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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 58-150

PRELIMINARY PROCESS INVESTIGATION TESTS ON URANIUM-BEARING ORE SAMPLES FROM THE KITTS PROJECT OF BRITISH NEWFOUNDLAND EXPLORATION LTD., NEWFOUNDLAND

by

H. H. McCREEDY

RADIOACTIVITY DIVISION

NOTE: This report relates essentially to the samples as received. It shall not, nor any correspondence connected therewith, be used in part or in full as publicity or advertising matter.

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H. H. McCreedy*

SUMMARY OF RESULTS

Sink-float and superpanner tests showed that the ore represented by these samples was not amenable to gravity concentration. Flotation methods, differential grinding techniques, and autooxidation pressure leach procedures were also unsatisfactory for uranium beneficiation.

Conventional acid leaching, at controlled pH, gave extractions as high as 98.6%. Acid consumption values ranged from 122 to 288 lb 100% H_2SO_4/ ton ore depending upon the sample tested. The acid consumptions varied because of the different acid-consuming mineral contents of the samples.

Direct precipitation of leach liquors, using an iron reduction procedure prior to neutralization, gave precipitates containing as high as 26.2% U_3O_8 with a $Mg(OH)_2$ consumption of about 27 lb/ ton of solution.

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*Scientific Officer, Radioactivity Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

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(30 pages, 20 tables, 2 illus.)

INTRODUCTION

British Newfoundland Exploration Limited, 1980 Sherbrooke St. West, Montreal, Quebec, under A. E. C. B. Exploration Permit MX 25/57, submitted four ore samples to the Radioactivity Division of the Mines Branch, Ottawa, between December 1957 and March 1958, for process application investigations. The request for the work was made by Dr. A. P. Beavan, General Manager of British Newfoundland Exploration Limited, in a letter dated December 9, 1957. Dr. Beavan stated in his letter that samples submitted were from the company's Kitts project at Makkovic, Labrador, Newfoundland.

The first sample, designated as K-1 by the company, weighed about 70 pounds and was a composite of pulverized assay rejects from diamond drill core. It was received on December 11, 1957, and was given Radioactivity Division No. 12/57-9. This sample was mainly for complete head analysis and preliminary leach tests.

The second sample consisted of approximately 70 pounds of split diamond drill core, and was for routine gravity and leach tests. This sample, designated as K-2 by the company, was the other half of sample K-1, but in an unpulverized form. It was received on February 20, 1958, and was given Radioactivity Division No. 2/58-14.

The third sample submitted, which consisted of approximately 399 pounds of lump ore, was received on December 26, 1957, and was given Radioactivity Division No. 12/57-20. This was the highest grade sample of the group (1.23% U_3O_8), and because of this, only a few gravity concentration tests were carried out. It was believed, at this time, that this high grade sample would not be representative of the mill feed at the property. No company identification number was forwarded for this sample.

The fourth sample, identified as K-3 by the company and supposed to be more representative of mine grade ore, was sent mainly for a continuation of leach tests. This lump ore sample, weighing about 710 pounds, was received on March 28, 1958 and was given Radioactivity Division No. 3/58-16.

HEAD SAMPLE ANALYSES

The various samples received for test work were assayed by chemical, radiometric and spectrographic methods. The results are given in Tables 1 and 2.

TABLE 1

Chemical and Radiometric Analyses of Head Samples

Sample	No. 12/57-9 K1	No. 2/58-14 K2	No. 3/58-16 K3
Chemical Analysis, %			
U_3O_8	1.16	0.74	0.92
U_3O_8 (secondary)*	0.15	0.086	0.086
ThO_2	0.03	<0.003	0.003
CO_2 (evolution)	3.52	2.94	1.42
CO_2 (combustion)	7.27	7.08	10.35
S (total)	2.03	2.18	2.41
Fe	8.22	8.87	7.93
As	<0.01	0.047	0.031
F	0.06	0.08	-
P_2O_5	0.11	0.08	0.06
V_2O_5	0.2	<0.02	-
TiO_2	0.51	0.75	0.89
Mo	0.096	0.055	-
$(RE)_2O_3$ (rare earth oxides)	0.4	<0.07	0.035
Cu	0.052	0.038	-
Zn	0.038	0.045	-
Mn	-	0.098	0.073
Radiometric Assays, %			
U_3O_8 gamma	1.14	0.765	0.93
U_3O_8 beta	1.14	0.776	0.93
U_3O_8	1.14	0.79	0.93
Fire Assays, oz/ton			
Au	0.005	trace	trace
Ag	0.225	0.05	0.08

* The sample was leached for 30 minutes in a hot 10% solution of Na_2CO_3 without added oxidizing agent. The uranium dissolved was taken as an indication of the secondary uranium present.

TABLE 2

Semi-Quantitative Spectrographic Analyses-(Elements-%)

	No. 12/57-9 K1	No. 2/58-14 K2	No. 3/58-16 K3
Si	P. C.	P. C.	P. C.
Fe	20	15	9
Al	12	12	10
Na	5	10	10
Mg	5	5	2.5
Ca	12	10	5
Pb	0.5	0.8	0.6
Mn	0.3	0.4	0.2
Mo	0.3	0.4	0.15
Cu	0.15	0.1	0.2
U	0.6	0.8	0.7
Ba	0.06	0.05	0.05
As	-	0.2	0.15
V	0.06	0.06	0.04
Cr	0.01	0.02	0.02
Zr	0.02	0.05	0.01
Ni	0.03	0.05	0.04
Ti	0.8	1.00	0.5
Co	0.009	0.02	0.006
Y	0.005	0.01	
B	-	-	0.006
Be	-	-	<0.001
Ag	0.2	-	-
Th	N. D.	-	-
Ce	N. D.	-	-
Yb	0.002	-	-
La	0.008	-	-
Sn	N. D.	-	-

P. C. -principal constituent
N. D. -not detected

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SINK-FLOAT INVESTIGATIONS

Test Procedure and Results

The samples were crushed to minus 1 inch. The screen analysis of sample No. 12/57-20, after crushing, is shown in Table 3. Sink-float test work was first carried out on the minus 1 inch plus 4 mesh size fraction of the ore, and the results of this procedure are shown in Table 4. Both the sink and the float products were then screened into four size fractions to determine at what size the uranium minerals were free, or whether a clean float product could be produced. The screen analyses of the sink-float products are shown on Table 5.

