V.F. Harrison

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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 58-90

PRELIMINARY TESTING OF URANIUM ORE (Ref. No. 1/58-9) FROM KLERKSDORP CONSOLIDATED GOLDFIELDS LIMITED, SOUTH AFRICA, SUBMITTED BY WRIGHT ENGINEERS LIMITED, VANCOUVER, B. C.

by

H. W. SMITH and V. F. HARRISON

RADIOACTIVITY DIVISION

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ABSTRACT

Preliminary physical concentration test work indicated that differential grinding concentration would be applicable, rejecting 56% of the weight with a loss of 7% of the uranium. Autooxidation pressure leaching gave extractions of 85% at 150 psig and 150°C. Acid leaching at room temperature and atmospheric pressure was not satisfactory.

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(21 pages, 12 tables, 2 figures)

INTRODUCTION

A 96 lb sample of crushed uranium ore (our reference No. 1/58-9) was received on January 22, 1958, by air freight, from Klerksdorp Consolidated Goldfields Limited, South Africa, for investigational test work, with particular reference to autooxidation pressure leaching. The request for the work arose out of discussions held during the Commonwealth Mining Congress meeting in Ottawa in 1957, and was channelled through Wright Engineers Limited, of Vanvouver, B.C.

Considerable test work had already been carried out on this ore by the South Africans (1) (2) which indicated that it was fairly refractory and required large amounts of acid to obtain a high extraction, but that it would respond to concentration by differential grinding. In their covering letter, dated March 12, 1958, Wright Engineers indicated that the test work should explore the possibilities of producing (a) a low grade concentrate by grinding and/or gravity separation for subsequent leaching, or (b) a high grade product suitable for direct sale to the Combined Development Agency or to the U.K. Atomic Energy Authority.

With the above factors in view, and since the sample was limited, the major part of the test work was planned to check the amenability of this sample to grinding concentration techniques, and to pressure autooxidation leaching. Preliminary gravity concentration and flotation tests were also carried out.

The results of this test work will be used to plan a more comprehensive test program, to be carried out on a larger sample of the same ore (which is now on hand).

SUMMARY

1. By differential grinding in a laboratory size ball mill it was possible to produce a grind that was 56% plus 48 mesh, with the minus 48 mesh fraction containing 92.5% of the uranium.

2. Autooxidation pressure leaching on the whole ore gave an extraction of 86%. On the grinding concentrate, extractions of 85% were obtained, with an overall recovery in solution, from the whole ore, of 76%.

3. Acid leaching, at a controlled pH of 1.5 at room temperature and atmospheric pressure, extracted 46% of the uranium in 48 hours, and consumed 66 lb H_2SO_4 per ton and 20 lb MnO_2 per ton. Strong acid leaching, with 200 lb 100% H_2SO_4 and 10 lb MnO_2 per ton, at 45°C and atmospheric pressure, gave an extraction of 71% in 48 hours. Pug leaching with 200 lb H_2SO_4 per ton gave an extraction of 69% in 48 hours.

4. Preliminary gravity concentration tests indicated that a commercial-grade product could not be produced by such methods. It may be possible to concentrate the coarser sizes by sink-float, since on the size -1/2 in + 10 mesh, 36% of the weight was rejected as float at 2.75 S.G., with a loss of 5% of the uranium. This point will be rechecked on the new sample.

5. By bulk fatty acid flotation, a product made up of flotation concentrate plus slimes, containing 40% of the original weight and 80% of the uranium, was produced.

ORE ANALYSES

A head sample cut from the crushed ore was assayed radiometrically, chemically and spectrographically; the results are shown in Tables 1 and 2.

TABLE 1

Results of Chemical Analyses on Head Sample
(Ref. No. 1/58-9)

(Chem. Lab. Number: RD 3962
Specific Gravity of Ore: 2.78)

Assayed for	%
U_3O_8, chemical	0.33
U_3O_8, radiometric	0.34
U_3O_8, secondary *	0.04
CO_2, by combustion	0.76
CO_2, by acid decomposition and evolution	0.33
Fe	7.6
S, total	2.35
S, sulphate	0.10
Mo	0.001
V	<0.03
ThO_2	0.18
Ti	0.65
As	0.05
P_2O_5	0.68
Rare Earth oxides	1.40
Au	0.045 oz/ton
Ag	0.08 oz/ton

* A sample is leached for 10 minutes in a hot 10% solution of Na_2CO_3. The uranium dissolved is taken as an indication of the secondary uranium present.

