

TREATMENT OF RUM JUNGLE URANIUM ORE

By

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ABSTRACT

The current operational practice at the Territory Enterprises Pty. Limited uranium oxide plant, and some aspects of research and development work at Rum Jungle are outlined with pertinent comments on some overseas practices.

PART A - ORE TREATMENTINTRODUCTION

The Rum Jungle Mining Field is located approximately 40 miles south of Darwin in the Northern Territory.

Uranium oxide production commenced late in 1954. Operations are at present confined to the treatment of ore from the Rum Jungle Creek South orebody, the open cut mining and stockpiling of which was completed in August, 1963.

The overall control of uranium operations is vested in the Australian Atomic Energy Commission, and the management responsibilities are carried out by Territory Enterprises Pty. Ltd., a member company of the Conzinc Riotinto of Australia Group (C.R.A.).

CRUSHING SECTION

The feed to the treatment plant contains uraninite disseminated in the cleavage planes of the chlorite schist host rock. Average uranium value is 7.0 lb  $U_3O_8$  per ton.

The ore, hauled from stockpile by diesel truck, is tipped into a 30 ton coarse ore bin through a 12 in. x 18 in. grizzly. Larger rocks are hand spalled. Feed from the coarse ore bin is via a 30 in. apron feeder to a 24 in. x 14 in. jaw crusher. A 2 in. fixed bar grizzly was positioned between the apron feeder and the jaw crusher. The ore from the crusher and the undersize from the fixed bar grizzly fall onto a 24 in. conveyor belt, designated No. 1, and thence via a 22 in. belt (No. 2) to an 8 ft x 4 ft double deck vibrating screen ( $\frac{3}{4}$  in. x  $\frac{3}{8}$  in.).

The  $+\frac{3}{8}$  in. fraction passes through a 3 ft short head crusher and is returned to the vibrating screen via No. 1 belt. The  $\frac{3}{8}$  in. and finer fractions from the screen are conveyed to the fine ore bins.

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Grinding Section Statistics

Average grinding rate	-	17.0 t.p.h.
" shell liner life	-	4,750 hours
" feed-end liner life	-	9,500 "
" discharge end liner life	-	21,280 "
Feed end trunnion liner life	-	11,500 "
3" dia. rods - consumption	-	0.5 lb/ton ore
Pyrolusite - "	-	7.0 " " "
*Lime - "	-	1.3 " " "

\*Lime is used to neutralize mill feed water, owing to its corrosive nature.

LEACHING AND LIQUID SOLID SEPARATION

Pulp from the rod mill is pumped via a 6 in./4 in. pump to seven 15 ft dia. x 39 ft high rubber lined pachucas. One pachuca is used as a leach vessel, one as surge, leaving five for pulp storage. Pulp transfer between pachucas is via a 4 in./3 in. pump.

Sulphuric acid is added into the leaching vessel and the pulp, diluted to  $\pm 1120$  density, is fed into the counter current decantation (C.C.D.) circuit. Batch leaching is carried out, retention time being  $2\frac{1}{2}$  hours.

The C.C.D. circuit employs four thickeners, one 100 ft dia., designated T1, into which leached pulp is fed via a 4 in. O.D.S. pump, and three 75 ft dia., designated T2, T3 and T4. Wash water at a specified rate, 150 g.p.m. at pH 3.5, is fed into T4, and the overflow wash water is channelled, counter current to the solids, to T1. The underflow is fed from T1 to T3 via 3 in./2 pumps, while the underflow from T4 at 1460 density is pumped via a 4 in./3 in. pump to a sump, where it is automatically sampled, and then via a 6 in./4 in. pump to the residue dam. Residue pH is  $\pm 2.5 - 3.0$ .

Flocculant used is gum guar.

The overall extraction efficiency of the leaching and C.C.D. circuit is 96.5 per cent and it is considered that secondary leaching takes place in the C.C.D. section. Sulphuric acid is added to T2 to maintain correct leaching conditions.

The overflow, pregnant liquor from T1 is clarified in sand clarifiers, four in number, 7 ft dia. by 12 ft high. Clarifiers are backwashed in sequence, one per day.

Leaching and liquid solid separation statistics:

Initial sulphuric acid (leaching)	-	125 lb/ton ore
Secondary " " (T2)	-	25 lb/ton ore
Total		<u>150 lb/ton ore</u>

Typical sizing of the "fine ore" is:

TABLE 1

Screen size (B.S.S.)	% by weight	Cum. % by weight
+ $\frac{1}{2}$ in.	Nil	Nil
- $\frac{1}{2}$ + $\frac{3}{8}$	6.8	6.8
+10	57.8	64.6
-10	35.4	100.0

Dust is collected at selected points in the crushing section and is cycled to the thickening circuit.

Some operating statistics for the crushing section are as follows

- (a) Nominal capacity 50 t.p.h.
- (b) Jaw crusher
  - Swing liners - average life 6,000 hours
  - Fixed liner - average life 12,200 hours
- (c) Symons cone crusher
  - Mantle liner - average life 4,300 hours
  - Bowl liner - average life 2,150 hours

A flowsheet for the crushing section is given in Fig. 1.

#### GRINDING SECTION

Ore from the fine ore bins (2 x 500 tons nominal capacity) is conveyed over a weightometer to a 5 ft x 10 ft rod mill. Pyrolusite, as an oxidant, is added at this point from a bin of nominal 100 ton capacity and blended with the ore in the rod mill, which is in open circuit.

The discharge pulp is kept at  $\pm 58$  per cent solids, and a typical sizing analysis of the pulp is as follows:

TABLE 2

#### Rod mill discharge pulp

Screen size (B.S.S.)	% by weight	Cum. % by weight
+ 10 mesh	-	-
+ 18 "	2.5	2.5
- 18/+ 25 "	4.5	7.0
- 25/+ 36 "	6.2	13.2
- 36/+ 60 "	13.5	26.7
- 60/+100 "	11.2	37.9
-100/+200 "	11.8	49.7
-200 "	50.3	100.0

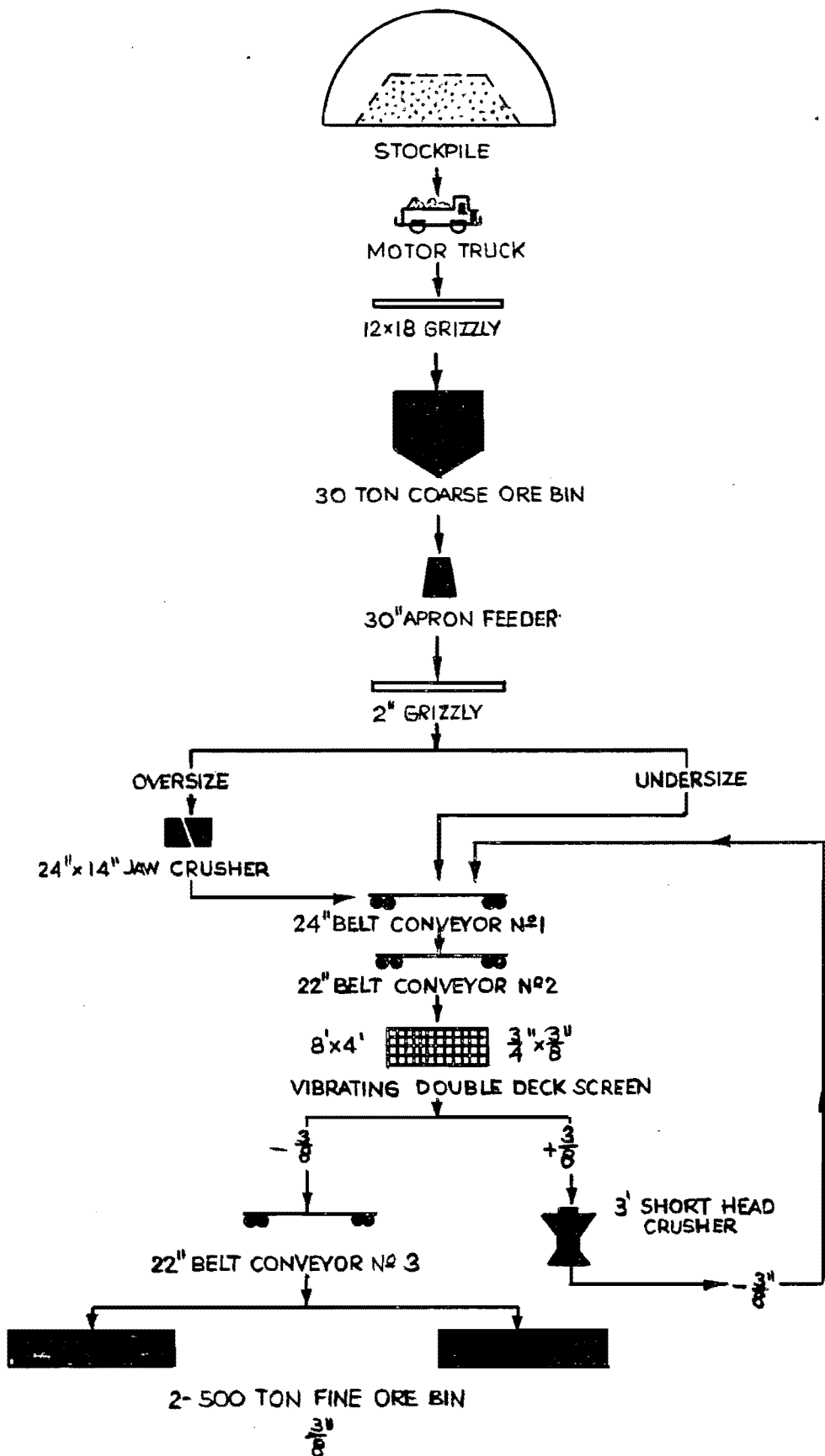


Fig. 1. Crushing section.

Gum Guar - (flocculant) - 0.27 lb/ton ore leached.

