DEPARTMENT OF MINES.

South Australia.

-RESEARCH AND DEVELOPMENT BRANCH-

REPORT No. R.D. 63.

EXTRACTION OF RUTILE FROM LEACH RESIDUES OF RADIUM HILL CONCENTRATE.

bv

G.D. Sheridan.

OBINALIA OFICIANED

Copy No.22 of .25 copies.

This document consists of 10 pages.

DATE: June, 1957.

EXTRACTION OF RUTILE FROM LEACH RESIDUES. OF RADIUM HILL CONCENTRATE.

-Contents-

	Pag	ge•
	Abstract,,,	1.
	Summary	
2.	Introduction	2.
3.	Material Examined	2.
4.	Equipment Used	3.
5•	Experimental Procedure and Results.	3.
6.	Cost Estimate	9•
7.	Discussion	9.

EXTRACTION OF RUTILE FROM LEACH RESIDUES OF RADIUM HILL CONCENTRATE.

-Abstract-

The residue obtained from leaching Radium Hill flotation concentrates contains rutile, haematite, ilmenite, silicates, and hydrolysed titania. Simple size classification followed by magnetic separation and flotation produced products containing 50 and 70 percent TiO₂ and representing overall TiO₂ recoveries of 14 and 44 percent respectively. The cost of a plant to produce these products is shown.

1. SUMMARY.

 \mathfrak{V}

The uranium content of the flotation concentrates from Radium Hill is leached with sulphuric acid. The leached residue, which constitutes over 90 percent by weight of the original flotation concentrate, contains rutile, ilmenite, haematite, silicates, sulphides and hydrolysed titania. The total TiO₂ content is 48.5 percent. This report describes experimental work aimed at recovering the rutile and ilmenite from the residue.

The granular fraction of the residue was separated by a simple size classification treatment involving a spiral classifier and a hydraulic cyclone, the reject from this treatment containing most of the fine slime and hydrolysed titania.

A concentrate containing 91.5 percent TiO₂ was produced from the classifier sand fraction by magnetic separation followed by flotation, with a recovery of only 9.4 percent of the TiO₂ in the residue.

Magnetic separation of the classifier sand, followed by flotation of the combined non-magnetics from the classifier sand together with granular cyclone fractions produced a concentrate containing 70.2 percent TiO₂ and representing a recovery of 44.3 percent of the TiO₂ content of the leach residue. A magnetic fraction — containing 51.4 percent TiO₂ and representing a TiO₂ recovery of 14.4 percent was produced as a by-product.

The cost of a plant to produce these products is estimated to be approximately £160,000, including a six month working capital allowance of £45,000. The operating cost is calculated to be £4.10.0. per ton of feed or approximately £15 per ton of rutile product. This assumes the "ilmenite" product to be unsaleable.

2. INTRODUCTION.

The residue from leaching Radium Hill uranium concentrates contains up to 50 percent titanium dioxide which is equivalent to ilmenite in TiO₂ content. It cannot be used for the usual pgiment manufacture by solution in sulphuric acid, since it consists mainly of rutile finely intergrown with haematite. The material also contains some ilmenite, siliceous minerals and finely divided titania precipitated during leaching.

Attempts to separate a rutile rich fraction are described in this report.

3. MATERIAL EXAMINED.

The work was carried out on approximately 15 tons of residues from the leaching of Radium Hill concentrates on a pilot plant scale. This material has been designated "Chemical Plant Residue".

The material contained 48.5 percent TiO2. Mineralogical examination was confined to the granular fractions after classification, as the original material contained a large amount of slime. The mineralogical composition of the classifier sands fractions and the 3 inch hydraulic cyclone underflow fractions were similar, being reported as follows:

Rutile		percent	weight.
Davidite (skeletal)	5.4	11	11
Haematite. Ilmenite.	15.3 6.8	14	Ħ
Sulphides (Pyrite)	1.0	17,	11
Silicates.	11.3	t!	11

4. EQUIPMENT USED.

- (a) 12 inch spiral classifier.
- (b) 3 inch diam. 120 hydraulic cyclone.
- (c) 1.2 inch diam. 200 hydraulic microcyclone.
- (d) Laboratory induced roll type magnetic separator.
- (c) Laboratory batch Fagorgren flotation cell.
- (f) Ancillary pumping and mixing equipment.

5. EXPERIMETNAL PROCEDURE and RESULTS.

5.1 Laboratory Test Work.

The essential details and final conclusions of each phase of testing are shown in the following sub-sections.

5.1.1 Size classification.

Owing to the large amount of slime material in the leached residue, an initial size classification was carried out in the following manner.