All of the above sink-float products were re-combined and crushed to minus 4 mesh and the minus 4 mesh plus 35 mesh fractions were retreated by heavy media separation techniques. The minus 35 mesh sizes were treated on the Haultain superpanner. The results of this work are shown in Table 6.

The sink-float tests were carried out using a heavy liquid medium of acetylene tetrabromide and carbon tetrachloride with a specific gravity of 2.90.

All plus 4 mesh size products from sink-float treatment were assayed radiometrically and the gamma equivalent U_3O_8 reported, while all minus 4 mesh size products were pulverized and assayed chemically.

Sample No. 2/58-14 was crushed to minus 1 inch and a representative portion of about 22 pounds was screened into a number of size fractions. The plus 65 mesh sizes were treated by sink-float in heavy liquid at specific gravity 2.90 as above, while the minus 65 mesh sizes were superpanned. All products were pulverized and assayed chemically for U_3O_8.

The results of the treatment of the various sizes and overall balance are shown in Table 7.

TABLE 3

Screen Analysis of Crushed Ore for Sink-float, Sample No. 12/57-20

Size	Wt, lb	Wt Dist., %
-1 in. + 4 mesh	305	76.8
-4 mesh	92	23.2
Total	397	100.0

TABLE 4

Sink-float Results on -1 in. + 4 mesh Fraction, Sample No. 12/57-20

(sp gr of medium, 2.90)

Product	Wt, lb	Wt, %	Assay, γ U_3O_8, %	U_3O_8 Dist., %
Sink	6.8	18.2	3.02	43.0
Float	30.5	81.8	0.89	57.0
Total	37.3	100.0	1.26	100.0

TABLE 5

Screen Analyses of Products from Sink-float Test on -1 in. + 4 mesh Fraction of Sample No. 12/57-20

Product and Size	Wt, %		Assay, U_3O_8%	U_3O_8 Dist., %	
	Product	Overall		Product	Overall
Sink					
+ 3/4 in.	8.4	1.53	2.46	6.9	2.9
-3/4 in. + 1/2 in.	36.0	6.55	2.78	33.2	14.3
-1/2 in. + 4 mesh	44.2	8.04	3.22	47.1	20.3
-4 mesh	11.4	2.08	3.40	12.8	5.5
Total	100.0	18.20	3.02	100.0	43.0
Float					
+ 3/4 in.	8.6	7.0	1.62	15.7	8.9
-3/4 in. + 1/2 in.	35.1	28.7	0.94	37.1	21.1
-1/2 in. + 4 mesh	47.0	38.5	0.74	39.1	22.3
-4 mesh	9.3	7.6	0.78	8.1	4.7
Total	100.0	81.8	0.89	100.0	57.0

TABLE 6

Sink-float and Superpanner Results on Sample No. 12/57-20

Crushed to -4 mesh

(sp gr of sink-float medium, 2.90)

Product and Mesh Size	Wt %		Assay,	U_3O_8 Dist., %	
	Fraction	Overall	% U_3O_8	Fraction	Overall
-4 + 10					
Sink	29.3	19.9	2.84	67.8	45.8
Float	70.7	48.1	0.56	32.2	21.8
Total	100.0	68.0	1.23	100.0	67.6
-10 + 35					
Sink	28.6	4.7	3.78	74.4	14.4
Float	71.4	11.8	0.52	25.6	5.0
Total	100.0	16.5	1.45	100.0	19.4
	= = = = =				
-35 + 100					
Tip	5.1	0.3	4.31	22.7	1.1
Tailing	94.9	6.1	0.79	77.3	3.9
Total	100.0	6.4	0.97	100.0	5.0
-100 + 200					
Tip	7.0	0.2	4.18	22.7	0.7
Tailing	93.0	3.3	1.07	77.3	2.8
Total	100.0	3.5	1.29	100.0	3.5
-200					
Tip	5.0	0.3	6.09	30.8	1.4
Tailing	95.0	5.3	0.72	69.2	3.1
Total	100.0	5.6	0.99	100.0	4.5
Grand Total		100.0	1.23		100.0
Total of Sinks and Tips		25.4	3.08		63.4

TABLE 7

Sink-float and Superpanner Results on Sample No. 2/58-14
Crushed to -1 inch
(sp gr of sink-float medium, 2.90)

Product and Size	Wt % Fraction	Wt % Total	Assay, % U_3O_8	U_3O_8 Dist., % Fraction	U_3O_8 Dist., % Total
-1 + 3/4 in.					
Sink	28.4	3.6	0.52	72.2	2.52
Float	71.6	9.0	0.08	27.8	0.97
	100.0	12.6	0.21	100.0	3.49
-3/4 + 1/2 in.					
Sink	24.9	11.5	2.23	79.5	34.45
Float	75.1	34.5	0.19	20.5	8.81
	100.0	46.0	0.70	100.0	43.26
-1/2 + 1/4 in.					
Sink	31.3	8.0	2.41	87.3	25.90
Float	68.7	17.7	0.16	12.7	3.80
	100.0	25.7	0.86	100.0	29.70
-3 + 8 mesh					
Sink	31.0	2.2	3.00	88.2	8.87
Float	69.0	4.9	0.18	11.8	1.19
	100.0	7.1	1.05	100.0	10.06
-8 + 65 mesh					
Sink	30.8	2.0	3.78	90.8	10.15
Float	69.2	4.5	0.17	9.2	1.03
	100.0	6.5	1.28	100.0	11.18
	= = = = =				
-65 + 200 mesh					
Tip	3.5	0.03	10.68	41.8	0.43
Middling	8.5	0.08	1.99	18.9	0.21
Tailing	88.0	0.77	0.40	39.3	0.41
	100.0	0.88	0.90	100.0	1.05
-200 mesh					
Tip	1.6	0.02	24.76	57.6	0.66
Middling	4.6	0.06	2.41	12.9	0.19
Tailing	93.8	1.14	0.27	29.5	0.41
	100.0	1.22	0.86	100.0	1.26
Grand Total		100.00	0.74		100.00
Total of Sinks, Tips and Middling		27.49	2.26		83.38

DISCUSSION OF RESULTS OF SINK-FLOAT INVESTIGATIONS

The sink-float investigations were done to investigate the possibility of making a gravity concentrate containing 10% U_3O_8 or higher.