TABLE 2

Results of Semi-quantitative Spectrographic Analyses on Head Sample, Ref. No. 1/58-9

(Assays in percent)

Si	20	Ti	0.6	Dy	0.02
Fe	5	Zr	0.3	Gd	0.03
Al	5	Cu	0.15	Yb	0.03
Mn	0.9	Nb	0.1	Sn	0.02
Mg	0.8	Ni	0.1	Cr	0.07
As*	0.2	La	0.4	Be	<0.001
P*	0.2	Ce	1.5	B	0.003
Pb	0.5	U*	0.8		
Ta*	0.3	Th	0.4		
Cu	0.2	Co	0.03		

* Identification not positive.

SAMPLING PROCEDURE

The size of the sample as received was approximately minus 1 inch. It was crushed to minus 1/2 in. and riffled into four quarters. One quarter was crushed to minus 20 mesh and the sample for head analysis was riffled out. The head sample was pulverized to all minus 100 mesh for assay. The remaining three quarters were retained separately for test work, and the test portions were obtained by riffling.

MINERALOGY

The complete mineralogy is reported in Mines Branch Investigation Report IR 58-48 (3). The abstract of this report is as follows:

> "Radioactivity occurs in finely disseminated form in the matrix of the pebble conglomerate rock. The grains of uraninite, the main uranium-bearing mineral, are usually less than 200 mesh in size. Zircon, coffinite and monazite account for a small proportion of the radioactivity."

DETAILS OF TEST WORK, AND RESULTS

The test work included preliminary gravity concentration, flotation, differential grinding, acid leaching at controlled pH, strong acid leaching, pug leaching, and autooxidation pressure leaching.

1. Gravity Concentration Test Work

The crushed minus 1/2 in. ore was sized and the screen analysis is given in Table 3. Sink-float concentration tests were applied to the plus 20 mesh sizes, and superpanner concentration tests to the minus 48 plus 100 mesh sizes. In the sink-float tests, a heavy liquid of specific gravity 2.75 (tetrabromoethane diluted with carbon tetrachloride) was used as a separating medium. The results of the gravity concentration tests on the sizes treated are given in Table 4.

TABLE 3

Screen Analyses of Ore Crushed to Minus 1/2 Inch

Fraction	% Wt	Assay, % U_3O_8 (gamma)	Dist. U_3O_8, %
-1/2 in. + 1/4 in.	42.4	0.18	23.6
-1/4 in. + 10 mesh	25.0	0.22	16.6
-10 mesh+ 14 mesh	4.1	0.18	2.3
-14 " + 20 "	4.8	0.21	3.1
-20 " + 28 "	4.1	0.28	3.5
-28 " + 35 "	3.3	0.35	3.5
-35 " + 48 "	3.1	0.40	3.8
-48 " + 65 "	2.2	0.40	2.7
-65 " + 100 "	2.4	0.57	4.2
-100 " + 150 "	2.2	0.77	5.2
-150 " + 200 "	0.8	1.00	2.5
-200 "	5.6	1.67	29.0
Head (calc.)	100.0	0.32	100.0