TABLE 3

Pregnant liquor analysis

U <sub>3</sub> O <sub>8</sub>	±	0.8	g.p.l.
Fe <sup>++</sup>		None found	
Fe <sup>+++</sup>		1.3	g.p.l.
Mg		1.3	g.p.l.
Mn		0.4	g.p.l.
Ni		0.018	g.p.l.
Pb		0.20	g.p.l.
Mo		0.001	g.p.l.
Cu		0.13	g.p.l.
SO <sub>3</sub>		15.2	g.p.l.
V		0.015	g.p.l.
Free acid		7.10	g.p.l.
SiO <sub>2</sub>		0.42	g.p.l.
pH		1.5	

Pumps - average impellor life

6/4 Pump (Rod mill discharge)	-	350 hours
3/2 " (T1 underflow)	-	5,000 hours
3/2 " (T2 underflow)	-	1,600 hours
3/2 " (T3 underflow)	-	1,600 hours
4/3 " (T4 underflow)	-	2,720 hours
6/4 " (Residue disposal)	-	530 hours
4/3 " (Pulp transfer pachucas)	-	800 hours

NOTE: Each unit of equipment has two pumps, one in use and one on standby.

A flowsheet for the grinding, leaching and L.S.S. separation sections is given in Fig. 2.

SOLVENT EXTRACTION (S.X.) SECTION

Clarified pregnant liquor is processed in the solvent extraction section consisting of four extraction stages and four stripping stages.

The extraction vessels are 12 ft dia. by 10 ft high and are epoxy resin lined.

The stripping vessels are 6 ft dia. by 6 ft high and are also epoxy resin lined. Pipe work in the S.X. section is fabricated from polyvinyl chloride (P.V.C.).

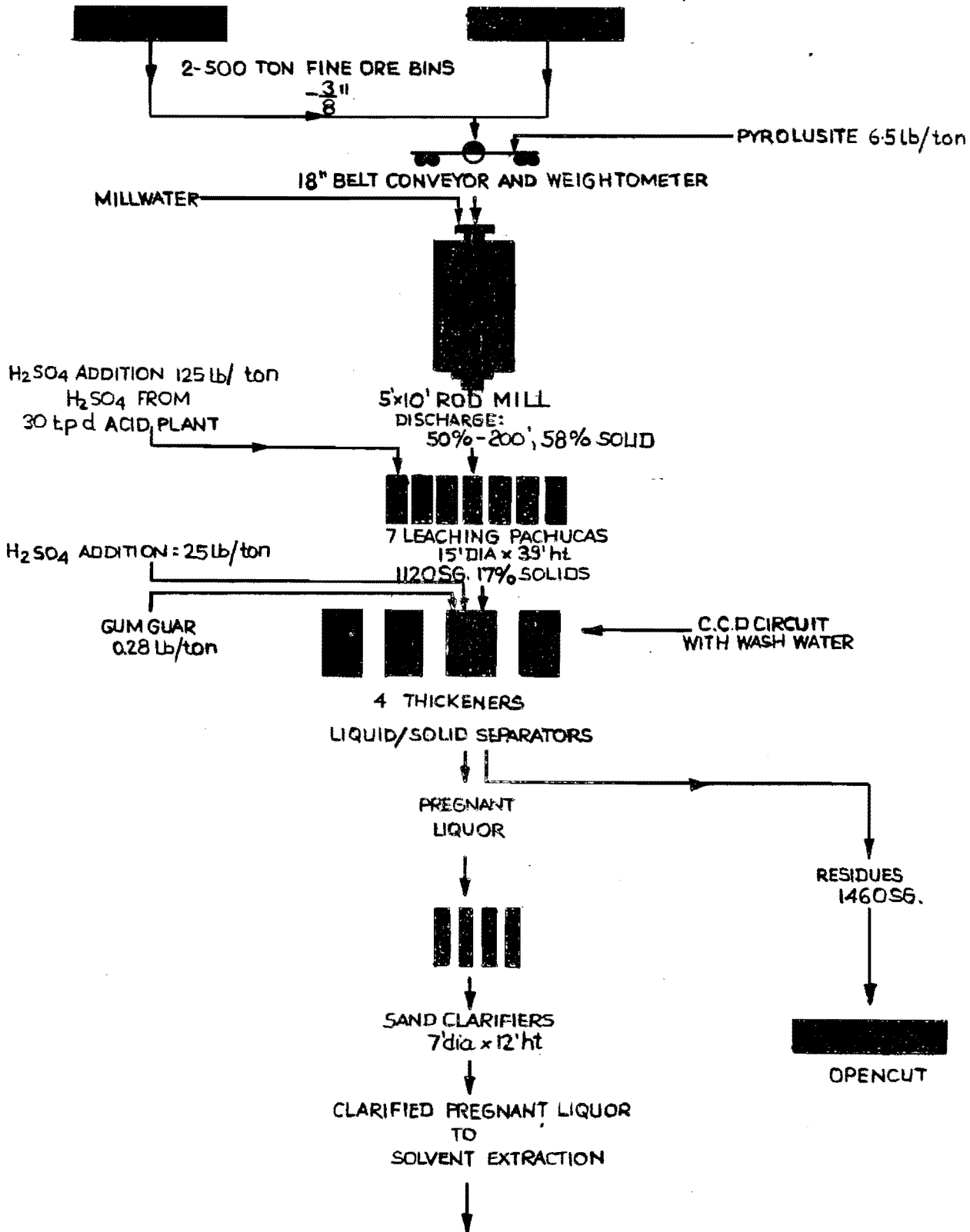


Fig. 2. Grinding, leaching and liquid solid separation sections.

Pregnant liquor is pumped to the S.X. via a 4 in./3 in. pump. Pump impellers are made of 316 stainless steel, and appear to have an indefinite life.

Figure 3 shows the arrangement of the extraction and stripping circuits, and Figure 4 gives details of the extractor mixer-contactor baskets.

The flow of pregnant liquor and solvent through the extraction stage is counter current.

The solvent consists of 5 per cent tertiary amine, 3 per cent nonanol, and 92 per cent kerosene. Solvent is fed to the extractors via a 2 in. pump and is contacted with the aqueous phase in P.V.C. contactor baskets. This constitutes the mixer part of the extractors, the remaining volume being for settling. Phase disengagement time is approximately 30 secs. (primary) and total (secondary) disengagement time  $\pm 90$  secs. When this plant was designed, total disengagement time was taken into account. Raffinate at pH 1.5 is pumped to waste. Present thinking on S.X. plant design is that primary disengagement time only should form the basis for calculations, therefore the Rum Jungle S.X. extraction circuit is probably oversized for settler volume. This has allowed the plant to be run at greater than design volume flows without excessive solvent losses or increase in raffinate grade because of loaded solvent carry-through.

Pregnant liquor retention time per extraction stage is in the region of 30 minutes.

Loaded solvent from the extractors, containing  $\pm 5.0$  g.p.l.  $U_3O_8$  is pumped to the stripping section via a 2 in. pump (see Figure 3).

Strip solutions consisting of 60.0 g.p.l. NaCl and at pH 5.8 is pumped to the stripping vessels through a 1 in./ $\frac{3}{4}$  in. pump, and flows counter-current to the loaded solvent. Aqueous transfer in the strippers is by means of air-lift. The solvent after stripping contains  $< 0.1$  g.p.l.  $U_3O_8$  and is recycled to the extractors.

Typical "high-grade strip" analysis is as follows:

#### Solvent extraction section statistics:

Flow ratio, extraction	-	Aqueous/organic 5 : 1
" " stripping	-	Organic/aqueous 5 : 1
Recycle solvent	-	$< 0.1$ g.p.l. $U_3O_8$
Raffinate	-	$< 0.001$ g.p.l. $U_3O_8$