With the minus 1 plus 4 mesh fraction of sample No. 12/57-20, a recovery of only 43.0% was obtained at a grade of 3.02% in 18.2% of the weight (Table 4). These results were obtained using a separating medium of specific gravity 2.90. Although recovery would be higher using medium of lower specific gravity, the grade of concentrate would be lower and weight percent higher. The sink at specific gravity 2.90 is more indicative of the results that would be obtained on a jig or concentrating table. The screen analyses of the products do not indicate an optimum size at which most of the mineral is free (Table 5). Recrushed to minus 4 mesh, the ore was still not amenable to production of a 10% grade concentrate (Table 6). Results show that a 3.08% concentrate could be produced with a recovery of about 63.4% in 25.4% of the overall weight.

Sample No. 2/58-14 was lower in grade (0.74% U_3O_8) than sample No. 12/57-20 and was also not amenable to production of a 10% U_3O_8 product. An overall recovery of about 83.4% was obtained at a grade of 2.26% U_3O_8 in about 27.5% of the overall weight (Table 7). Products of over 10% U_3O_8 were obtained in the superpanner tip fractions of the minus 65 plus 200 and minus 200 mesh sizes. Grades of about 10.7 and 24.8% U_3O_8, respectively, were obtained in these fractions but they represented a total of only 1.1% of the total uranium in the ore in about 5.1% of the overall weight.

DIFFERENTIAL GRINDING TESTS

This test work was carried out on two 1000-g lots of minus 4 mesh sample No. 2/58-14, ground in a porcelain Abbe ball mill with 20 pounds of steel balls. One sample was ground for 10 minutes and the other for 30 minutes. The products were then screened for a size analysis. The results of the two tests are shown in Table 8.

Data indicated that no appreciable concentration of uranium occurred in the fine sizes after grinding the comparatively coarse feed samples for periods of 10 and 30 minutes.

TABLE 8

Differential Grinding Test Data for Two Grinding Periods, Sample No. 2/58-14

Mesh Size	10 min grind				30 min grind			
	Wt		Assay,	U_3O_8 Dist.,	Wt		Assay,	U_3O_8 Dist.,
	g	%	% U_3O_8	%	g	%	% U_3O_8	%
-4 + 6	223	22.3	0.74	22.0	193	19.3	0.50	13.2
-6 + 8	132	13.2	0.71	12.5	109	10.9	0.67	10.0
-8 + 10	125	12.5	0.75	12.5	103	10.3	0.74	10.4
-10 + 14	83	8.3	0.81	9.0	62	6.2	0.63	5.3
-14 + 20	49	4.9	0.66	4.3	33	3.3	0.77	3.5
-20 + 28	36	3.6	0.85	4.1	22	2.2	0.70	2.1
-28 + 65	75	7.5	0.94	9.4	57	5.7	0.77	6.0
-65 + 100	26	2.6	0.87	3.0	28	2.8	0.88	3.4
-100 + 200	46	4.6	0.70	4.3	64	6.4	0.76	6.6
-200	205	20.5	0.69	18.9	329	32.9	0.88	39.5
Total	1000	100.0	0.75	100.0	1000	100.0	0.73	100.0

FLOTATION TESTING

Flotation testing, carried out on sample No. 2/58-14, was very preliminary in nature. One test was done with a view to floating uranium and four tests were done to attempt to float off carbonate minerals.

Flotation Test No. 1

The object of this test was the flotation of the carbonate minerals.

Reagents added:	lb/ton
Na_2CO_3 (pH 10.0)	5
Conditioned - 5 min	
Dowfroth 250	0.5
Double-distilled oleic acid, (3 stages)	0.5
Conditioned - 10 min	
Rougher conc. flotation - 5 min	
Cleaner conc. flotation - 5 min	

The metallurgical balance is shown in Table 9.

TABLE 9

Flotation Results Test No. 1 -(Sample No. 2/58-14)

Product	Wt		U_3O_8		CO_2	
	g	%	Assay, %	% Dist.	Assay, %	% Dist.
Cleaner Conc.	621	64.4	0.54	49.7	4.48	93.9
Cleaner Tailing	118	12.3	0.80	14.0	0.47	1.9
Rougher Tailing	225	23.3	1.09	36.3	0.56	4.2
Total	964	100.0	0.70	100.0	3.07	100.0

Flotation Test No. 2

The object of this test was to produce a higher grade concentrate by a reduction in the frothing agent. The same procedure as in Test No. 1 was used, except the Dowfroth reagent was omitted.

The metallurgical balance of Test No. 2 is shown in Table 10.

TABLE 10

Flotation Results Test No. 2-(Sample No. 2/58-14)

Product	Wt		U_3O_8		CO_2	
	g	%	Assay, %	% Dist.	Assay, %	% Dist.
Cleaner Conc.	342	33.2	0.45	21.4	8.05	72.8
Cleaner Tailing	268	26.0	0.61	22.8	0.97	5.0
Rougher Tailing	420	40.8	0.95	55.8	0.22	22.2
Total	1030	100.0	0.70	100.0	3.01	100.0

Flotation Test No. 3

The object of this test was to float only the uranium.