TABLE 4

Gravity Concentration Tests on Sized Fractions

Fraction	% Weight		Assay, % U_3O_8 (gamma)	Distribution of U_3O_8, %	
	Fraction	Overall		Fraction	Overall
-1/2 in. + 4 mesh*					
Sink	48.2	20.4	0.32	85.6	20.2
Float	51.8	22.0	0.05	14.4	3.4
	100.0	42.4	0.18	100.0	23.6
-4 mesh+10 mesh*					
Sink	43.0	10.8	0.45	89.6	14.9
Float	57.0	14.2	0.039	10.4	1.7
	100.0	25.0	0.22	100.0	16.6
-10 mesh+14 mesh*					
Sink	41.0	1.7	0.39	87.0	2.0
Float	59.0	2.4	0.04	13.0	0.3
	100.0	4.1	0.18	100.0	2.3
-14 mesh+20 mesh*					
Sink	45.5	2.2	0.42	89.7	2.8
Float	54.5	2.6	0.04	10.3	0.3
	100.0	4.8	0.21	100.0	3.1
-20 mesh+28 mesh**	100.0	4.1	0.28	100.0	3.5
-28 mesh+35 mesh**	100.0	3.3	0.35	100.0	3.5
-35 mesh+48 mesh**	100.0	3.1	0.40	100.0	3.8
-48 mesh+65 mesh***					
Tip	4.2	0.1	0.63	6.5	0.2
Middling	8.3	0.2	0.6	12.3	0.3
Tailing	87.5	1.9	0.37	81.2	2.2
	100.0	2.2	0.40	100.0	2.7
-65 mesh+100 mesh***					
Tip	2.0	0.05	1.10	3.8	0.2
Middling	4.4	0.1	0.83	6.3	0.3
Tailings	93.6	2.25	0.55	89.9	3.7
	100.0	2.4	0.57	100.0	4.2
-100 mesh+150 mesh**	100.0	2.2	0.77	100.0	5.2
-150 mesh+200 mesh**	100.0	0.8	1.00	100.0	2.5
-200 mesh**	100.0	5.6	1.67	100.0	29.0
Overall Head (Calc.)		100.0	0.32		100.0

* Treated by sink-float at sp. gr. 2.75.
** Untreated.
*** Treated by superpanning.

2. Flotation Test Work

Two preliminary flotation tests were done to check the possibilities of fatty acid flotation as a U_3O_8 concentration step. The ore was ground to 50% minus 200 mesh, deslimed using 0.5 lb sodium silicate per ton, and then floated using 0.8 lb Acintol FA-2* per ton, stage added. The frother was Dowfroth 250. Table 5 gives the results of one test.

* Acintol FA-2 is a tall oil product supplied by Charles Albert Smith Ltd., Montreal, and manufactured by the Arizona Chemical Company, Inc., New York 20, N.Y.

TABLE 5

Fatty Acid Flotation Test

Product	% Wt	Assay, % U_3O_8	Dist. U_3O_8, %
No.1 conc	5.3	1.01	17.2
No.2 conc	5.0	0.90	14.4
No.3 conc	16.0	0.40	20.4
Tailing	59.7	0.10	19.2
Slimes	14.0	0.64	28.8
	100.0	0.31	100.0

3. Differential Grind Concentration Tests

Since, according to previous reports, concentration by differential grinding had appeared promising on other samples of this ore, a number of tests were made to check the method on this sample, and also to produce concentrate for leach tests. The ore was tested with various types of laboratory grinds and, generally speaking, it was found that, after grinding, the plus 48 mesh fraction could be rejected as waste with minimum grades, and the minus 48 mesh fractions could be considered concentrate. Table 6 gives results of various grinds and Table 7 shows a more complete screen analysis on one test.

TABLE 6

Differential Grinding Tests

Test No.	Type of Grind	Concentrate			Reject		
		% Wt	Assay, % U_3O_8	Dist. U_3O_8,%	% Wt	Assay, % U_3O_8	Dist. U_3O_8, %
9 B	Steel balls 2000g, dry ore 1000g, 20 min, -4 mesh feed.	44.0	0.67	92.6	56.0	0.041	7.4
5	Steel balls 2000 g, dry ore 1000 g, 20 min, -4 mesh feed.	44.0	0.67	92.3	56.0	0.044	7.7
9A	Steel balls 2000g, dry ore 1000 g, 20 min, -1/2 in. feed.	38.8	0.70	83.4	61.2	0.088	16.6
11	Rods 2700 g, dry ore 1000 g, 20 min, -4 mesh feed.	50.5	0.58	91.8	49.5	0.002	8.2
12	Rods 2700 g, dry ore 1000 g, 10 min, -1/2 in feed.	32.3	0.70	81.9	67.7	0.08	18.1
17A	Pebbles 2000g, dry ore 1000 g, 5 min, -4 mesh feed.	45.4	0.62	90.6	54.6	0.055	9.4
20	Attrition grind on +48 mesh in centrifugal mill, followed by 15 min in ball mill on +48 mesh fraction.	34.4	0.83	88.2	65.6	0.059	11.8