#### Reagent consumption -

Amine	-	0.004 lb/lb $U_3O_8$
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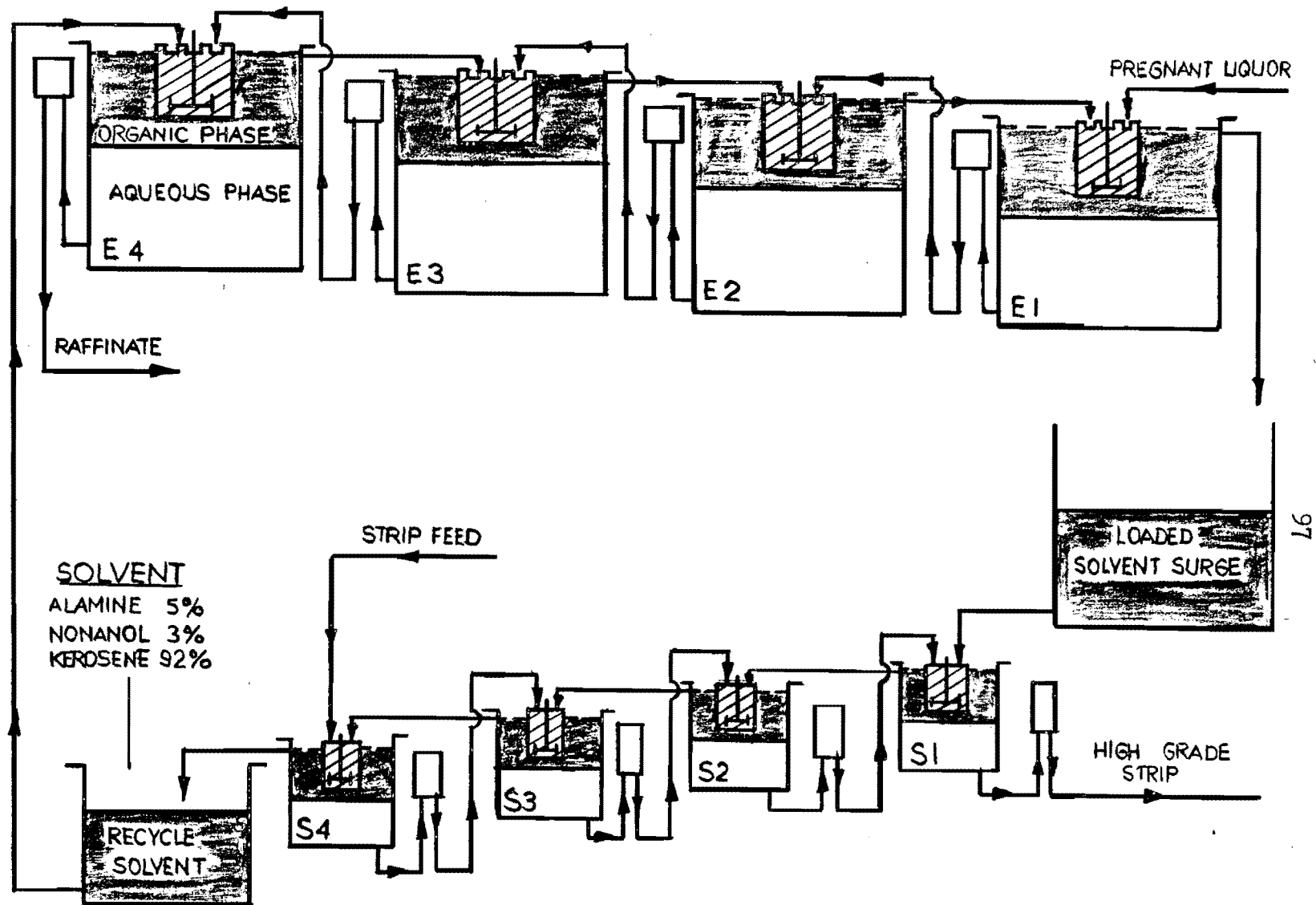
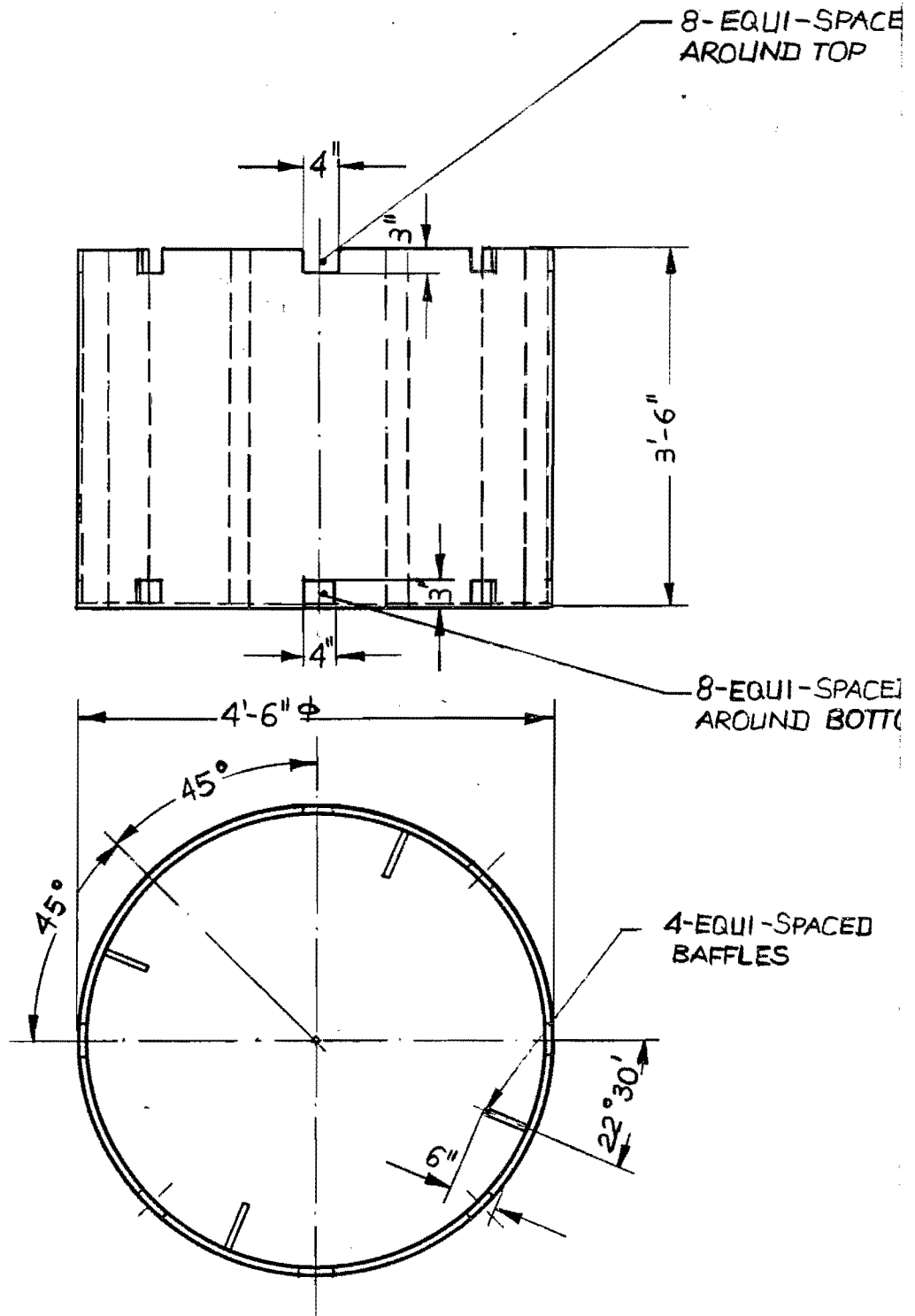


Fig. 3. Extractions and strippers.





ES:

BRICATED FROM  $\frac{1}{4}$ " THICK P.V.C.  
 ITATOR IS STAINLESS STEEL &  
 OLDING BOLTS ARE TITANIUM.

Fig. 4 Extractor mixer unit.

Nonanol	-	0.071 lb/lb $U_3O_8$
Kerosene	-	0.017 gal/lb $U_3O_8$
NaCl	-	1.5 lb/lb $U_3O_8$

TABLE 4High grade strip

$U_3O_8$	$\pm$ 23.0	g.p.l.
$Fe^{++}$	None found	
$Fe^{3+}$	0.02	g.p.l.
Mg	0.60	g.p.l.
Mn	0.005	g.p.l.
Ni	0.003	g.p.l.
Pb	0.01	g.p.l.
Mo	0.004	g.p.l.
Cu	0.0035	g.p.l.
$SO_3$	109.5	g.p.l.
V	0.002	g.p.l.
Free acid	2.8	g.p.l.
$SiO_2$	0.14	g.p.l.
pH	2.1	

PRECIPITATION SECTION

In the precipitation section, uranium is precipitated from high grade strip with 5 per cent NaOH solution on a batchwise basis. Earlier plant practice used MgO slurry as precipitant. Precipitations are carried out in a rubber lined conical vessel at a temperature of 60°C. The caustic soda solution flow is automatically controlled by a pH recorder/controller. After the precipitate has settled, it is then transferred to a second conical vessel where it is given a five hour "hot wash" at 90°C.

Filtering -

After settling, the washed precipitate is transferred to a first stage drum filter, 4 ft x 3 ft dia., 18 panels, 1/3rd submerged. This filter is fitted with a terylene polyester cloth and roller discharge, and after filtering the precipitate is repulped and transferred to a 25 ft dia. thickener via a 3 in./2 in. pump.

From the thickener the pulp is transferred to a second stage drum filter, 4 ft x 3 ft dia., 12 panels, 1/3rd submerged, also fitted with terylene polyester cloth and a string discharge.

### Dryer -

The precipitate falls into a repulp tank where it is agitated and pumped via a  $1\frac{1}{2}$  in. pump to a 12 ft x 5 ft dia. multiple hearth calciner. The calciner feed has a specific gravity of  $\pm 1.85$ . Total residence time in the calciner is  $\pm 30$  minutes and operating temperatures here are approximately  $800^{\circ}\text{C}$ .

The calcined precipitate passes through a roll crusher 8 in. dia. by 6 in. wide for size reduction and then via a 14 ft long water-cooled screw conveyor, where the precipitate is cooled before packing in polythene lined 44 gallon drums. Packed moisture content is 0.5 per cent  $\text{H}_2\text{O}$ .

### Drumming -

During drum filling, the drums are automatically vibrated, and present packed weight of drums is  $\pm 800$  lb/drum. After sampling and weighing, the drums are sealed and removed for storage.

A description of the possible structure and composition of the 'yellow cake' and some observations on precipitation are given in Part B of this paper.

Table 5 compares the analysis of the NaOH "yellow cake" with that obtained when MgO slurry was used as a precipitant. It should be noted that the analysis of the MgO "yellow cake" reflects the best grade of product obtained using this precipitant at the Rum Jungle plant.

A flowsheet from the solvent extraction through to the drumming section is given in Fig. 5.

### SULPHURIC ACID PLANT

This is a nominal 30 ton/day plant; 2.8 tons acid are produced per ton of sulphur consumed.

### CONTROL LABORATORY

This laboratory carries out plant control and research analysis using, amongst other, Atomic Absorption Spectroscopic techniques.