Reagents added:	lb/ton
Dowfroth 250	0.25
Conditioned - 5 min	
Sulphonated whale oil	2.0
Fuel oil	1.0
Conditioned - 10 min	
Rougher conc. flotation - 4 min	
Cleaner conc. flotation - 4 min	
Recleaner conc. flotation - 4 min	

The metallurgical balance of Test No. 3 is shown in Table 11.

TABLE 11

Flotation Results Test No. 3-(Sample No. 2/58-14)

Product	Wt		U_3O_8		CO_2	
	g	%	Assay, %	% Dist.	Assay, %	% Dist.
Recleaner Conc.	482	48.2	0.80	52.4	5.52	89.6
Recleaner Tailing	76	7.6	0.56	5.8	0.68	1.7
Cleaner Tailing	112	11.2	0.56	8.6	0.76	2.9
Rougher Tailing	330	33.0	0.74	33.2	0.52	5.8
Total	1000	100.0	0.74	100.0	2.97	100.0

Flotation Test No. 4

A sulphide float followed by a carbonate mineral float was the general procedure followed for this test.

Reagents added:	lb/ton
Sodium isopropyl xanthate	0.2
Dowfroth 250	0.08
Conditioned - 2 min	
Flotation - 3 min (sulphides)	
Sodium isopropyl xanthate	0.2
Flotation - 2 min (sulphides)	
Sodium carbonate	3.5
Conditioned - 5 min	
Sodium silicate	2.0
Conditioned - 2 min	
Double-distilled oleic acid (3 stages)	0.8
Flotation - 3 min (carbonates)	

The results of Test No. 4 are shown in Table 12.

TABLE 12

Flotation Results Test No. 4 (Sample No. 2/58-14)

Product	Wt		U_3O_8		CO_2		S	
	g	%	Assay, %	% Dist.	Assay, %	% Dist.	Assay, %	% Dist.
Sulphide Conc.	85	18.0	0.52	17.2	1.34	6.8	7.96	85.4
Carbonate Conc.	63	13.3	0.50	12.3	19.1	71.3	0.66	5.2
Tailing	325	68.7	0.56	70.5	1.14	21.9	0.23	9.4
Total	473	100.0	0.55	100.0	3.57	100.0	1.70	100.0

Flotation Test No. 5

The object of this test was the same as Test No. 4 but with some variation in reagents.

Reagents added:	lb/ton
Sulphuric acid (pH 6.0)	8
Sodium isopropyl xanthate	0.2
Dowfroth 250	0.08
Conditioned - 5 min	
Flotation - 5 min (sulphides)	
Sodium carbonate (pH 9.8)	20.
Sodium silicate	2.5
Double-distilled oleic acid (stagewise)	2.5
Conditioned - 2 min	
Flotation - 3 min (carbonates)	

The test results are shown in Table 13.

TABLE 13

Flotation Results Test No. 5-(Sample No. 2/58-14)

Product	Wt		U_3O_8		CO_2		S	
	g	%	Assay, %	% Dist.	Assay, %	% Dist.	Assay, %	% Dist.
Sulphide float	86	17.9	0.60	16.0	0.97	6.0	11.95	89.3
Carbonate float	39	8.1	0.56	6.7	27.6	77.5	0.32	1.1
Tailing	356	74.0	0.70	77.3	0.64	16.5	0.31	9.6
Total	481	100.0	0.67	100.0	2.88	100.0	2.39	100.0

DISCUSSION OF FLOTATION RESULTS

The results of the test for flotation of only the uranium were poor in that a concentrate containing 48.2% of the total weight contained only 52.4% of the uranium and 89.6% of the CO_2 contents. The best of the four carbonate mineral flotation tests resulted in a concentrate containing 77.5% of the CO_2 content at a grade of 27.6% CO_2 in 8.1% of the feed weight, along with 6.7% of the uranium at a grade of 0.56% U_3O_8. In this test the sulphide concentrate contained 89.3% of the sulphur and 16% of the uranium in 17.9% of the total weight.

Although good recovery of the carbonate minerals was obtained by flotation, the reagent consumption was high. Since subsequent samples from the mine showed comparatively low CO_2 content, this work was not continued.

AUTOOXIDATION PRESSURE LEACH INVESTIGATION

One autooxidation pressure leach test was carried out on sample No. 2/58-14. A sample of 2000 grams, dry weight, was repulped to 60% solids with water and agitated in an autoclave under a pressure of about 135 psig for six hours. The autoclave was heated to maintain a temperature of 150°C during the test. An airflow rate through the autoclave of about 2000 standard cm^3/min was used.

The initial pH of the pulp was 8.6 and the final value was 7.5. The initial feed assay was 0.74% U_3O_8 and the final residue after 6 hours was 0.65% U_3O_8.

In view of these poor results and since the more familiar sulphuric acid leach could be used successfully on this ore, the pressure leach investigations were discontinued.

CONTROLLED pH ACID LEACHING OF THE VARIOUS SAMPLES

A number of controlled pH sulphuric acid leach tests were carried out on the various samples. Sodium chlorate was used for the oxident. The majority were on sample No. 2/58-14, since this was thought at the time to be more representative of mine grade ore. Two tests of a preliminary nature were carried out on sample No. 12/57-9. This sample consisted of finely-ground assay rejects. Sample No. 3/58-16 was of a high grade nature and only three tests were tried to note any variation in results from the previous sample (K2).