TABLE 7

Screen Analyses of Ground Ore, Test 9B

Mesh Size	% Wt	Assay, % U_3O_8	Dist. U_3O_8, %
+10	25.1	0.041	3.3
-10+48	30.9	0.041	4.1
-48+65	4.5	0.087	1.3
-65+100	5.3	0.11	1.9
-100+200	10.4	0.38	12.8
-200	23.8	1.00	76.6
Head (calc.)	100.0	0.31	100.0

4. Acid Leach Tests

Beaker scale sulphuric acid leach tests were made on the ore to check the responses to controlled pH leaching at room temperature and to strong acid leaching at 45°C. The ore was ground to 57% minus 200 mesh and leached at 60% solids for 72 hours. Manganese dioxide was used as the oxidizing agent. In the so-called strong acid leaches, 200 lb H_2SO_4 per ton was added, either all at once at the start of the leach, or in two stages (at the beginning of the leach, and 24 hours after the first addition). This amount of acid was about four times that required in the controlled pH leaches. The test data and results of this work are shown in Tables 8 and 9.

TABLE 8

Acid Leach Test Data

Test No.	35	37	36	38
Ore charge, g	1000	1000	1000	1000
Acidity	pH, 1.5	pH, 1.5	strong	strong
Acid added, lb/ton	50	66	200(in 2 stages)	200
MnO_2 added, lb/ton	10	20	10	10
Residue Assays,% U_3O_8				
6 hr	0.19	-	0.16	-
24 hr	0.17	0.20	0.16	0.092
48 hr	0.18	-	0.12	0.098
72 hr	0.18	0.18	0.12	0.097
Leach Solution Assays				
U_3O_8, g/1	2.41	2.32	2.66	3.61
pH	1.5	1.5	1.2	1.0
Free acid, g/1	-	-	-	9.0
Fe tot, g/1	7.7	7.8	20.2	25.7
Fe^{+2}, g/1	4.1	3.8	13.7	18.7

TABLE 9

Results of Acid Leach Tests

Test No.	35	37	36	38
Leach Solution				
Volume, ml	455	530	430	440
U_3O_8, g/1	2.41	2.32	2.66	3.61
Wash Solution				
Volume, ml	745	750	765	710
U_3O_8, g/1	0.40	0.40	0.80	0.91
Residue				
Weight, g	969	988	975	941
U_3O_8 assay, %	0.18	0.18	0.12	0.097
Calc. head, % U_3O_8	0.31	0.33	0.32	0.32
U_3O_8 ext'n, %	44.4	46.3	63.6	71.1
ThO_2 in residue, %	0.15	0.13	0.14	0.12
ThO_2 ext'n, %	17	27	22	33

5. Pug Leach Tests

Pug leach tests were made by mixing moist concentrate with concentrated (93%) sulphuric acid and allowing it to stand for 24 and 48 hours. The pulp was then diluted to 60% solids for 10 minutes, filtered, and washed (see Table 10).

TABLE 10

Pug Leach Tests on the Grinding Concentrate

Test No.	4-15A	4-15B	4-16A	4-16B
Charge, g	125	125	125	125
Moisture, %	15	15	15	15
Acid added, lb/ton	100	100	200	200
Max. pulp temp, °C	40	40	55	55
Leach time, hr	24	48	24	48
Leach Solution Assays After Dilution				
Vol, ml	130	130	120	120
U_3O_8, g/l	1.04	1.25	1.69	1.47
Fe tot, g/l	4.64	4.70	8.16	8.4
Fe^{+2}, g/l	1.15	0.62		
pH	2.3	2.3	1.5	1.5
Residue				
Wt, g	50.1	51.5	48.0	47.3
* U_3O_8 assay (tot), %	0.27	0.29	0.19	0.18
Calc. head, % U_3O_8	0.54	0.60	0.60	0.56
U_3O_8 ext'n, %	50	52	69	68

* Only 3-4 % of the total uranium in the final residue was secondary or soluble uranium. See footnote to Table 1.

