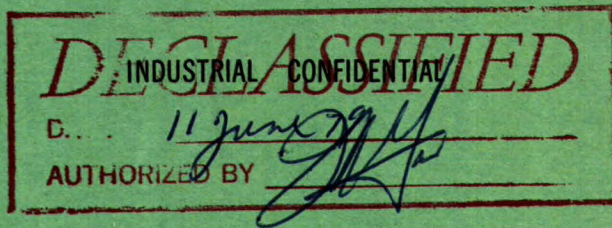


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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 58-177

DIRECT PRECIPITATION TESTS ON SOLUTION PRODUCED IN ACID
LEACHING OF URANIUM ORE FROM THE KITTS PROPERTY OF
BRITISH NEWFOUNDLAND EXPLORATION LIMITED, NEWFOUNDLAND

by

V. F. HARRISON & W. A. GOW

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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Mines Branch Investigation Report IR 58-177

DIRECT PRECIPITATION TESTS ON SOLUTION PRODUCED IN ACID
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SUMMARY OF RESULTS

The uranium values were recovered from pilot plant acid solutions by direct precipitation methods. Magnesia neutralization, aluminum reduction-phosphate precipitation and iron reduction-magnesia neutralization yielded precipitates assaying 40.38%, 53.23% and 14.00% U_3O_8 , respectively. Reagent costs varied from 6.8cents/lb to 37.7 cents/lb U_3O_8 depending on the precipitation procedure employed.

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

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(13 pages, 5 tables)

INTRODUCTION

This work was done in conjunction with pilot plant leaching of bulk samples of uranium ore (Sample Nos. 4/58-12 and 5/58-14) from the Kitts property of British Newfoundland Exploration Limited at Makkovik, Labrador, Newfoundland (1). The resultant uranium-bearing solutions were treated for uranium recovery by ion exchange (2), solvent extraction (3) and direct precipitation processes. Direct precipitation, described in this report, was undertaken to evaluate three standard methods, and to reclaim the soluble uranium from pregnant solutions which were not required for other test purposes. Magnesia neutralization, aluminum reduction-phosphate precipitation and iron reduction-magnesia neutralization were the methods investigated. Product grades and reagent consumptions were obtained using magnesium hydroxide, magnesia MgO "90", magnesia CX-17 and phosphoric acid precipitants. Details regarding the source of these reagents are given in Appendix 1.

Table 1 gives the assays of typical pregnant solutions obtained from leach pilot plant runs described in detail in Mines Branch Investigation Report IR 58-139 (1).

TABLE 1

Pregnant Solution Assays

Leach Pilot Plant No.	pH	Assays (g/l)		
		U ₃ O ₈	Fe ⁺³	Fe(tot)
LPP 85	1.75	3.17	0.30	5.10
LPP 86	1.72	3.43	0.6	5.6
LPP 87	1.75	5.95	0.8	5.4

GENERAL SUMMARY

The results of the test work done on the pregnant solution produced in the acid leaching of the ore from British Newfoundland Exploration Limited, are summarized in Table 2.

Neutralizing the pregnant solution with magnesia resulted in uranium precipitates assaying approximately 20% and 40% U_3O_8 from liquors containing approximately 3 g U_3O_8/l and 7 g U_3O_8/l , respectively.

Precipitation by the aluminum reduction method resulted in precipitates containing over 50% U_3O_8 from both the lower and higher grade solutions. However, the recovery from the lower grade solution was not satisfactory by this method.

Where precipitation of the higher grade liquor was effected by neutralization with CX-17 magnesia, the reagent cost was 6.8 cents/lb U_3O_8 . Tests using other precipitation methods or lower grade liquors indicated the reagent cost would range between 13 cents and 37.7 cents/lb U_3O_8 . These costs compare with a reagent cost of 12 cents/lb U_3O_8 for ion exchange methods (2).

Precipitation with magnesium hydroxide directly, or with magnesia after reducing the solution with metallic iron was not satisfactory.

At the present time only the precipitate produced by the aluminum reduction-phosphate precipitation method is acceptable to the refinery (See Appendix 2).