### SERVICES

#### Workshop

Adequate engineering workshop facilities are available for maintenance

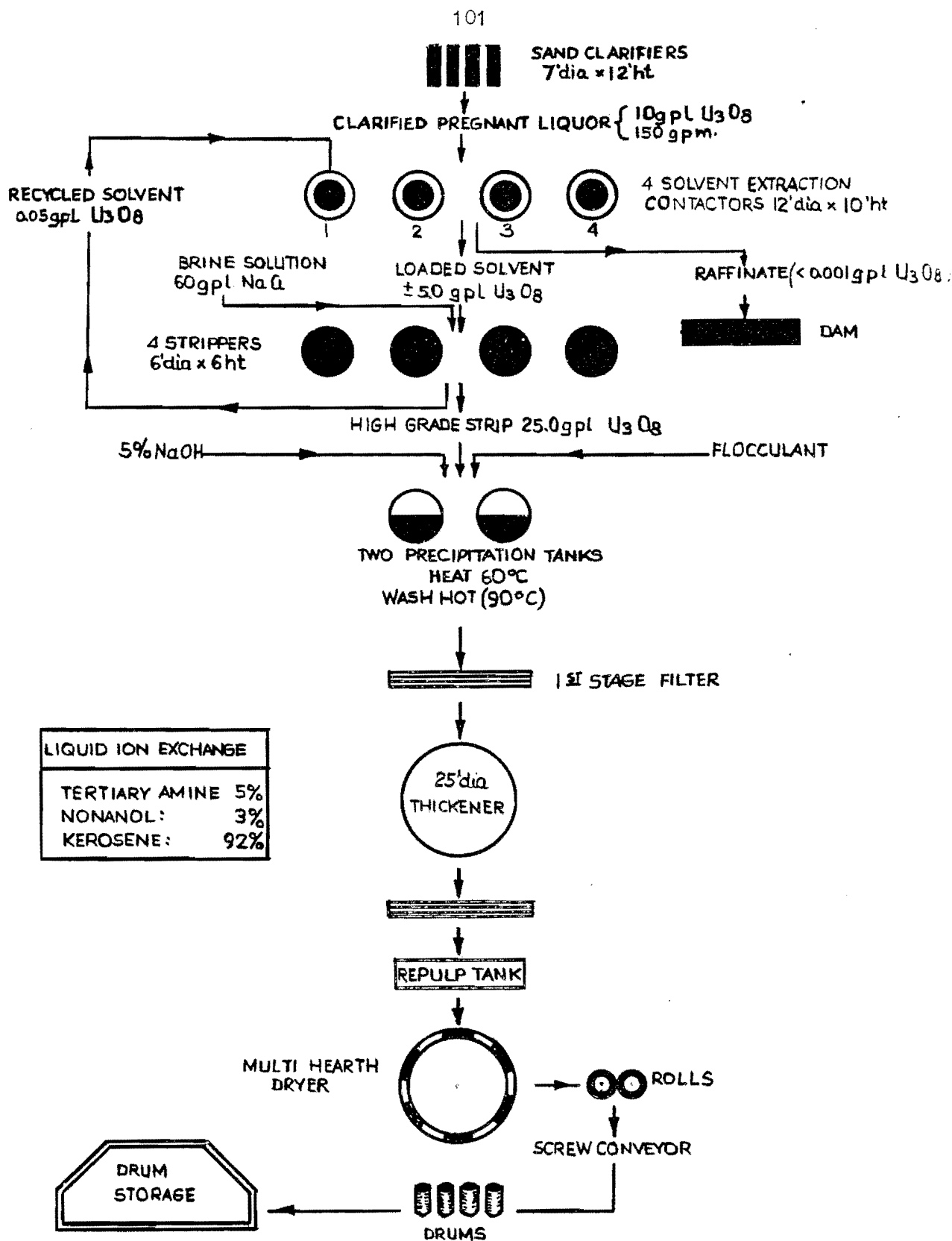


Fig 5. Solvent extraction section.

TABLE 5

Constituent		"Yellow cake"	
		NaOH product	MgO product
U <sub>3</sub> O <sub>8</sub>		92.8 %	89.25 %
Boron		<5 ppm	<4 ppm
Cadmium	cd	<5 ppm	<5 ppm
Thorium oxide	Tho <sub>2</sub>	<10 ppm	<12 ppm
Rare earth oxides		0.002%	0.006%
Vanadium	V	<10 ppm	<10 ppm
Ferric oxide	Fe <sub>2</sub> O <sub>3</sub>	0.25 %	0.26 %
Phosphorous Pentoxide	P <sub>2</sub> O <sub>5</sub>	0.02 %	0.029 %
Silica	SiO <sub>2</sub>	0.16 %	0.49 %
Molybdenum	Mo	<10 ppm	<10 ppm
Aluminium oxide	Al <sub>2</sub> O <sub>3</sub>	0.04 %	0.41 %
Arsenous oxide	As <sub>2</sub> O <sub>3</sub>	1 ppm	3.95 ppm
Calcium oxide	CaO	0.06 %	0.70 %
Cobalt	Co	0.002 %	0.0022 %
Mercury	Hg	<0.15 ppm	<0.15 ppm
Nickel	Ni	0.002 %	0.019 %
Sulphur trioxide	SO <sub>3</sub>	0.74 %	0.70 %
Magnesium oxide	MgO	0.05 %	0.61 %
Sodium oxide	Na <sub>2</sub> O	3.10 %	2.26 %
Chloride	Cl	0.004 %	0.007 %
Potassium oxide	K <sub>2</sub> O	0.14 %	0.11 %
Titanium oxide	TiO <sub>2</sub>	0.002 %	0.004 %
Copper	Cu	0.012 %	0.175 %
Manganese	Mn	0.006 %	0.070 %
SiO <sub>2</sub> and insols.		0.18 %	0.54 %

Reagent consumption - precipitation section:

NaOH

0.45 lb/lb U<sub>3</sub>O<sub>8</sub> produced

and general work in the treatment plant, and also for the barrication of experimental required in the research and development section.

#### Power House -

Power is company generated at 415V by four diesels driving alternators -

Installed b.h.p.	2670
" kVA	2350

#### Compressed air -

Air supply is provided by four diesel driven compressors located in the power house.

#### SOME OVERSEAS PRACTICES - COMPARISONS AND COMMENTS

##### Crushing and grinding

These sections essentially incorporate jaw and cone crushers with vibrating screens in close circuit.

Preconcentration of ore by radiometric detectors or sorters is not extensively adopted. The treatment plant of the Commissariat a l'Energie Atomique, Bessines, France, obtains a rejection efficiency of 83% (with the waste containing approximately 0.4 lb  $U_3O_8$ /ton by radiometric detection) of their washed granitic ore prior to final crushing and grinding.

Rod and ball mills either individually or conjointly comprise the grinding circuit, together with classifiers (rake or spiral cyclones or separator screens), dependent on the characteristics of the ore and the sands/slimes ration.

In some Wyoming (U.S.A.) plants and in Bessines (France), the uranium values are contained in the sands necessitating their separation from the slimes by drag classifiers or hydro classifiers.

At Rum Jungle, single stage grinding only is necessary because a one pass rod mill grind gives adequate liberation of uranium minerals. (0.5mm - 1.0 mm).

The ore grindability is shown by the comparison -

	<u>Rod consumption</u>
	<u>lb.ton ore</u>
Rum Jungle	0.5
Wyoming area (U.S.A.)	1.0
Elliot Lake area (Canada)	3.5

## Leaching and liquid solid separation

The use of either acid or alkaline carbonate leachant is dependent on the ore type, which essentially decides the relative economics.

The greater proportion of operating plants carry out leaching with sulphuric acid, and the acid consumption ranges from -

Rum Jungle	150 lb/ton of ore
Wyoming	60-100 " " " "
Canada	60-70 " " " "
France	80-120 " " " "

Dissolution is carried out either in air-agitated pachuca tanks, rotary drum trommels, or turbine agitation tanks.

Oxidants used include pyrolusite and sodium chlorate.

The liquid solid separation (L.S.S.) phase and the items of equipment used is dependent on the routes the subsequent uranium recovery methods follow, i.e. ion exchange, resin-in-pulp (R.I.P.) solvent extraction or Eluex. All require a clear pregnant solution except the R.I.P., which will tolerate considerable fines.

Most L.S.S. equipment uses the counter current system in which solids are moved against the solution flow. These include thickeners, agitators and filters. Flocculants are sometimes used.

## Uranium recovery methods

Early commercial methods of recovering uranium from solutions (pre 1950) had the disadvantages of high reagent costs, incomplete precipitation and loss of product in barren solutions. Subsequent developments arising from chemical research and application to the industry gave the chemical engineer and hydro metallurgist greater flexibility in the choice of recovery methods.

### Ion exchange process (I.X.) -

The first major improvement came with the introduction of ion exchange in South Africa during 1950.

In this process, sulphuric acid leach solutions are passed through a bed of anion exchange resin. The uranium is adsorbed (preferentially) on the resin and the barren solution discarded. Under ideal conditions, high recoveries are possible. The uranium may be removed from the resin by passing through it a strong electrolyte containing nitrate, chloride or sulphate and thus achieving a high concentration factor.

The uranium may then be precipitated from the eluate by the use of an

alkali. Ammonia, magnesia and caustic soda are commonly used.

Of the plants observed in the U.S.A., only two use I.X. exclusively for concentration. One in Wyoming - Utah Coy. - utilizes moving resin bed techniques, whilst in Union Carbide's plant in Colorado, fixed resin bed techniques are used.

In Canada, the Nordic Mill uses fixed bed column ion exchange, and when the column becomes loaded, uranium is eluted from the resin with Sodium nitrate.

#### Resin-in-pulp process (R-I-P) -

This is an adaptation of the I.X. process and designed to handle slimes which defy normal liquid solid separation methods.

The chemistry is similar to column I.X. but the uranium is loaded on resins which are contained in wire screen baskets from a desanded pulp. No filtration or thickening step is necessary.

The R-I-P was first used in earlier uranium production plants before the advent of efficient flocculents. It is debatable whether modern designed plants would incorporate R-I-P unless the slimes problem was a difficult one to overcome.

To offset its apparent advantages, its capital cost is high, and water consumption is up to four times that needed for column I.X. The consumption of eluting chemicals and resin is high, as is the maintenance.

At the Split Rock Mill of Western Nuclear in Wyoming, the original basket R-I-P circuit (in which baskets of resin were oscillated in pulp) has been altered to a moving bed circuit, and the resin is removed by vibrating screens. However, this plant, along with the Federal Mill (Wyoming), have added a solvent extraction stage which is known as

#### The Eluex process -

This is a two-stage process of R-I-P and S.X. and despite the additional steps, it saves the cost of a nitrate strip by using cheaper ammonium sulphate. The grade of final product is improved.

#### Solvent extraction process (S.X.) -

In this process, as applied at Rum Jungle, the solvents are liquid ion exchange materials which are diluted in kerosene.

Intimate contacting of solvent and acid leach liquors result in the rapid transfer of uranium from the aqueous phase to the organic phase. The mixture is allowed to settle for phase disengagement and, after separation of the two immiscible phases, the uranium may be stripped from the organic phase by reagents similar to those used for I.X. resins.



Rum Jungle uses sodium chloride as a stripping agent.

There are both anionic and cationic solvents which exhibit high selectivity for uranium, giving greater flexibility than any of the foregoing processes.

The main problems with S.X. are,

1. solvents can become poisoned with elements which must be removed by regeneration treatment,
2. there is, in addition, a need for make up of the solvent, because of slight losses in the aqueous phase, and
3. a third phase is formed.

At Rum Jungle, a tertiary amine in a kerosene diluent is used, with nonanol as a carrier added for emulsion control. This is the usual overseas practice, though tridecanol may replace nonanol, which is cheaper when available.

#### Precipitation processes

Overseas treatment plants use  $\text{NH}_3$ ,  $\text{NaOH}$  or  $\text{MgO}$  as precipitants, depending on the economics of procurement.

All use the batch system of precipitation and there are no continuous precipitation methods adopted.

