 0.08 lb as 5% TIOA in kerosene per lb U_3O_8 (or 0.004 lb TIOA per lb U_3O_8).

Overall solvent losses would therefore be a small part of the operating cost.

2. Thorium Recovery

2.1 Batch Extraction Tests

Three amines made up as 5% v/v in kerosene were tested as possible extractants. Equilibrium data for di-tridecyl amine, Armeen 2-12 and 9D-178 are given in Figure 4. The lower part of each curve is subject to the assay error for low grade samples. The extract assays were calculated values. The curves show possible loadings of 3 to 4 gm/l for a feed of 1.5 gm ThO_2/l.

2.2 Continuous Extraction and Stripping

The operating data and results from a series of 5 continuous runs are given in Table 4. The thorium content of the feed had been originally reported as 1.57 gm/l but calculated to be 1.75 to 1.85 gm/l. Since the A/O flow ratio in the first two runs with 9D-178 was set on the basis of the lower assay, extract loadings were therefore higher than expected, at the expense of recovery. Improved recovery will be noted with di-

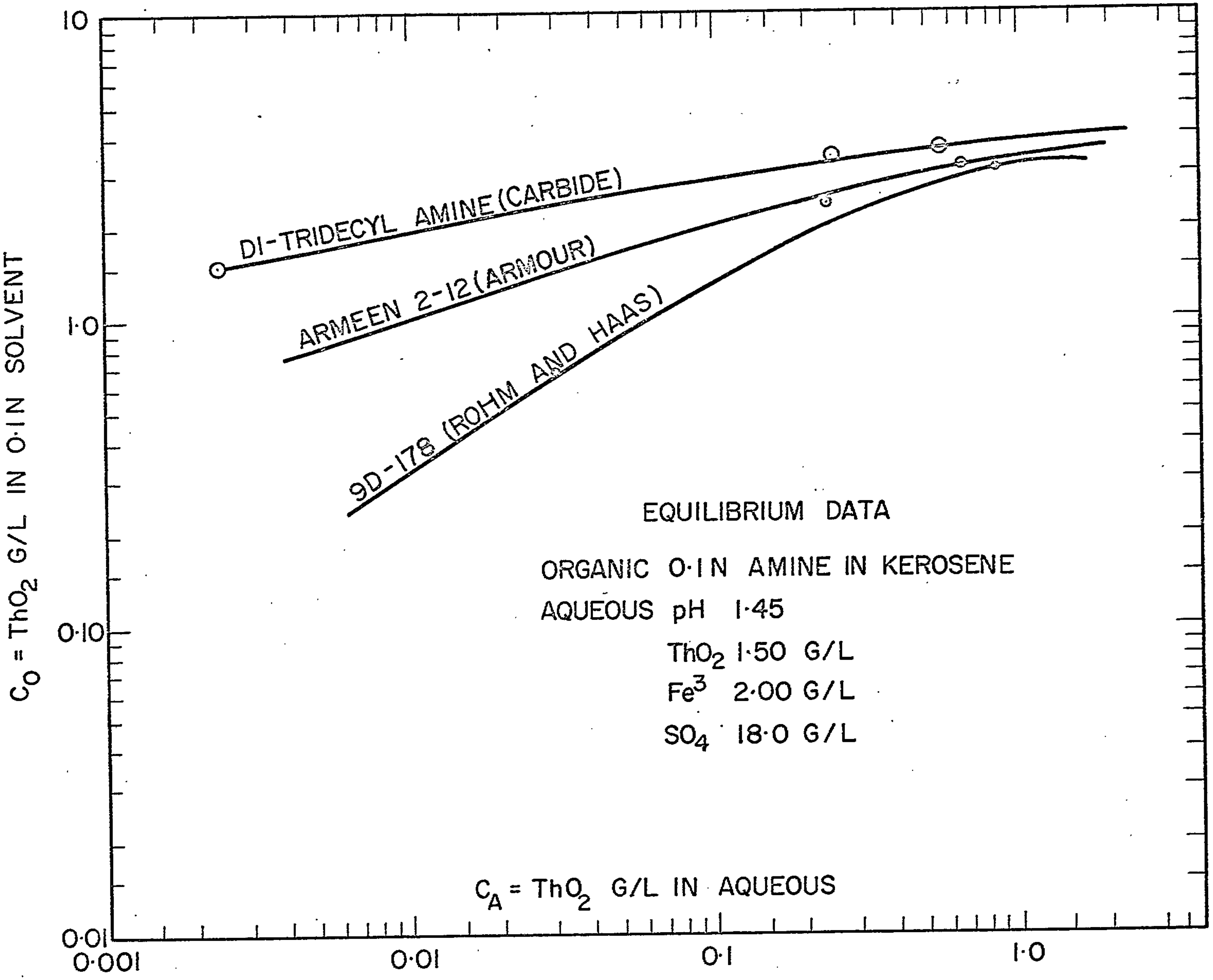

FIGURE 4

THORIUM OPERATING EQUILIBRIA

tridecyl amine at a loading of 4.0 gm ThO_2/l. Stripping efficiency was very good with 10% Na_2CO_3, and results indicate a maximum concentration of 19-20 gm ThO_2/l of strip.

Table 4

Recovery of ThO_2

Continuous Extraction and Stripping Operating Data and Results

Run No.	7-353	7-354	7-355	7-357	7-358
Solvent used	9D-178	9D-178	DTDA	DTDA	DTDA