6. Autooxidation Pressure Leach Tests

Pressure leach tests were carried out on the whole ore and the grinding concentrate, in a 2 litre No. 316 stainless steel autoclave (see Figure 1). The pulp was agitated by a central stirrer and aerated by means of an injection pipe discharging close to the impeller.

The tests were carried on for periods of from 6 to 20 hours. The ore slurries were sampled at intervals during the test to determine the extraction rate. These samples were filtered and washed; twice with 1/4% acid and once with water.

The results are shown in Tables 11 and 12, and the uranium extraction curves are shown in Figure 2.

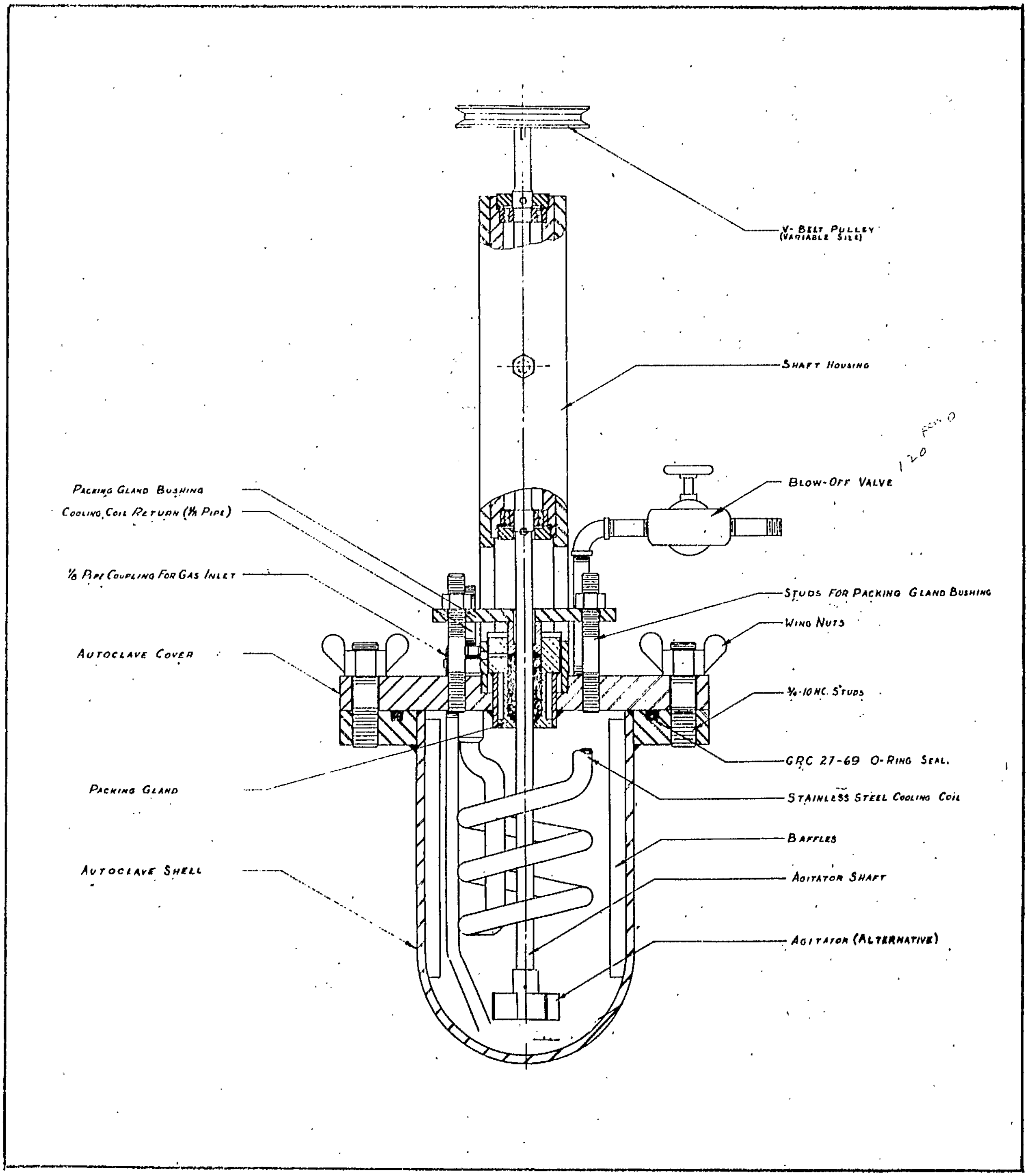

FIGURE 1

LABORATORY STAINLESS STEEL PRESSURE AUTOCLAVE

TABLE 11

Pressure Leach Tests on Whole Ore

Test No.	4-14	8-700	8-701	8-702	8-703	8-704	8-706
Feed assay, % U_3O_8	0.31	0.28	0.30	0.29	0.33	0.33	0.30
Screen analyses, % -200 mesh	59	72	77	77	77	84	83
Leach Conditions							
Aerating gas	air	air	air	air	oxygen	oxygen	oxygen
Pressure, psig	135	200	175	150	100	168	200
Temperature, °C	150	150	150	150	150	175	175
Ore charge, g	1000	1000	1000	1000	1000	875	900
Air flow, cc/min/kg	2000	2000	2000	2000	500	2000	2000
Pulp density, % solids	60	50	50	50	50	50	50
Leach Solution Assays							
pH	1.45	1.4	1.20	1.25	1.43	1.30	0.95
U_3O_8, g/l	3.5	3.3	3.6	3.30		3.66	3.70