The feed for each test was ground in an Abbé porcelain laboratory ball mill with steel balls. Leach conditions were as listed in Table 14. Intermediate residue samples were taken to check the leaching rate. On two samples (No. 12/57-9 and 2/58-14), a leach test was tried after the magnetic minerals and abraded metallic iron

TABLE 14

Leach Test Data

Sample No.	Sample No. 12/57-9		Sample No. 2/58-14												Sample No. 3/58-16		
Test No.	9A	9B	14A	14B	14C	14D	14E	14F	14G	14H	14J	14K	14L	14N	16A	16B	16C
Grind, % -200 mesh	76.4	76.4	70	70	70	78	78	44	66	66	66	66	66	66	66	66	66
Sodium chlorate added, lb/ton	0	5	20	5	10	5	5	5	nil	5	5	5	2.5	5	5	2.5	5
pH controlled at	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	2.0	1.75	2.5	1.5	1.0	1.5	1.5	1.0
Acid consumption, lb 100% H_2SO_4/ton ore at:																	
0 hr	200	200	126	124	128	142	126	102	126	110	118	100	126	160	62	65	100
8 hr	228	230	178	170	174	180	164	142	156	136	154	120	168	180	98	99	138
24 hr	258	262	208	198	196	200	190	162	184	158	170	142	192	222	110	117	166
32 hr	270	272	216	204	204	202	196	168	188	160	174	144	196	226	118	122	184
48 hr	288	282	220	208	208	204	204	180	192	162	182	146	198	236	122	126	196
Residue assay, % U_3O_8 at:																	
8 hr	0.93	-	0.041	0.093	0.036	0.093	0.073	0.098	0.64	0.085	0.071	0.45	0.15	0.035	0.077	0.22	0.043
24 hr	0.94	0.025	0.017	0.021	0.025	0.019	0.022	0.066	0.58	0.034	0.030	0.21	0.14	0.014	-	0.19	0.017
32 hr	-	-	0.012	0.017	0.016	0.015	0.013	0.039	0.58	0.034	0.032	0.17	0.13	0.012	-	0.18	0.016
48 hr	0.89	0.018	0.010	0.016	0.017	0.014	0.014	0.027	0.60	0.034	0.028	0.16	0.091	0.009	0.030	0.13	0.015
Leach solution																	
Volume, ml	460	610	365	375	410	560	375	405	430	395	450	375	375	370	410	345	395
U_3O_8, g/l	3.24	11.3	9.93	9.53	10.06	7.75	13.05	11.41	1.19	10.95	10.18	8.32	10.17	11.26	15.07	11.65	14.7
U_3O_8, % Dist.	13.8	64.9	53.5	54.0	53.8	60.7	66.5	66.3	7.9	62.1	66.3	47.1	56.7	61.1	74.4	46.1	69.6
Reducing Power, g/l	17.8	7.6	12.9	10.9	12.0	9.3	8.0	12.3	12.7	10.7	11.4	6.0	12.2	11.4	5.0	14.9	17.1
Total Fe, g/l	18.2	10.0	15.4	10.2	12.8	8.7	9.6	12.4	11.3	9.4	10.1	6.7	10.5	15.3	12.8	15.5	20.1
Wash Solution																	
Volume, ml	760	790	775	770	745	760	740	740	740	760	755	740	760	760	740	745	760
U_3O_8, g/l	1.49	4.51	3.94	3.77	4.54	3.52	3.15	2.82	0.40	3.05	2.73	2.66	2.71	3.38	2.50	4.67	3.15
U_3O_8, % Dist.	10.7	33.5	45.1	43.8	44.1	37.4	31.7	30.0	4.6	33.3	29.9	29.7	30.6	37.7	22.3	39.9	28.7
Reducing Power, g/l	8.1	3.1	4.8	4.3	5.6	4.4	2.0	2.0	4.1	3.0	3.1	1.93	0.53	3.4	2.5	1.4	3.6
Total Fe, g/l	8.2	4.1	7.5	3.9	5.9	3.9	2.15	1.9	3.4	2.5	2.7	2.2	2.8	4.6	2.5	3.7	4.3
Final Residue																	
Wt, gm	901	956	910	915	934	960	962	960	946	947	947	960	941	949	908	935	943
U_3O_8, %	0.89	0.018	0.010	0.016	0.017	0.014	0.014	0.027	0.60	0.034	0.028	0.16	0.091	0.009	0.030	0.13	0.015
U_3O_8, % Dist.	75.5	1.6	1.4	2.2	2.1	1.9	1.8	3.7	87.5	4.6	3.8	23.2	12.7	1.2	3.3	14.0	1.7
Calc. feed assay, % U_3O_8	1.18	1.06	0.74	0.72	0.82	0.74	0.77	0.73	0.69	0.74	0.73	0.69	0.72	0.68	0.91	0.93	0.88
Extraction, %, based on calc. feed assay	24.5	98.4	98.6	97.8	97.9	98.1	98.2	96.3	12.5	95.4	96.2	76.8	87.3	98.8	96.7	86.0	98.3
		Magnetics removed 6% of total Wt	←	Various Chlorate	→	Fine Grind	Magnetics removed	Coarse Grind	No Chlorate	←	Various pH	→	Low Chlorate				

had been removed with a hand magnet.

Chlorate addition, fineness of grind, and acid level were varied in the group of tests.

The leach test results are shown in Table 14 and Figures 1 and 2.

One batch leach was carried out on a 100-lb batch of sample No. 3/58-16. This was done to compare results with bench-scale leach data, to provide pulp for settling and filtering tests and to produce leach liquor for preliminary solvent extraction tests. The test was continued for 48 hours at pH 1.5 and 5 lb $NaClO_3$/ton ore. The data obtained are shown in Tables 15 and 16.

TABLE 15

Large Scale Batch Leach Data (Sample No. 3/58-16)

Contact Time, hr	100% H_2SO_4 Used, lb/ton ore	Residue Assay, % U_3O_8	Extraction, %
0	-	0.93	0
4	-	0.15	83.9
24	143	0.032	96.6
48	156	0.024	97.4

TABLE 16

Analysis of Leach Liquor from Large Scale Batch Leach, (Sample No. 3/58-16)

Analysed for	Assay, g/1	Analysed for	Assay, g/1
U_3O_8	4.59	F	0.11
S/SO_4	7.80	P_2O_5	0.10
R.P.*	5.91	$(RE)_2O_3$	0.026
Total Fe	4.88	ThO_2	0.007