Solvent normality, final	0.100	0.095	0.0965	0.097	0.0965
Flowrates, ml/min					
Pregnant Solution	425	375	400	420	425
Solvent	185	180	175	168	190
10% Na_2CO_3 Strip	45	35	40	35	38
5% H_2SO_4 Scrub	25	25	25	25	25
Operating Time, hr	5	7	2 1/3	6	6
Assays (ThO_2 gm/l)					
Barren Stage 1	1.73	1.69	no sample	1.89	1.84
2	1.44	1.26	"	1.58	0.98
3	0.70	0.70	"	0.81	0.43
4	0.16	0.14	0.01	0.17	0.02
Extract	3.90	3.60	4.0	4.25	4.1
Recycle Solvent	0.009	0.01	0.005	0.001	0.003
Pregnant Strip	14.49	18.8	14.0	18.0	19.6
Volume of Strip, litres	14.0	14.2	4.5	13.5	13.0
pH of Strip	9.2	9.15	9.5	9.5	8.6
Precipitation					
Iron cake, gm	7.5	12.3	-	2.0	2.5
NaOH added, gm/l	26	32.0	36.0	23	33
" ", lb/lb ThO_2	1.8	1.6	1.8	1.3	1.68
Ppt, dry wt, gm	238	Retained	100	375	Retained
", ThO_2 %	70.7	wet for	65	65	wet for
		retreatment	(calc)	(calc)	retreatment

The thorium-to-iron selectivity factor is much greater for DTDA, as shown by the weight of the iron cake, which dropped from 0.075 lb/lb ThO_2 in run 7-354 to 0.010 lb/lb ThO_2 in run 7-358.

2.3 Precipitation

Thorium was recovered quantitatively as $ThOCO_3$ by adding solid NaOH to a pH of 12.1-12.2. The product settled to 30% of the volume in 2 to 3 hours and filtered at a rate of 1 to 2 lb per hr per sq ft of filter. A filter aid was used as a precoat to prevent initial losses of fines, and also blinding, but did not speed up the overall rate. A water wash equivalent to 2-3 displacements was used.