The residue was fed as a pulp to a spiral classifier from which a sand fraction was obtained. The classifier overflow was then pumped through a three inch hydraulic cyclone to produce an underflow product and overflow material which was pumped through a 1.2 inch hydraulic microcyclone to produce underflow and overflow products.

The weight percentage, TiO₂ content and percentage distribution of TiO₂ for each of the four final fraction are shown in Table 1.

TABLE 1.

Details of Products of Size Classification.

Product.	Percent Weight.	TiO2 Percent.	Percent Distribution TiO ₂
Classifier sand. 3" cyclone U/F. Microcyclone U/F. " O/F.	20 .6 38.8 14.1 26.5	58.0 55.6 38.8 35.5	24.7 44.6 11.3 19.4
FEED.	100,0	48 . 5	100.0

Although fine in size the first two fractions consisted mainly of granular material, whereas the microcyclone fractions were comprised of slime material with most of the titanium being present as hydrolysed titania.

All further work therefore was centred around recovery of titanium oxide from the classifier sand and three inch cyclone underflow products.

5.1.2 Gravity Concentration.

Little success was achieved by gravity concentration methods, this being mainly due to the fact that there is no significant difference in specific gravity of the minerals present.

5.1.3 Magnetic Separation.

Dry magnetic separation tests were made on the classifier sand fraction using an induced roll high intensity magnetic separator. Several trials were made to establish the best conditions for producing a clean non-magnetic tailing containing the rutile, silicates and sulphides, from which a rutile concentrate could be produced by flotation using AGC 825 in an acid circuit.

Typical results obtained are shown in Table 2.

TABLE 2.

Magnetic Separation of Classifier Sand Product.

	Percent Weight of mag- of orig. netic. G.P.R.* feed.		Percent	Percent Distrib. TiO2		
Fraction.			TiO ₂	of mag- of orig. netic C.P. R. feed.		
Magnetics.	66.0.	13.6	51.0	58.0	14.3	
Non-Magnetics.	34.0	7.0	72.0	42.0	10.4	
FEED.	100.0	20.6	58.0	100.0	24.7	

■ C.P.R. signifies "Chemical Plant Residue".

5.1.4 Roasting.

Reducing roast tests were carried out on both the classifier sand and cyclone underflow products using Leigh Creek coal as the reducing agent. The object of these tests was to selectively convert the haematite to the strongly magnetic state in order to remove it by low intensity magnetic separation. The tests were not successful as the reducing roast also increased the magnetic susceptibility of the ferruginous rutile.

5.1.5. Flotation.

Flotation tests were carried out on the non-magnetic fraction of the classifier sand product, described in section 5.1.3. The procedure consisted of first floating the sulphides with xanthate and a frother. Various reagents were used in an attempt to float the silicates but all the reagents tested left the coarse silica in the non-floated rutile. Floating the rutile from depressed silicates was more successful. The best results obtained are shown in Table 3.

TABLE 3.

Flotation of Classifier Sand Non-Magnetic
Fraction.

	Percent Weight.			Percent Distribution TiO ₂	
Fraction.	Flotation Feed.	Original C.P.R.	TiO ₂	Flotation Feed.	Original C.P.R.
Rutile Conc. Sulphide Conc) Tailing.	71.0 29.0	5•0 2•0	91.5 23.5	90.0 10.0	9•4 1•0
FEED.	100.0	7.0	72.0	100.0	10.4

Flotation tests were also carried out on combined classifier sand and cyclone underflow products. Typical results using the most suitable reagent combination are given in Table 4.

TABLE 4.

Flotation of Combined Classifier Sand and Cyclone U/F Products.

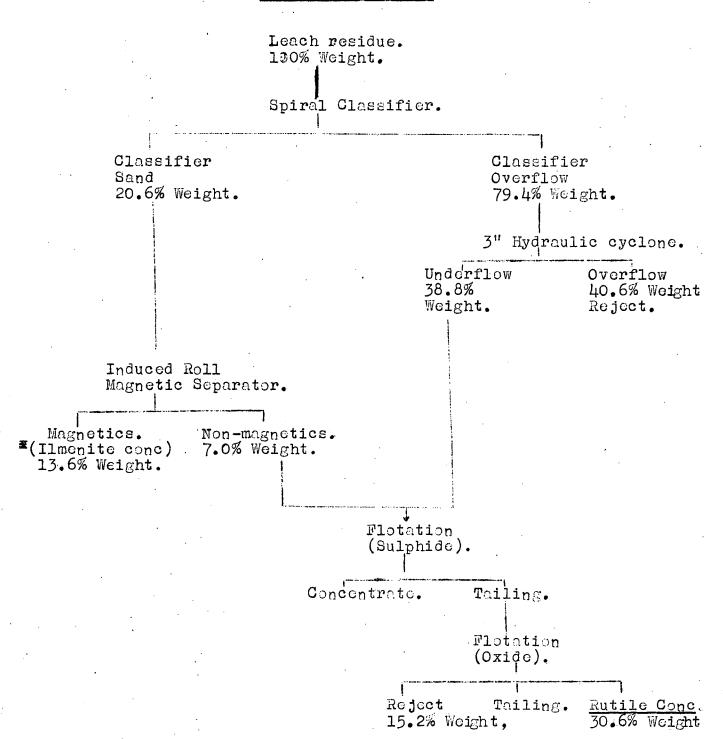
Fraction.	eaction. Percent Weight.		Domoout	Percent Distribution TiO ₂	
	Flotation Feed.	Original C.P.R.	Percent	Flotation Feed.	Original C.P.R.
Concentrate. Tailing.	84.2 15.8	50.0 9.4	62.0 27.6	92.2 7.8	63 . 9 5 . 4
FEED.	100.0	59•4	56.6	100.0	69.3