*R.P. - reducing power

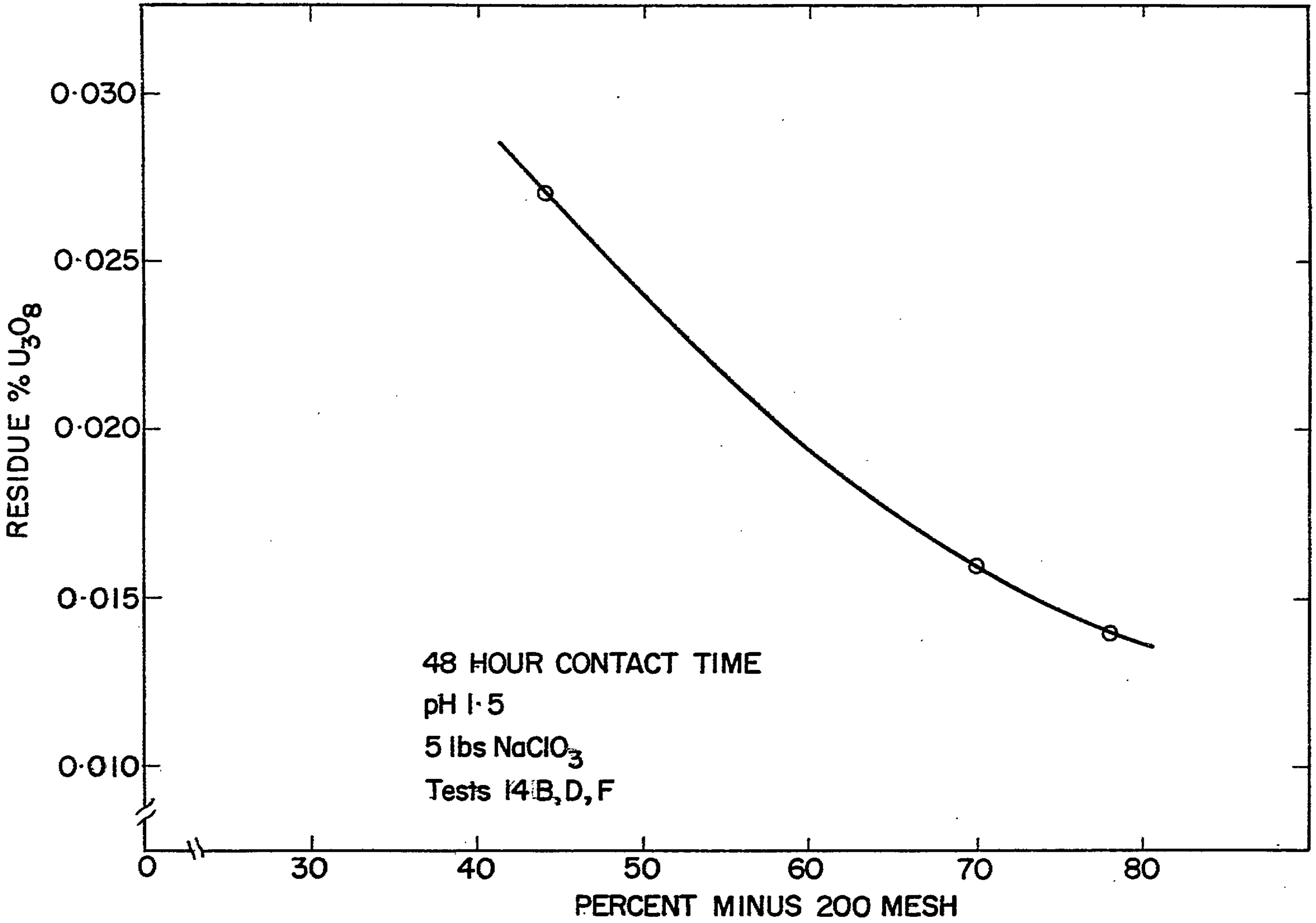

FIGURE 1

EFFECT OF GRIND ON LEACH RESIDUE - SAMPLE 2/58-14(K2)