#### Quality of product

There is a growing awareness amongst currently active overseas producers of "yellow cake" of the importance of producing a higher grade low impurity product in order to meet buyers' requirements. The supplier is called upon - in these times of over-supply of product - to tailor his product to the market needs during the so-called "stretch out" programme.

One buyer in particular - Allied Chemicals, Illinois, - in their process to convert "yellow cake" to uranium tetrafluoride ( $\text{UF}_6$ ) requires a low impurity product, with penalties for product above a specified content of  $\text{V}_2\text{O}_5$ ,  $\text{PO}_4$  and Na.

In appreciation of this fact, many suppliers have modified their uranium recovery section, this being the main reason why Utah Coy. are planning to supplement their I.X. with S.X., and Western Nuclear have extended their circuit to Eluex. In addition, the swing is towards  $\text{NH}_3$  as precipitant to minimise impurity content, because of its relative cheapness and availability.

Table 7 compares the "yellow cake" requirement of Allied Chemicals, and the product of Rum Jungle.

TABLE 7Uranium concentrates for UF<sub>6</sub> conversion -Specifications by Allied Chemicals, U.S.A.

		"Standard" Conc.	"Maximum" Limit Conc.	Rum Jungle Conc.	
		%	%	NaOH ppn. %	MgO ppn. %
Uranium	as U	75.0	65.0 min	78.6	75.7
Vanadium	V <sub>2</sub> O <sub>5</sub>	0.10	1.80 max	0.001	0.001
Phosphorous	PO <sub>4</sub>	0.10	0.70 "	0.027	0.039
Chloride	Cl	0.05	0.10 "	0.004	0.007
Molybdenum	Mo	0.10	0.30 "	0.001	0.001
Sulphur	SO <sub>4</sub>	3.00	8.00 "	0.890	0.840
Iron	Fe	0.15	1.00 "	0.180	0.180
Arsenic	As	0.05	0.15 "	0.0001	0.0003
Calcium	Ca	0.05	0.75 "	0.040	0.050
Sodium	Na	0.50	7.50 "	2.300	1.700
Boron	B	0.005	0.10 "	0.0005	0.0005
Potassium	K	0.10	3.00 "	0.120	0.090
Water	H <sub>2</sub> O	2.00	4.00 "	0.400	0.400

Drying and packing of "yellow cake"

Multi-hearth rabble operated dryers are commonly used for drying "yellow cake". Either ore or gas firing is employed. Two plants, one in U.S.A. and the other in France, employ steam heated belt dryers.

Packing is univerally done in drums of 40 to 44 gal capacity where transport over long distances is necessary.

PART B

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RESEARCH AND DEVELOPMENTPRECIPITATION OF URANIUM FROM HIGH GRADE STRIP

The initial Rum Jungle plant design incorporated ion exchange (I.X.).

At the time of the commissioning of the solvent extraction section (S.X.) at Rum Jungle in October, 1962, the general principles by which a desired uranium precipitate could be obtained from the high grade strip were not fully appreciated. Research has since been carried out in this field at Rum Jungle by Stockley (1963, 1965), Mather (1966-67) and Harris (1966-67). The better appreciation of the variables involved has led to significant advances in plant precipitation techniques for the batch process.

The effect on product grade and impurity levels is illustrated in Table 5. As stated earlier, the uranium product prior to September, 1966, was precipitated with a slurry of high grade magnesium oxide. From September, 1966, onwards, the precipitant has been a caustic soda solution.

During the commissioning period of the solvent extraction section, low grades of uranium precipitate were obtained with sulphate as the main impurity. The average grade product on the dry basis ( $110^{\circ}\text{C}$ ) for the first four weeks was 81.07 per cent  $\text{U}_3\text{O}_8$  compared with an average value of 93.19 per cent  $\text{U}_3\text{O}_8$  (on a dry basis) for the four weeks in January, 1968.

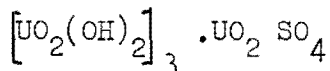
Treatment plant practice at the commissioning of the solvent extraction section was as follows:-

The uranium contained in the high grade strip liquor from the solvent extraction section was precipitated approximately every 24 hours. The technique was to add the magnesium oxide slowly over a period of about 6 hours, in the form of a slurry containing about 30 per cent solids.

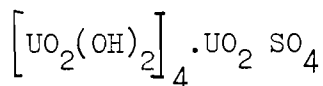
In order to minimise the consumption of magnesium oxide "double chargin" was employed. This entailed pumping the product slurry from one precipitation into the next tank of high grade strip to be precipitated. All precipitations were conducted at ambient temperatures.

Stockley (1963) showed that the slow rate of magnesium oxide addition and "double charging" led directly to the formation of a sulphate impurity in the precipitate.

Investigations into the chemical processes involved in the precipitation on the nature of the sulphate containment showed that at Rum Jungle during a precipitation at ambient temperature, the most basic sulphate compound stable in solution had the empirical formula



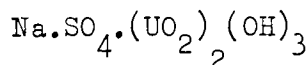
This did not agree with the results obtained by Arden, Humphreys and Lewis (1948, p.17), who found the most basic sulphate stable in solution at ambient temperature had the empirical formula



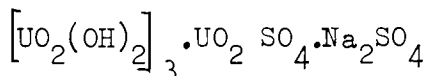
No sulphate was reported by these workers in their products. The difference between their results and those found at Rum Jungle is thought to be due to the use by Arden (op. cit.) of pure solutions low in sulphate, compared with the high sulphate content solutions which are an integral part of the amine solvent extraction process.

An understanding of the nature of the sulphate contaminant in the solids was an essential step in the improvement of precipitation techniques.

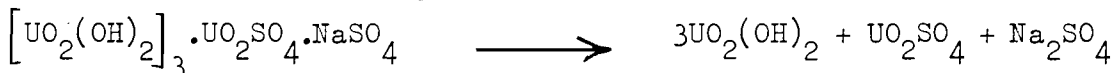
Following isolation of the sulphatic contaminant, work at Rum Jungle strongly suggested that it was a mixture of sodium sulphate and a complex compound of the empirical formula



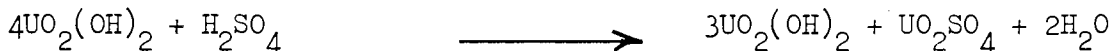
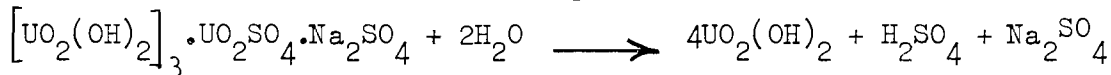
The chemical behaviour of this complex compound was such as to suggest it to be



This compound was shown to quantitatively decompose in boiling water according to the following overall reaction



The following steps were postulated to explain the observed release of some hydrogen ions during the decomposition



Knowledge of this reaction was instrumental in the adoption of a "hot wash" for the precipitate as standard plant practice. For a hot wash to be effective, when significant quantities of the basic uranium sulphate are present, a further addition of precipitant is essential during the hot wash stage. Such a step was found necessary on some occasions in the past.

The formation of the basic sulphate was a fairly slow process. It formed predominantly in the pH range of 4.8 to 5.1. By raising the pH as rapidly as possible to 5.1, the formation of all but small amounts of sulphate impurity was avoided. The terminal pH could then be approached slowly to avoid "overshooting" it. An "overshot" precipitate resulted in the formation of excess diuranate, which was shown to

occur as a direct function of increase in pH.

The temperature of precipitation had a substantial effect on the settling characteristics of the resultant precipitate. Table 8 indicates the results of a series of tests carried out.

TABLE 8

The effect of temperature on settling characteristics

TEMP. °C	% Solids A	% Solids B	TIME C	
			(i)	(ii)
26	10.0	11.4	45	40
38	11.0	13.1	45	30
49	13.6	14.7	40	20
58	16.5	19.0	30	10
69	22.0	26.4	20	5
80	27.4	34.0	5	5
92	37.1	46.1	5	5

- A. % solids  $\frac{W}{W}$  in settled pulp after 2 hours with and without flocculant addition.
- B. % solids  $\frac{W}{W}$  in settled pulp after 2 hours with and without flocculant addition.
- C. time taken in minutes for pulp to settle to 90% of that achieved in 3 hours -  
     (i) no flocculant;  
     (ii) with flocculant.

The precipitate formed at the higher temperatures was not only of superior settling characteristics but of vastly superior filtering characteristics.

As a result of these findings it became standard practice to precipitate the uranium product at approximately 85°C.

The overall effect of the work was to raise the grade of product from the initial 81.1 per cent  $U_3O_8$  at the end of 1962 to 87.7 per cent  $U_3O_8$  at the end of 1963. However, the continued use of magnesium oxide as a precipitant was considered undersirable for the following reasons.

1. The precipitant was a solid which was not fully consumed till about two hours after it had been added.
2. The reactivity of the solid precipitant was not constant but varied from shipment to shipment, thus introducing considerable margins for error in precipitant addition and consequently product grade.

3. The precipitant itself was approximately 93 per cent MgO and carried with it undersirable impurities such as silica and carbonate.

Because of these objections to the use of MgO as a precipitant, pilot scale work was carried out to evaluate the use of  $\text{NH}_4\text{OH}$  and NaOH. The use of  $\text{NH}_4\text{OH}$  as a precipitant was discounted because<sup>4</sup> of its cost and NaOH was tested extensively. These studies indicated that a higher grade precipitate could be obtained through the use of caustic soda, but that this would occur to the detriment of the settling, filtering and drying characteristics of the product.

Preliminary tests on a plant scale in 1963/64 indicated that the filtration rate of the product could be inadequate. The available capacity of the plant dryer was inadequate.

The installation of a new rotary oil-fired multi-dryer in August, 1966, removed the major restriction of dryer capacity and an extended plant run on caustic soda was initiated.

By heating the slurry to be filtered to about  $90^\circ\text{C}$ , filtration rate was barely adequate for budgeted production at 120 - 150 lb per operating hour. At lower temperatures the filters imposed a limitation on production. Steam consumption at a filter feed temperature of  $90^\circ\text{C}$  was excessive. Mather (1967) by diluting the precipitant strength from 14 per cent NaOH w/w to 9 per cent NaOH w/w, was able to increase filtration rate to 150 - 180 lb/hour.

Prior to the changeover to NaOH precipitant, a feed slurry of 45 per cent solids had been pumped to the multi-hearth dryer.