5.2 Selection of Flowsheet.

Based on the results obtained in Section 5.1 the following flowsheet was drawn up. The flowsheet assumes that a market can be found for "ilmenite" and "rutile" products containing approximately 50 percent and 70 percent TiO₂ respectively.

-7-

PROPOSED FLOWSHEET.



mFinal Products.

5.3 Results of Flowsheet.

5.3.1 Material Balance.

Table 5 shows a material balance, titanium grades and distributions for a parcel of residue treated in accordance with the flowsheet.

-8-

TABLE 5.

Material Balance and Titanium Distribution.

Fraction.	Percent. Weight.	Percent TiO ₂	Percent Distribution. TiO ₂
Magnetic product. Rutile Product. Flotation Rejects. Cyclone O/F.	13.6 30.6 15.2 40.6	51.4 70.1 33.7 36.6	14.4 44.3 10.6 30.7
FEED.	100,0	48.5	100.0

5.3.2 Details of Final Products.

Screen and chemical analyses of the rutile and ilmenite concentrates are given in Tables 6 and 7 respectively.

TABLE 6.
Screen Analyses of Rutile and Ilmenite Concentrates.

	Percent Weight.				
Mesh (Tyler).	Rutile Concentrate	Ilmenite Concentrate.			
+ 65. - 65 + 100. -100 + 150. -150 + 200. -200 + 325. -325.	0.4 1.6 4.3 6.1 25.4 62.2	2.2 11.9 24.5 20.7 31.5 9.2			
FEED.	100.0	100.0			

TABLE 7.

Chemical Analyses of Rutile and Ilmenite Concentrates.

Common and the out	Percent.	Percent.		
Composition.	Rutile Concentrate.	Ilmenite Concentrate		
TiO ₂ Fe ₂ O ₃ . Total Fe. SiO ₂ Cr. S. Al ₂ O ₃ Pb. V. Zn Mg. Ca. U ₂ O ₈	70.1 N.D. 16.7 2.0 0.20 0.16 0.75 0.03 0.45 0.10 0.04 0.08 1.6 lb/ton.	51.4 45.4 N.D. 1.8 0.14 N.D.		

N.D. denotes "note determined".

6. COST ESTIMATE.

A cost estimate has been prepared for a plant operating 3 shifts a day, for 7 days a week, and a throughput of 18,000 tons a year.

The capital cost of such a plant is estimated to be approximately £160,000. This cost includes a working capital of £45,000 to cover the operating costs for six months operation. The operating cost is calculated to be £4.10.0 per ton of feed to the plant, or approximately £15 per ton of rutile concentrate, any value of the magnetic product being disregarded.

The capital cost does not include administration, maintenance, buildings, or equipment required in these buildings. No allowance is made for spare parts or duplication of equipment. The cost of land, fresh water supply and electric power supply is not included.

As the tailing from this plant would be approximately 95 percent minus 300 mesh it could be disposed of by pumping the material out to sea. Sea water could be used for most of the plant requirements.

To reclaim the present tailing dump, more equipment costing approximately £20,000 would have to be purchased. For the purpose of this cost estimate it has been assumed that this material would not be treated concurrently with current material from the uranium treatment works.

7. DISCUSSION.

The two materials used as a source of titanium are ilmenite for pigment manufacture and rutile for metal manufacture. They have not yet become competitive in the metal field, but when they do, the difference in price will be the cost of iron removal. This higher cost will be the results of increased reagent requirements, and the more complicated plant that is

CONFIDENTIAL.

-70-

necessary. At this time it will be possible to use materials containing 70 percent TiO₂, and to quote a price in which penalties for contained ore are known. It may then be possible to readily sell the products of Pt. Piric Chemical Plant residue at a price higher than those indicated production costs.