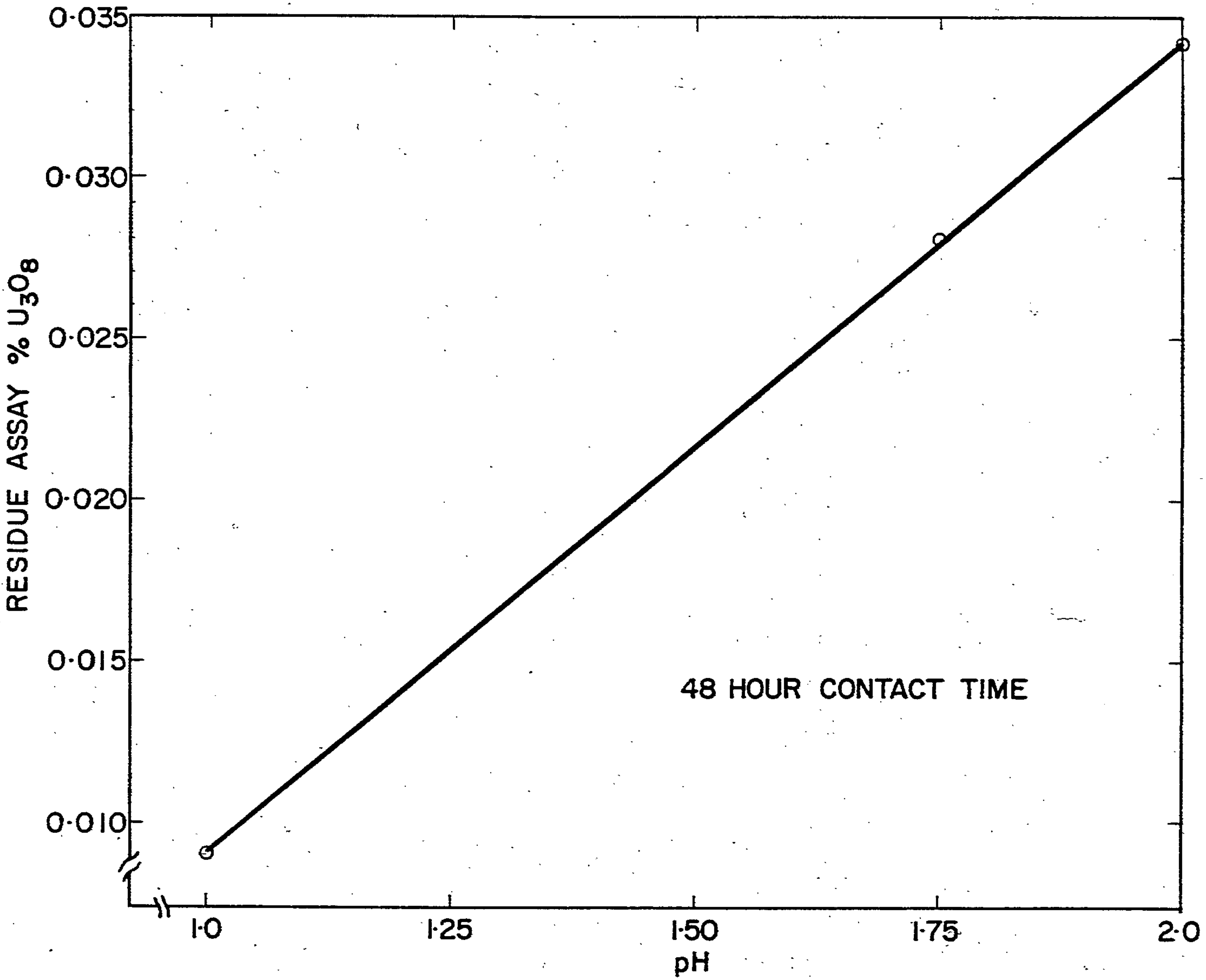

FIGURE 2

EFFECT OF pH ON LEACH RESIDUE-SAMPLE 2/58-14(K2)

DISCUSSION OF LEACH RESULTS

Tests on sample No. 12/57-9 indicated the necessity of using an oxidizing agent for good extraction (Table 14, Tests 9A and 9B). Acid consumption ranged from 282 to 288 lb 100% H_2SO_4/ ton ore at a controlled pH of 1.5. The removal of the magnetics did not appear to affect the metallurgy.

Tests on sample No. 2/58-14 showed that this sample required less acid than sample No. 12/57-9 for similar leaching conditions. Increased chlorate, higher acid content, and finer grinding all gave improved uranium extraction (Table 14).

Tests on sample No. 3/58-16 showed that this sample required less acid than either of the two previous samples. Increased chlorate addition gave improved extraction as with previous samples (Table 14).

EMF MEASUREMENTS ON LEACH PULP

The purpose of the work was to determine the period of time required for the primary reducible elements (metallic iron, etc) to be oxidized, as indicated by EMF readings.

EMF determinations were made on leach pulp, using sample No. 3/58-16, to which sulphuric acid was added to maintain a pH of 1.5. No sodium chlorate was added.

The EMF readings were taken using a platinum-calomel electrode combination connected to a Beckman, Model H, pH meter.

Negative EMF readings were taken as reducing potentials. The results of this test are shown in Table 17.

TABLE 17

EMF Measurements on Leach Pulp (Sample No. 3/58-16)

Contact Time, hr	pH	EMF, mv	Acid Consumption, lb 100% H_2SO_4/ton
0	6.8	-220	0
0	1.5	-180	74
0.5	1.5	-170	84
1	1.5	-170	-
2	1.5	-120	92
18	1.5	+110	130

URANIUM RECOVERY FROM SOLUTION BY DIRECT PRECIPITATION

Three tests were carried out to investigate the direct precipitation of uranium by neutralization from a composite pregnant solution (leach liquors plus washes) produced in the bench scale leach tests on ore sample No. 2/58-14. Two of the tests included a reduction step with metallic iron on the liquor before the addition of the neutralizing agent. In each case, one litre of solution was used and Dow $Mg(OH)_2$ was added as the neutralizing agent. The precipitation step contact time was one hour. The metallic iron used as the reducing agent was Belmont grade D iron filings. The reduction step in Test No. 2 required approximately one hour. In Test No. 3 the reduction step was completed in about one-half hour followed by a 1-hour neutralization period. Results are shown in Table 18.

TABLE 18

Direct Precipitation Test Data and Results
(Sample No. 2/ 58-14)

Test No.	1	2	3
Method	Direct	I. R. *	I. R.
Precipitant Used**	$Mg(OH)_2$	$Mg(OH)_2$	$Mg(OH)_2$
Quantity of precipitants used, g	50	15.0	13.4
Vol of pregnant solution, ml	1000	1000	1000
Iron added, g	nil	7	7
Feed Assays			
pH	1.55	1.55	1.4
EMF, mv	+360	+360	+380
U_3O_8, g/1	4.49	4.49	4.49
Total Fe, g/1	7.9	7.9	7.9
R. P., *** g/1	12.0	12.0	12.0
Solution After Reduction			
pH		1.9	1.65
EMF, mv		+120	-40
Final Barren Assays			
pH	7.2	6.0	6.1
EMF, mv	-	-240	-210
U_3O_8, g/1	0.012	0.045	0.043
Total Fe, g/1	-	6.2	-
R. P., g/1	-	11.0	-
Precipitate Assays			
Wt, g	62.5	24.0	21.8
U_3O_8, %	6.6	16.5	26.2
Total Fe, %	2.8	12.0	2.5
Remarks	Brown to green at pH 6.2.	Green. Slow iron filtration.	Dirty green. Rapid iron filtration.

* I.R. - Iron reduction

** Produced by Dow Chemical Co.

*** R.P. - Reducing power

The highest grade uranium precipitate obtained was 26.2% U_3O_8, (Test No. 3). These preliminary tests did not indicate the maximum grade possible but rather that a product assaying over 10% U_3O_8 could be produced by the neutralization method.