With the advent of NaOH precipitant, the slurry became too viscous to pump even at 25 per cent solids w/w. As a temporary expedient, steam was used to heat the feed but this proved to be expensive. An evaluation of the fundamental cause of the trouble suggested that certain organic surface active agents might have a beneficial effect by particle to particle contacting.

Enquiries on this line brought forward a dispersant which, when used at about 0.008 lb/lb of  $\text{U}_3\text{O}_8$ , had a particularly beneficial effect on the viscosity and its use virtually eliminated the need for steam. Later developments in precipitation technique ultimately overcame the need even for this reagent.

The grade of product obtained with caustic soda at this stage was not as high as had been indicated by early pilot scale testwork and a disturbing trend had been noticed, viz. the product grade and handling characteristics were both showing indications of having undergone a steady deterioration from the time the caustic had been initially introduced. The decrease in product grade was due to increases in the level of sodium as an impurity.

The deterioration appeared to correlate with the gradual depletion of  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$  from the circulating solutions. It was postulated that the reason for the deterioration in both grade and handling characteristics was attributable to a lesser degree of flocculation of the precipitate formed. On these grounds, after several trials, a particular flocculant was added to the high grade strip prior to precipitation of uranium. This produced significant improvements in product handling characteristics without having any apparent effect on product grade.

In order to further evaluate this variable, a series of pilot scale tests were carried out.

During the standardisation of this pilot scale testwork, some interesting anomalies were observed.

- a. The product handling characteristics and, to some extent, the grade were directly a function of the rate of addition of precipitant.
- b. The rate of addition of precipitant at a time when no precipitate was visible still had a significant effect on product handling characteristics.

By slowing down the rate of addition of precipitant, a hundredfold increase in the standard pilot scale filtration test was achieved. The slow precipitation resulted in significant quantities of sulphate forming in the product, but this was easily removed by the normal hot-washing step, together with caustic addition to the hot-wash.

When this technique of slow precipitation was applied to the plant, it led to appreciable increases in filtering rate, even without the prior addition of flocculant to the high grade strip.

The dilution of the precipitant to 5 per cent NaOH w/w and the use of a spray nozzle manifold instead of a plug flow discharge from a pipe, contributed substantially to the benefits now obtained. Filtering rate is now approximately ten times the initial rate. These techniques resulted in considerable reductions in dryer operating time, with subsequent savings in fuel oil costs.

The slower precipitation rate was simplified by automation of the caustic addition based on pH measurement. Final pH is now approximately 6.0 and precipitation temperature has been reduced to 60°C.

The real parameter that had affected precipitate grade and handling characteristics in the above case was not rate of addition of precipitant, but the change in degree of local inhomogeneity caused by changes in rate of addition of precipitant. The effect on precipitate handling characteristics caused by differing degrees of local inhomogeneity occurring before precipitates became visible is tentatively attributed to variations in the number of seed crystals formed.

The observations made on this aspect have suggested a method of continuous precipitation which is currently being examined.

During the investigation into the high viscosity slurries produced with NaOH precipitant, and later when the deterioration in product grade from that initially obtained with NaOH posed a problem, hypothetical structures of the chemical compounds involved in the precipitate were investigated.

Mather, (1967B) has outlined current thoughts on these hypothetical structures. These are given below.

In the precipitation section of the Rum Jungle Plant, uranium is present in solution as the uranyl ion  $\text{UO}_2^{++}$ . Also present in the uranium solution are sulphate, sodium and chloride ions (traces of others are also present).

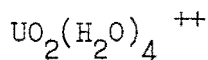
Caustic soda is at present used to precipitate uranium from solution as "yellow cake", or what is loosely termed sodium diuranate. In fact, the precipitate composition is somewhat indefinite, being between uranyl hydroxide,  $\text{UO}_3$ , and sodium diuranate  $\text{Na}_2\text{U}_2\text{O}_7$ .

It has been variously postulated that in fact a range of compounds of complex but definite formulae are precipitated (for example, by Arden and Harbutt (1949)) but it is suggested that application of co-ordination chemical theory leads to a much simplified picture of the reactions involved. In this picture the precipitate consists basically of a hydrated  $\text{UO}_3$  structure with an indefinite amount converted to a diuranate structure in a manner postulated below.

The most basic entity present in uranyl solutions is  $\text{UO}_2^{++}$  which is stated by Emeleus and Anderson (1960) to have a linear structure  $(\text{O} = \text{U} = \text{O})^{++}$ . In addition to the two oxygen atoms, it is proposed that four other positions are available around the uranium atom so that, in all, six entities may be grouped or co-ordinated around it. The uranium atom is thus said to be octahedrally co-ordinated, and this is the usual state for transition metal ions (of which uranium is one) in solution.

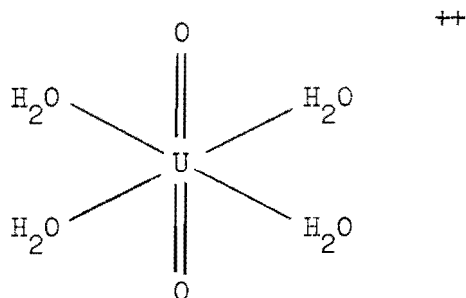
This structure and its attendant reactions are described by Emeleus and Anderson (1960, p.246-251) and it is the basic concepts so outlined which have been herein adopted.

In a strongly acid solution, the uranyl ion should persist as



which may be shown diagrammatically as:





The water molecules are arranged in a square with the uranium atom at the centre. The oxygen atoms are above and below the plane containing the water molecules. Note that the water molecules are not attached to the uranium atom by normal chemical bonds - they are said to be "co-ordinated".

During a precipitation in the plant, as the solution becomes progressively less acid, by addition of caustic soda, the co-ordinated water molecules are replaced by hydroxyl groups.

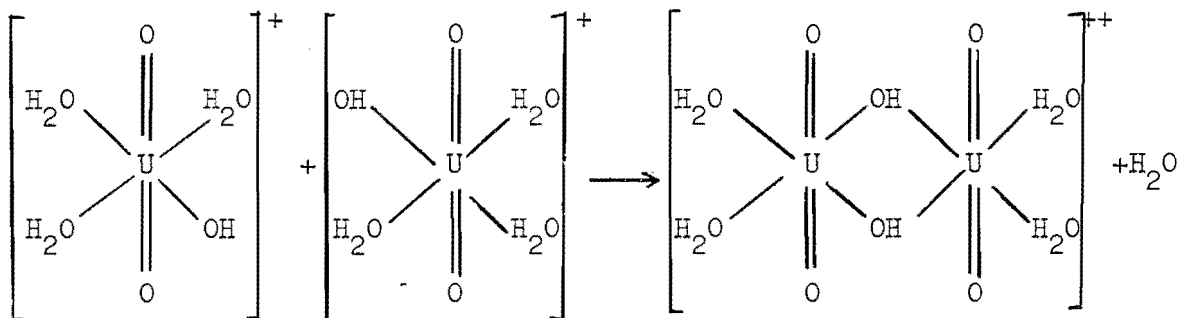


or in simpler form



This step is typical of heavy metal ions in general.

There is a strong tendency for two  $\text{UO}_2(\text{H}_2\text{O})_3(\text{OH})^+$  ions to combine by elimination of two water molecules.



or in simpler terms



The resultant is known as a diol complex and this reaction is thought to be typical of many metals other than uranium (Emeleus and Anderson, 1960). The ion owes its stability to the ability to delocalise or spread the charge associated with it.

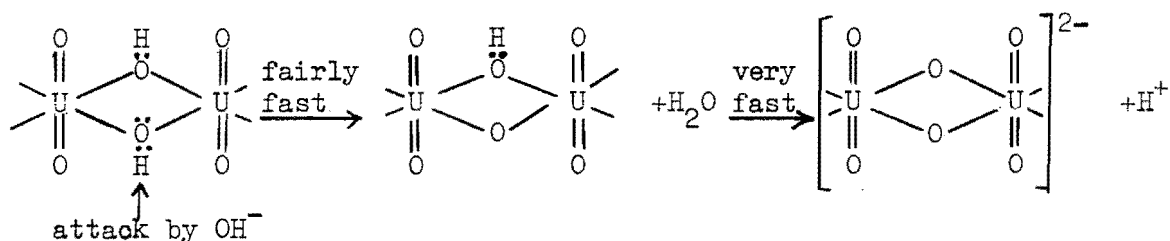
The process of "condensation" (so-called because water is produced) may hypothetically continue to any degree by steps similar to the above, as the pH is raised further, doubly charged polycations always being formed.

It may be noted that the simplified formulae of these polycations are  $UO_2^{++}$ ,  $U_2O_5^{++}$ ,  $U_3O_8^{++}$  and so on. These correspond to the formulae derived in work by Arden, Humphreys and Lewis (1948).

The limit of condensation is reached after a few such steps, however, when the cation associates with an anion or anions and precipitates from solution. At Rum Jungle it appears that the only anions directly involved are  $OH^-$  and  $SO_4^{=}$ .

Tentatively, the very mechanism that stabilises the polycation in solution can also cause the formation of a diuranate structure. Early thinking at Rum Jungle held that diuranate was formed only after a precipitate had formed, since it was known to occur with the solid material. Present thinking is that the diuranate structure can be formed in solution prior to precipitation as well as in the solid after precipitation.

It is suggested that the H on each OH bridge has a slight excess of positive charge and is more readily removed by excess  $OH^-$  to form the diuranate structure and water.



The postulated mechanism suggests that the uranyl hydroxide precipitate may have any degree of saturation with diuranate structure from zero to 100 per cent depending upon the local severity of hydroxide attack. The cation for the structure will be determined mainly by the relative concentration of various cations in the region of the diuranate structure's formation.

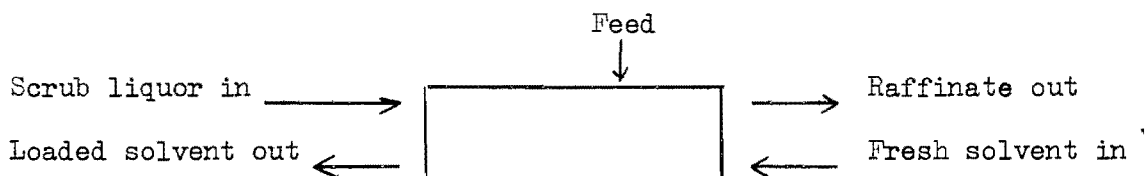
This provides further incentive to minimise the extent of local pH inhomogeneities.