Fe^{+3}, g/l	1.1	1.7	0.2	2.1			
Fe^{+2}, g/l	1.7	0.6	1.4	1.1			
Residue Assays, U_3O_8%							
2 hr	0.095(1 hr)	0.067	0.069	0.055	0.064	0.046	0.047
4 hr	0.066	0.050	0.050	0.047	0.052	0.043	0.043
6 hr	0.058	0.049	0.051	0.044	0.047	0.049	0.040
U_3O_8 ext'n, %	81.2	82.5	83.0	84.8	85.8	85.0	86.6

TABLE 12

Pressure Leach Tests on Grinding Concentrate

Test No.	4-19	4-21	4-25	8-705
Feed assay, % U_3O_8	0.60	0.72	0.64	0.55
Screen analyses, % -200 mesh	74	69	69	87
Leach Conditions				
Aerating gas	air	air	air	oxygen
Pressure, psig	140	140	200	165
Temperature, °C	145	150	150	175
Ore charge, g	1090	1215	943	950
Air flow, cc/min/kg	2000	4000	2000	2000
Pulp density, % solids	65%-30%* (Dilution increased during test)	50	50	50
Leach Solution Assays				
pH	1.35	1.5	1.45	1.30
U_3O_8, g/l	2.4 ***	8.1	6.8	5.76
Fe^{+3}, g/l	1.1 (0.7 at 6 hr)	3.1 (1.5 at 4 hr)	2.5 (3.5 at 4 hr)	0.6
Fe^{+2}, g/l	0.1 (0.6 at 6 hr)	0.6 (5.1 at 4 hr)	0.7 (2.8 at 4 hr)	
Residue Assays, U_3O_8%				
2 hr	0.25	0.20	0.16	0.092
5 hr	0.14	0.12**	0.12	0.099 (4 hr)
11 hr	0.11	-	-	0.085 (6 hr)
20 hr	0.091			
U_3O_8 ext'n, %	84.7	83.3	75.0	84.6
Overall extraction from ore, %	76	75	69	79

Notes: * Test 19 was run in three stages, at increasing dilutions, for a total time of 20 hours.

** Test 21-Retreatment of residue for an additional 6 hours with 50 lb H_2SO_4 per ton gave a final residue of 0.10 (U_3O_8%).