COMPARISON OF FLOCCULANTS FOR USE AS FILTERING AIDS

The main purpose of the tests was to find the most efficient filtering aid for leached ore of the type represented by sample No. 3/58-16. The method of testing consisted of comparing the rates of filtering one-half litre batches of leached pulp, using the flocculants as listed in Table 19. The pulp was taken from the large scale batch leach (Table 15) at room temperature (26°C), and was filtered on a 11.5 cm diameter Buchner funnel in a two-litre filter flask at 15 inches Hg vacuum. The filtering aid under test was added to the half-litre of pulp in a graduated cylinder and agitated end-over-end approximately five times, before pouring onto a moistened Whatman No. 3 filter paper in the funnel. The time required for the leach liquor to disappear from the surface of the cake in the funnel was recorded. A further five-minute drying time was allowed before the wet cake was weighed. The cake was reweighed after 24 hours of drying time in the oven at 100°C. The filtrate volume was also measured. The results of these tests are recorded in Table 19.

Jaguar MDC appeared to give the best results in filtering. Although Separan and glue resulted in satisfactory filter rates, the cakes cracked during the drying period. All filtrates appeared to have the same clarity.

TABLE 19

Summary of Filtering Tests on Leached Pulp with Various Flocculants
(Sample No. 3/58-16)

Test No.	Additive Type	Quantity, lb/ton	Time to Filter, sec	Moist Wt, g	Dry Wt, g	Cake** Moisture, %	Filtrate Vol, ml	Feed,* % Solids (Calc.)	Filter*** Rate, lb/hr	Remarks
1	nil	-	20							—500 ml water only
2	nil	-	187	599	478	20.2	235	57.3	20	
3	Jaguar MDC	0.1	27	559	462	17.4	245	57.5	136	
4	Jaguar MDC	0.05	60	588	475	19.2	260	56.0	63	
5	Separan	0.033	120	574	470	18.1	255	56.7	31	
6	Separan	0.066	76	595	468	21.3	240	56.0	49	
7	Separan	0.1	50	563	470	16.5	210	60.8	75	- Few cake cracks
8	SE Glue	0.4	41	584	464	20.5	215	58.1	90	- Many cake cracks
9	SE Glue	0.2	60	573	471	17.8	240	57.9	62	- Few cake cracks
10	SE Glue	0.1	100	581	484	16.7	217	60.7	38	- Few cake cracks
11	Polyox	0.025	177	586	484	17.4	245	58.2	22	
12	Polyox	0.05	165	581	473	18.6	230	58.3	23	
13	Polyox	0.1	108	568	474	16.5	260	57.2	35	
14	Polyox	0.2	114	572	476	16.8	235	59.0	33	

*Feed 61% -200 mesh

**After 5 minutes suction time

***Comparative filter rates through the 11.5 cm diam. Buchner funnel

SETTLING RATE DETERMINATIONS ON LEACHED PULP

The settling tests were carried out on diluted leach pulp from the large scale batch leach of sample No. 3/58-16. The acid pulp was diluted to approximately 35% solids with water before the flocculating aid was added. The cylinders of pulp were then agitated end-over-end approximately five times, before the settling rate was measured. This rate was determined by the level of the solids-liquids interface at various time intervals.

The summary of the settling tests is recorded in Table 20.

The lowest required settling area of 0.1 sq ft/ton/day was obtained with 0.1 lb Polyox/ton ore (Test 116) but the supernatent liquor was very cloudy, indicating incomplete settling. Jaguar MDC also gave fast settling rates but the overflow liquids again ranged from very hazy to very cloudy in clarity, depending upon the quantity added. One-tenth lb Jaguar MDC per ton ore gave a settling rate of 3.1 ft/hr and required an area of 0.5 sq ft/ton/day (Test No. 113). Separan, in the amount of 0.1 lb/ton ore, gave a rate of settling of 2.7 ft/hr and required an area of 0.6 sq ft/ton/day (Test No. 114). The supernatent solution in this latter case was crystal clear. The addition of a flocculating agent in all cases reduced the required area for settling below any design figure used in practice.

GENERAL DISCUSSION

The test work carried out indicated that there is little likelihood of economic recovery of the uranium except by acid leaching.

TABLE 20

Summary of Settling Tests on Leached Pulp (Sample No. 3/58-16)

Test No.	112	113	114	115	116	117	118	119	120	121	122
Additive, Type and lb/ton	nil	Jaguar MDC 0.1	Separan 0.1	Glue 0.2	Polyox 0.1	Separan 0.05	Jaguar MDC 0.025	Jaguar MDC 0.05	Jaguar MDC 0.1	Jaguar MDC 0.2	Jaguar MDC 0.4
% solids to start	34.8	34.8	35.5	35.9	34.9	34.8	35.4	35.1	35.9	34.6	35.3
% solids in 1/2 hr	-	72	72	48	68	66	66	69	70	68	74
Rate, ft/hr	0.1	3.1	2.7	0.7	1.5	1.8	2.4	2.7	3.1	3.8	5.1
Coe-Clevenger Area, sq ft/ton/day	16.0	0.5	0.6	2.1	0.1	0.9	0.6	0.6	0.5	0.4	0.3
Clarity of Overflow	Extra Cloudy	Very Hazy	Crystal Clear	Very Hazy	Very Cloudy	Clear	Very Hazy	Very Hazy	Very Hazy	Very Cloudy	Extra Cloudy

Since the uranium mineral is pitchblende, the ore is amenable to the controlled pH sulphuric acid leach process, provided that an oxident is used. The leach solutions are high enough in U_3O_8 grade to be suitable for recovery of the uranium by ion exchange, solvent extraction or bulk neutralization methods.

The comparatively high titanium content of the ore might be a factor worthy of note in subsequent liquor treatment. In leaching, all the samples submitted produced considerable H_2S gas upon the initial acid addition. Although not appearing to affect the metallurgy, this chemical reaction would necessitate adequate and efficient ventilation in the plant.

The wide range of uranium and acid-consuming mineral contents of the different samples gave considerable variation in the results obtained. It is therefore difficult to estimate the probable residue assays and reagent requirements of the proposed treatment plant at the property.

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HHM:eew