TABLE 2

Summary of Results

Recovery Method	Test No.	Solution Assays (g/l)		% Recovery (U ₃ O ₈)	Precipitate Assays (%)		Reagent Consumptions (lb/lb U ₃ O ₈)						Approximate Precipitation Cost (cents/lb U ₃ O ₈)		
		Pregnant	Barren		U ₃ O ₈	Fe	Mg(OH) ₂	MgO "90"	MgO CX-17	Iron Powder	Aluminum Powder	P ₂ O ₅ ⁽¹⁾		100% H ₂ SO ₄	
Magnesia Neutralization	86-4	3.62	0.011	99.6	14.81	12.1	6.29 ⁽³⁾							37.7	
	86-14	3.21	0.15	95.4	20.90	8.60		4.64 ⁽²⁾							27.8
	87-5	6.79	0.027	99.6	40.11	5.09		2.16 ⁽²⁾							13.0
	85-9	2.92	0.002	99.9	19.36	18.0				2.81 ⁽⁴⁾					22.5
	87-7	7.02	0.10	98.6	40.38	5.68				0.852 ⁽²⁾					6.8
Aluminum Reduction - Phosphate Precipitation	86-8	3.29	0.34	89.7	51.49	-						0.349	0.773	3.09	32.6
	87-6	6.76	0.042	99.4	53.23	-						0.185	0.627	1.34	21.2
Iron Reduction - Magnesia Neutralization	86-13	3.39	0.022	99.4	14.00	11.38		4.72			0.742				35.7

(1) Phosphoric acid was used as the source of P₂O₅.

(2) In these tests, the magnesia was added as a slurry with water.

(3) In this test, the magnesium hydroxide was added as dry powder.

(4) In this test, the magnesia was added as dry powder.

DETAILS OF TEST WORK AND RESULTS

The tests were carried out on 350 to 400-litre batches of pregnant solution. The precipitation step was done in a Linatex-lined steel tank, and agitation was effected by a marine-type impeller. The precipitate was filtered on a large (3 ft x 3 ft) pan vacuum filter. Details of the procedures used in the different precipitation methods investigated are described separately in the following sections. The details of the test conditions and results are given in Tables 3 and 4.

(1) Magnesia Neutralization

The solutions were neutralized with either Dow $Mg(OH)_2$, Alcan MgO "90" or Alcan CX-17 MgO to a final slurry pH of 6. In some of the tests, the magnesia was added directly as a dry powder. In others, the magnesia was agitated vigorously for 15 minutes in water at a temperature of 25°C. After agitation, the contents were allowed to settle for 5 minutes, and then the fines were decanted off for use in neutralization. The settled fraction was discarded, but its weight was included in the consumption figure.

(2) Aluminum Reduction-Phosphate Precipitation

The uranium was recovered by reducing the solution with aluminum metal powder, and precipitating the tetravalent uranium with phosphoric acid. The amount of aluminum used was equivalent to a 0.5 g/l excess over the theoretical requirement for reducing the uranium and the iron present in the pregnant solution. It was found that sulphuric acid was needed to maintain the solution pH below 1.8 after the aluminum had been added. The amount of phosphoric acid added was equivalent to

TABLE 3

Test Conditions and Reagent Consumptions

Test No.	Recovery Method	Test Conditions			Reagent Consumptions										
		Form in which reagents were added	Agit'n Time (hr)	Final Slurry pH	Magnesia		Aluminum		P ₂ O ₅		100% H ₂ SO ₄		Iron		
					g/l	lb/lb U ₃ O ₈	g/l	lb/lb U ₃ O ₈	g/l	lb/lb U ₃ O ₈	g/l	lb/lb U ₃ O ₈	g/l	lb/lb U ₃ O ₈	
85-4	Aluminum reduc'n-phosphate precipitation	Al powder and H ₃ PO ₄	4	1.70			0.868	0.549	2.10	1.33	2.57	1.62			
86-1		"	6	1.38			1.50	0.478	2.10	0.669	17.1	5.46			
8		"	12 1/2	1.31			1.03	0.349	2.28	0.773	9.13	3.09			
87-1		"	12	1.10			1.74	0.413	4.21	0.998	14.0	3.34			
6		"	3 1/2	1.48			1.24	0.185	4.21	0.627	9.00	1.34			
86-13	Iron reduc'n - MgO "90" neut	Filings and Powder	5	5.98	15.9	4.72							2.50	0.742	
85-1	Neutralization with Mg(OH) ₂	Powder	1	6.10	22.7	9.64									
2		"	1 1/2	5.90	"	7.87									
3		"	1	6.22	"	6.09									
5		"	2	6.25	"	7.31									
6		"	2	6.18	"	7.59									
7		"	1	6.00	"	7.36									
8		"	1	5.98	"	7.58									
86-2		"	1	6.25	"	8.05									
3		"	1	6.35	"	7.34									
4		"	1	6.08	"	6.29									
86-5		Neutralization with MgO "90"	Powder	1	6.15	22.7	7.38								
6			"	1	6.18	17.0	4.67								
7			"	1	6.10	17.5	5.82								
9			"	1	6.00	22.7	7.80								
10	"		4	5.70	13.6	4.27									
11	"		3	5.82	17.0	5.16									
12	"		3	6.10	24.0	6.19									
14	"		3	5.70	14.2	4.64									
87-2	"		Slurry	4 1/2	5.98	13.7	2.24								
3	"		"	2	6.12	15.0	2.25								
4	"	"	2	5.88	11.6	1.75									
5	"	"	1 1/4	6.05	14.6	2.16									
87-7	Neutralization with CX-17 MgO	Slurry	2	6.15	5.89	0.852									
8		"	3	6.10	5.97	0.990									
85-9		"	Powder	2	5.95	8.20	2.81								
10		"	"	2	5.82	6.94	2.72								