The wet cake from runs 7-354 and 7-357, redissolved in HCl and precipitated with oxalic acid, produced a pure white oxalate which filtered very fast. Assays of these products are shown in Table 5. From the last run (7-358), a portion of the strip was acidified directly with H_2SO_4 to pH 1.0 and precipitated with oxalic acid. Assays, as shown in Table 5, indicate a high sodium sulphate content, which did not wash out through occlusion. This procedure does not seem promising and was not studied further.

Table 5

Thorium Product Assays

(In percent)

Solvent used	9D-178	9D-178	DTDA	DTDA
Product	$ThOCO_3$ as is from 7-353	ThO_2 from Retreatment of 7-354	ThO_2 from Retreatment of 7-357	ThO_2 from Acidification of 7-358
Assay Lab.No.	RD-2948	RD-3165	RD-3355	RD-3354
ThO_2	70.7	97.4	98.8	83.5
CO_2		-	-	-
Na	8.90	-		15.0 (s)
SO_4	1.50	-		-
Fe	0.20	<0.002		0.01 (s)
RE_2O_3		-		
TiO_2		0.10 (s)		
Zr		0.05 (s)		
Al		0.019		
Cu		<0.0005		
Ni		<0.0003		
Mn		<0.003		
U_3O_8	0.07	-		

(s) = semi-quantitative spectrographic analysis

2.4 Reagent Consumption

When caustic is used for precipitation the sodium carbonate is regenerated to its original 10% concentration. The only reagent consumption will be of NaOH, and, as shown in Table 4, it will vary, according to the amine used and the loading, from 1.3 to 1.8 lb per pound of ThO_2 recovered.

Retreatment will consume:

0.7-0.8 lb 100% HCl/lb ThO_2
0.9-1.0 lb $(COOH)_2\ 2\ H_2O$/lb ThO_2

2.5 Solvent Losses

Solubility losses for 9D-178 or DTDA cannot be estimated from the short duration of the test runs performed, but may be assumed to be of the order of 0.02 gm/1 or 0.01 lb/lb ThO_2 as found from previous work.

As for entrainment losses, DTDA showed better separation rates in both acid and alkaline circuits. Also, the smaller weight of iron cake produced with DTDA will mean less trapped solvent in the iron product, although this would represent less than 0.05 lb of 5% 9D-178 in kerosene per lb ThO_2.

GENERAL CONCLUSION

Uranium and thorium can be effectively recovered by amine solvent extraction of sulphuric acid leach liquor from the present flotation concentrate of ore Ref. No. 7/57-7 to produce acceptable high grade products. Reagent cost will be of the order of 12 to 14 cents per lb U_3O_8, when using tri-isooctyl amine and the sodium carbonate strip procedure. For thorium, overall reagent cost will not exceed 8 to 10 cents/lb ThO_2, exclusive of the retreatment step.

If the fatty acid flotation reagent can be well washed out prior to leaching, uranium could be recovered at a reduced reagent cost of 5 to 6 cents/lb U_3O_8.

This problem of fatty acid removal was studied by V. F. Harrison of this Division, and his results are included in the present report as an appendix (see pages 21-24).

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(An appendix follows)
(on pages 21-24.)

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APPENDIX

Tests on the Removal of Fatty Acid Coatings from Mineral Surfaces of the Flotation Concentrate Produced from Ore Sample No. 7/57-7

by

V. F. Harrison*

A report on the pilot plant flotation of the uranium ore sample, reference No. 7/57-7, is given in Radioactivity Division Special Report SR-480/57. Reagent consumptions for floating the uranium minerals were:

Flotation Reagent	Use	Consumption, lb/ton
Na_2SiO_3	Scrubbing	0.55
Neo-Fat 42-12	Promotor	4.55
Dowfroth 250	Frother	0.13

Neo-Fat 42-12 was added to the conditioner as a 10% emulsion containing one gram NaOH per litre. Neo-Fat, a fatty acid product from Armour and Company, has the following composition:

Oleic	45%
Linoleic	38%
Linolenic	3%
Rosin acids	14%

The mechanism of resin formation in the solvent extraction circuit is not fully explained but it was shown that the operation would benefit by a near complete removal of the resin. Two preliminary shake-out tests confirmed the fact that the fatty acid from the acid leach liquor was taken up by the amine solvent along with the uranium. Pilot plant liquor (see

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Radioactivity Division Report No. SR-483/57) was contacted with (1) kerosene containing tri-isoctylamine, and (2) kerosene alone. Both solvents were then tested for resin, as described below. The kerosene alone did not pick up any resin.