#### SOLVENT EXTRACTION INVESTIGATIONS

Research on solvent extraction is currently directed towards the production of a nuclear grade product via the amine/sulphate and tributylphosphate (T.B.P.)/nitrate routes. Current work is by no means complete and it is the intention of this paper to indicate the direction in which work is proceeding.

To date, solvent extraction in the uranium industry has been carried out as a simple counter-current operation. More recent trends are towards the adoption of a system in many ways similar to distillation

with reflux. By this technique, extraction and what is known as a "scrub" are carried out as a single unit operation. Marked benefits are obtained by the use of this technique compared with the more conventional process. The contacting pattern of liquors is shown diagrammatically below. The number of theoretical stages required varies from system to system, so no attempt has been made to indicate numerical values.



In the crude aqueous feed to the extractors, conditions are maintained such that uranium loading of the organic solvent reaches a high level, thus minimising extraction of impurities. Those impurities that are extracted pass with the loaded solvent into the scrubbing section which follows immediately after the extraction system. The spent scrub liquor is fed directly into the extraction system with the crude aqueous feed.

In the scrub system, the scrub flow rate and composition are adjusted to strongly favour removal of the impurities back into the aqueous phase, whereas uranium losses are prevented by careful placement of the operating line with respect to the equilibrium line, so forming a "pinch point".

The net result is that an accumulation of both uranium and impurities occurs in the centre of the extraction-scrub unit. The impurities, by virtue of the strong scrubbing action, are forced out in the aqueous raffinate, whereas scrubbing of uranium is limited by the pinch effect so that uranium leaves in the slightly underloaded solvent.

Work has begun at Rum Jungle on the pilot scale production of high grade uranyl nitrate and sulphate solutions, using a Belgian design mixer-settler.

For such work, some knowledge is needed of the allowable impurity levels in the product. Specifications for a material probably designed for conversion to ceramic uranium dioxide fuel elements are stringent, as the following Canadian specification shows. (Table 9).

The Canadian specification is used as a preliminary guide for the work at Rum Jungle on solvent extraction.

TABLE 9Specification for Ceramic grade UO<sub>2</sub>

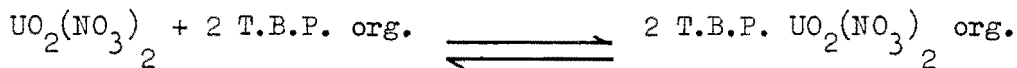
Element	Specification, ppm based on U
Aluminium	30
Boron	0.2
Cadmium	0.2
Chromium	10
Copper	10
Iron	35
Magnesium	40
Manganese	5
Nickel	15
Silicon	20
Vanadium	30
Molybdenum	1

A general outline of how solvent extraction experiments were carried out at Rum Jungle is as follows:

PRINCIPLE FOR NUCLEAR GRADE PRODUCT VIA T.B.P. EXTRACTION

In the crude aqueous feed to the extractors, nitrate content is kept high and this by mass action effect greatly enhances the extraction of uranium and impurities into the organic solvent.

(The metals form a neutral complex with T.B.P., viz:



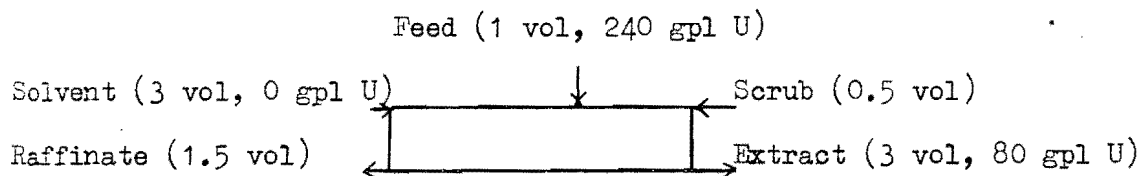
- the effect of excess NO<sub>3</sub> can readily be seen )

In the case of 20 per cent tri-n-butyl phosphate (T.B.P.) the theoretical loading is close to 90 g.p.l. U. It was preferred that loaded solvent left the mixer-settler unit at about 85 per cent of maximum loading or 75 g.p.l. U, which for an aqueous feed of 300 g.p.l. U gives an organic to aqueous flow ratio of

$$\frac{300}{75} = 4 : 1, \text{ or for 240 g.p.l. U a ratio somewhat over } 3 : 1.$$

For the T.B.P./nitrate system in general aqueous feed should be 300 g.p.l. U, and acid strength 4M. However, for this test feed was slightly less than 240 g.p.l. U after adjusting acid concentration to 4M.

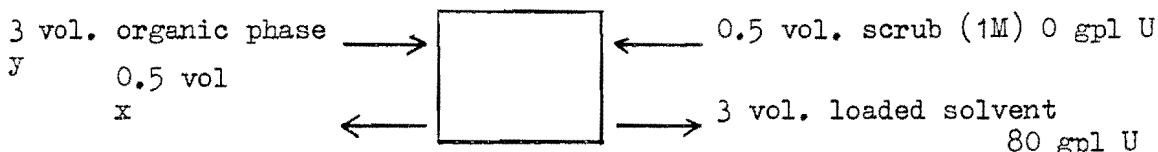
From the foregoing a picture of the set-up was.



(N.B. Scrub feed rate is typically about half feed rate.)

The next step was to examine scrub conditions to ensure that they did have a "pinch point" for uranium that limits back-extraction and prevents overloading of the extraction section with resultant raffinate losses. 1M  $\text{HNO}_3$  was chosen for scrub acidity (Wood and William, 1956).

Now, in the scrub section calculations were -



∴ equation for operating line

$$3y + 0 = 0.5x + 3 \times 80$$

$$y = 0.17x + 80$$

giving

x	0.1	1	10	100
y	80.0	80.2	80.7	97

In order to determine whether this operating line was acceptable, it was necessary to know the equilibrium data, which were available but not very accurate. Both uranium and nitric acid are extractable and their extractions are interdependent. Assuming that the nitric acid concentrations in the aqueous phase remain constant, calculations become simple (when based on nitric acid concentration). For the scrub section, the solvent was near loaded throughout, which meant that little acid was extracted to or from the organic.

On constructing the above operating line on a McCabe-Thiele diagram, it was found that it did not intersect the equilibrium line for 1M acid in the aqueous phase and hence the chosen conditions were unsatisfactory, and it was necessary to lower the scrub operating line by lowering the loading of the extract (N.B. the final flowsheet can only be tested by experiment).

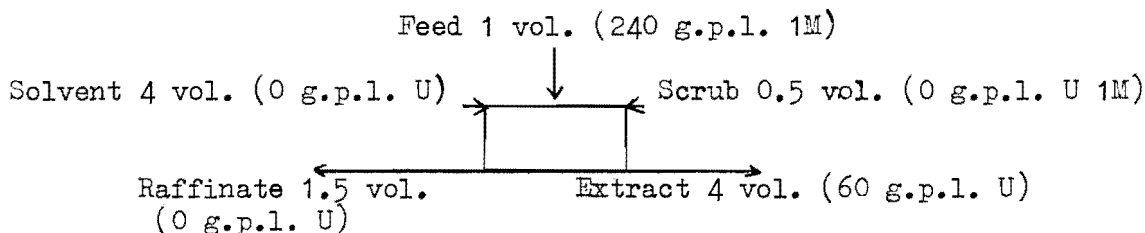
For a 4 : 1 P/A ratio, the scrub operating line became:

$$4y + 0 = 0.5x + 4.60$$

$$y = 0.125x + 60$$

This line intersected the 1M equilibrium line with about 4 - 5 g.p.l. safety factor which sufficed for the test.

Final conditions for the test were:



As the ratio of feed : organic : scrub chosen were 1 : 4 : 0.5 and the maximum throughput through the mixer-settler unit was 40 ml/min total phase flow, all flow rates were thus fixed.

Individual flows were:-

feed	$\frac{40 \times 1}{5.5}$	=	7.27 ml/min
solvent		=	29.1 " "
scrub		=	3.63 " "

As mentioned earlier, the control of the run was by means of determining the acid concentration in the organic extract and aqueous raffinate phases. The analysis log was as follows:

TABLE 10

Time	Organic extract		Aqueous U <sub>3</sub> O <sub>8</sub>		Raffinate acid
7.40 p.m.	53.6 g.p.l. U <sub>3</sub> O <sub>8</sub>	0.057M acid	2.94 g.p.l.	2.218M	
8.30 p.m.	60.3 "	.035 "	.07 "	3.53 "	
9.30 p.m.	65.9 "	.030 "	.007 "	3.23 "	
10.30 p.m.	51.7 "	.045 "	-	4.32 "	
11.15 p.m.	58.8 "	.032 "	.25 "	3.34 "	
		Aqueous feed 263	"	3.8 "	
		" scrub -		0.968"	
9.20 a.m.	64.1 " "	0.26 M acid	0.96 "	3.34 "	
10.30 a.m.	67.2 "	0.025 "	.15 "	2.98 "	
11.30 a.m.	66.9 "	.026 "	.029 "	3.02 "	
12.30 a.m.	74.1 "	.027 "	.020 "	3.04 "	
1.30 a.m.	62.6 "	.026 "	.004 "	3.35 "	
2.30 a.m.	62.8 "	.028 "	.055 "	3.16 "	
3.30 a.m.	62.1 "	.032 "	.003 "	3.04 "	

Table 10 reflects the experimental difficulties associated with the first run using the mixer-settler, which were mainly concerned with obtaining correct flow settings, in that the pumps and tubing used developed leaks. These difficulties were overcome to some extent and for the last four hours of the run conditions were fairly steady with a solvent loading of 62 g.p.l.  $U_3O_8$  (52.5 g.p.l. U), a raffinate of 0.004 g.p.l.  $U_3O_8$ , free acid in solvent 0.03M and in the raffinate 3.1M. A mass balance of  $U_3O_8$  in gives 2.0 gram/min and 1.9 gram/min out, therefore the error was a bit high. Subsequent runs gave better balance.

Table 11 lists the levels of certain impurities in the feed, raffinate and extract.