*** Pulp density, 30% solids.

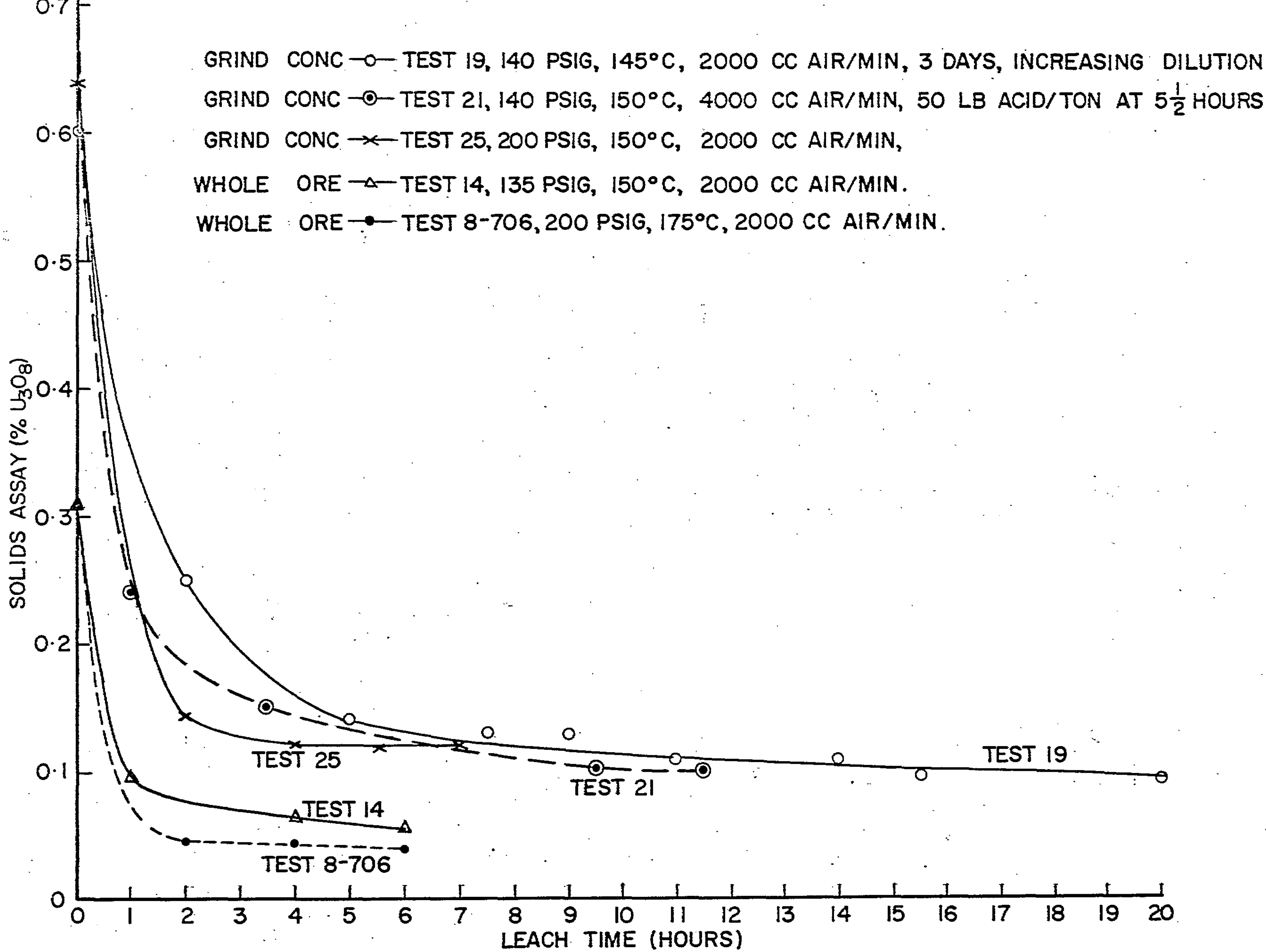

FIGURE 2

EXTRACTION CURVES FOR PRESSURE LEACH TESTS

DISCUSSION

The uranium is finely disseminated in the matrix of a quartz pebble conglomerate, and the ore minerals are, for the most part, softer than the quartz gangue, as evidenced by the results of differential grinding tests. However, some of the uranium minerals are refractory (coffinite has been identified) and near complete extraction has not yet been attained. Strong acid (200 lb H_2SO_4 per ton) releaching of pressure leach residues did not effect any additional extraction.

On the basis of the work reported here, pressure leaching of the whole ore is the most attractive treatment method, in that this process gives optimum overall extraction. Concentration of the ore by differential grinding, followed by pressure leaching of the concentrate, is only feasible if the economics of the process indicate that a smaller pressure leach plant warrants an additional 10% uranium loss.

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