Washing tests were performed on 1000-gm samples of the flotation concentrate, to try to remove the residual organic reagents before leaching. These tests consisted of repulping the ore at 50% solids for 2 hours' contact time with (1) 25°C and 75°C temperature water; (2) individual 25°C water solutions of 1% detergent (Ethofat 242/25), 1% emulsifier (Ethofat 242/60), and 1% wetting agent (Ethomeen c/15);* (3) 25°C sulphuric acid solution of pH 3; and (4) 25°C 1% NaOH solution. The washed ores were leached with sulphuric acid at pH 1.8, plus 3 lb $NaClO_3$ per ton leach feed, giving approximately a 1500 ml volume of leach solution for the solvent extraction shakeout tests. A 200 ml portion of 5% TIOA in kerosene was loaded with uranium and resin-producing ingredients. This was stripped with 50 ml 10% Na_2CO_3 solution, followed by acidification with

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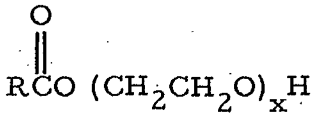
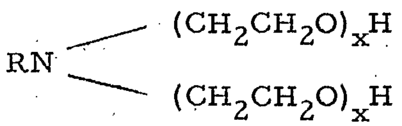
* The three chemicals used in the washing were from Armour and Company, and the solution strengths used were as recommended by that company. The Ethofats are mono-fatty or rosin acid esters of polyoxyethylene glycols. These esters are fairly stable in mildly acid or alkaline solutions.



$$R\overset{\overset{O}{\|}}{C}O\ (CH_2CH_2O)_xH$$

Ethomeen is a tertiary amine having one fatty alkyl group and two polyoxyethylene groups attached to the nitrogen



sulphuric acid to pH 2.5, at which point resin products would separate if washing had not been successful. For a basis of comparison a quantity of resin was obtained in this manner from 1000 gm of unwashed concentrate.

Of the various washing treatments tried on ore sample ref No. 7/57-7 concentrate, hot-water repulping and the mild caustic wash were the only ones in which a substantial amount of the flotation reagents was removed from the concentrate. In both of these tests, the volume of resin obtained was about 25 to 50% of that obtained from the unwashed concentrate. Some resin was deposited on the sides of the vessel at the pulp surface while leaching the washed and unwashed concentrates. Reagent consumption for the caustic wash treatment was 20 lb per ton of concentrate. Repulping with 25°C water alone, or with 25°C water containing either H_2SO_4, Ethofat 242/25, Ethofat 242/60 or Ethomeen c/15 at the concentrations mentioned, was not promising. The amount of resin obtained under these conditions was equal to that obtained from the unwashed ore. In all cases, the repulp filtrates were coloured yellow and they had the characteristic odour of Neo-Fat 42-12, indicating that some dissolution of the fatty acids had been effected.

The preliminary program outlined for preventing this resin problem has been completed, although it was suggested that a blend of the Ethofats and Ethomeen should be tried. For instance, a 50/50 mixture of 1% solutions of detergent and emulsifier might prove more beneficial than either the detergent or emulsifier alone. When time permits this work will be resumed. For solvent extraction of acid leach liquor produced from flotation concentrates of this sort, it is desirable to have a method outlined for eliminating a re-occurrence of the resin condition, since this would permit the simpler sulphate strip system to be employed rather than the carbonate strip.

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