TABLE 11

Impurity	Feed		Raffinate		Extract
Na	9.5 g.p.l.	6.5 g.p.l.	(expected 6.34 g.p.l.)		2.8 p.p.m.
Cu	0.05 "	0.03 "	( " 0.03 "		0.4 p.p.m.
Fe	0.44 "	0.35 "	( " 0.29 "		7.5 p.p.m.
Ca	0.16 "	0.23 "	( " 0.11 "		0.15 p.p.m.
Cd	0.5 p.p.m.	not detected	( " 0.2 p.p.m.)		0.2 p.p.m.
Si	0.048 gpl.	0.011 g.p.l.			not detected 1 p.p.m.

Further extraction runs were carried out on the above lines, the idea being to obtain enough extract to conduct a stripping run, for the purpose of obtaining some fairly high grade strip for precipitation and ignition tests.

For the stripping run loaded solvent of about 53 g.p.l. U was stripped with acidified de-ionized water (.005M). Flow ratios were calculated to be: Organic = 1 vol; Aqueous = 0.663; and for a total phase flow of 40 ml/min. became

$$\text{Organic} = \frac{40}{1.663} = 24 \text{ ml/min.}$$

$$\text{Aqueous} = \frac{40}{1.663} \times .663 = 16 \text{ ml/min.}$$

Loaded solvent 1 vol. 53 g.p.l. U

H.G.S. 80 g.p.l.

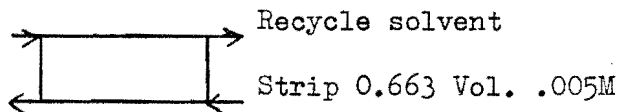


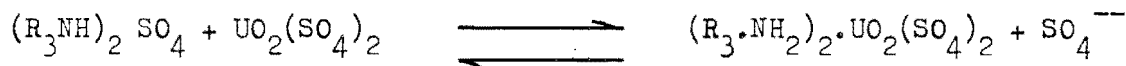
Table 12 gives the analysis log on hourly samples during the stripping run.

TABLE 12

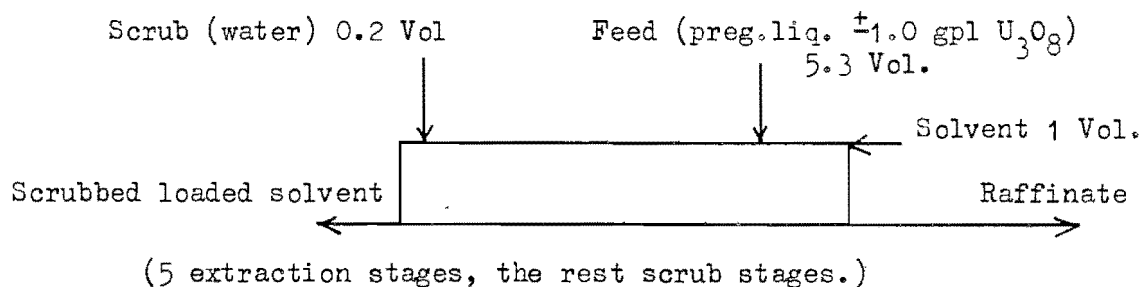
Organic (recycle solvent)			Aqueous (high grade strip)		
Sample	U <sub>3</sub> O <sub>8</sub>	Acid	Sample	U <sub>3</sub> O <sub>8</sub>	Acid
01	.03 g.p.l.	.001M	A1	89.9 g.p.l.	.061M
2	.018 "	"	2	93.1 "	.057
3	.051 "	"	3	99.2 "	.041
4	.037 "	"	4	97.9 "	.037
5	.065 "	"	5	99.2 "	.042
6	.051 "	"	6	101.6 "	.046
7	.048 "	"	7	100.5 "	.039
8	.047 "	"	8	99.2 "	.032

PRINCIPLE FOR NUCLEAR GRADE PRODUCT VIA AMINE EXTRACTION

The reaction involved in the amine-solvent extraction process is generally understood to be:-



and an amine-sulphate extraction run was carried out using treatment plant recycle solvent and pregnant liquor. The same general principles for calculations as for the T.B.P./nitrate runs hold.



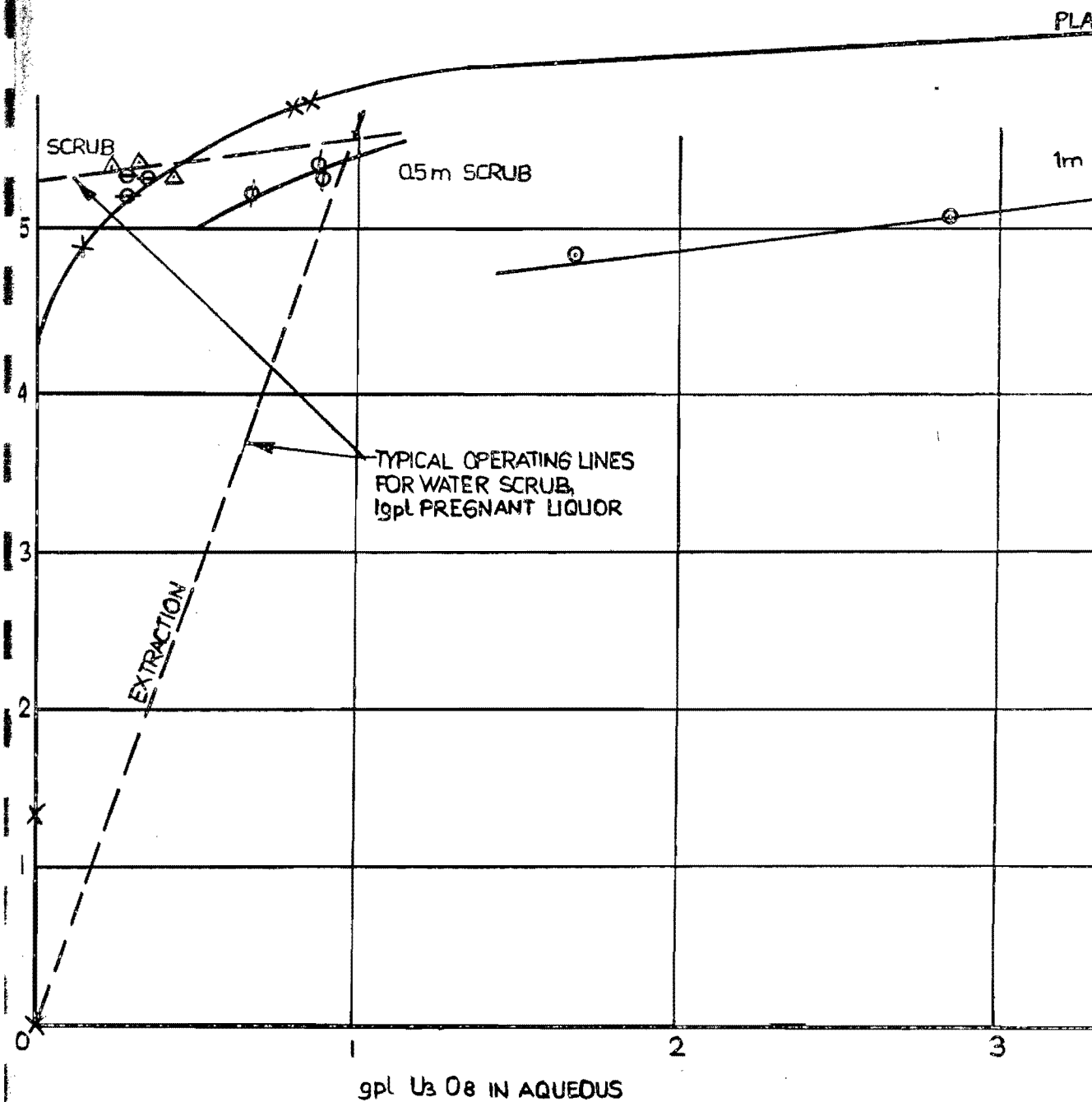
By inspection the  $\frac{A}{O}$  in the extraction circuit was chosen to give an intersection with the scrub line at about 5.5 g.p.l. U<sub>3</sub>O<sub>8</sub> in the organic for 1 g.p.l. U<sub>3</sub>O<sub>8</sub> in the aqueous.

$$\frac{A}{O} = 5.5 : 1 \text{ (extraction circuit)}$$

$$\frac{A}{O} = 0.2 : 1 \text{ (scrub circuit)}$$

∴ for 1 Vol. solvent, have 5.3 Vol. feed  
0.2 Vol. scrub





### LEGEND:

DATA FROM PLANT.

EQUILIBRIA DATA, 1m  $H_2 SO_4$  SCRUB

" 0.5m  $H_2 SO_4$  SCRUB

" 0.2m  $H_2 SO_4$  SCRUB

" 0.0m  $H_2 SO_4$  SCRUB

Fig 6. Scrub equilibria on plant pregnant liquor and solvent.

and total flow in the extraction circuit = 6.5 Vol.

Individual flows were:

$$\begin{array}{rclcl}
 \text{Feed} & \frac{40}{6.5} & \times & 5.3 & = & 32.62 \text{ ml/min.} \\
 \text{Solvent} & \frac{40}{6.5} & \times & 1 & = & 6.15 \text{ " " } \\
 \text{Scrub} & \frac{40}{6.5} & \times & 0.2 & = & 1.23 \text{ " " }
 \end{array}$$

During this run, which served as a preliminary try out for obtaining high grade uranyl sulphate solution, it was observed that the mixer-settler unit could be made to start off with organic-continuous mixing either by prefilling with solvent or by raising the impellers high after about 50/50 solvent/aqueous was present, then slowly lowering them to the correct position. In either case the system soon reverted to aqueous-continuous, with its attendant solvent losses in the raffinate. (To date no analysis results are available for the amine/sulphate run and although this is disappointing, this paper's main aim is to make known the type of work being carried out at Rum Jungle.)

Figure 6 shows the typical operating line for a water scrub, and a pregnant liquor of 1 g.p.l.  $\text{U}_3\text{O}_8$ . It can be seen from the diagram that if a 1M  $\text{H}_2\text{SO}_4$  scrub was used, the solvent loading would have to be reduced to  $\pm 4.5$  g.p.l.  $\text{U}_3\text{O}_8$ .

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