TABLE 4

Test Results

Test No.	Recovery Method	Pregnant Solution (Precipitation Feed)							Ratio Fe(tot)/U ₃ O ₈	Barren		U ₃ O ₈ Recovery (%)	Estimated U ₃ O ₈ Recovered (g)	Final Precipitate		Precipitate Slurry emf (mv)
		Volume (l)	pH	emf* (mv)	Assay (g/l)					Assay (g/l)				Assay (%)		
					U ₃ O ₈	Fe ⁺²	Fe ⁺³	Fe(tot)		U ₃ O ₈	Fe(tot)			U ₃ O ₈	Fe	
85-4	Aluminum reduc'n - phosphate ppt'n	400	1.90	+290	3.36	4.36	0.29	4.65	1.38	1.78	-	47.0	632	34.47	-	-85
86-1		375	2.02	+265	3.25	5.15	0.79	5.94	1.83	0.11	-	96.6	1178	39.92	-	-110
-8		375	1.62	+292	3.29	5.14	0.46	5.60	1.70	0.34	-	89.7	1107	51.49	-	-70
87-1		390	1.68	+278	4.26	6.26	1.00	7.26	1.70	0.038	-	99.1	1646	51.67	-	-100
6		390	1.75	+292	6.76	4.70	0.45	5.15	0.76	0.042	-	99.4	2619	53.23	-	-160
86-13		Iron reduc'n - MgO "90" neut	400	1.60	+300	3.39	6.44	0.85	7.29	2.15	0.022	6.00	99.4	1347	14.00	11.38
85-1	Neutralization with Mg(OH) ₂	400	1.80	-	2.34	5.43	0.69	6.12	2.62	0.009	3.96	99.8	932	8.18	11.78	
2		400	1.72	+300	2.90	4.74	0.31	5.05	1.74	0.015	3.52	99.7	1154	9.28	6.10	
3		400	1.70	+300	3.73	5.32	0.34	5.66	1.52	0.012	-	99.7	1491	10.69		
5		400	1.95	+280	3.12	4.08	0.21	4.29	1.38	0.015	-	99.5	1242	10.00	7.20	
6		400								0.010	-			10.98		
7		380	2.10	+280	3.11	3.70	0.51	4.21	1.35	0.019	2.45	99.4	1174	11.49	7.40	
8		400	1.89	+315	3.01	4.64	0.44	5.08	1.69	0.018	-	99.5	1197	10.02	8.40	
86-2		400	1.90	+272	2.89	4.36	0.34	4.70	1.63	0.070	-	97.6	1128	11.53	7.30	
3		400	1.82	+290	3.10	5.00	0.28	5.28	1.70	0.008	-	99.7	1237	10.17	8.90	
4		400	1.65	+285	3.62	5.17	0.28	5.45	1.51	0.011	0.68	99.6	1444	14.81	12.1	
86-5	Neutralization with MgO "90" neut	380	1.89	+295	3.08	4.38	0.64	5.02	1.63	0.006	1.90	99.8	1168	14.81	12.1	
6		400	1.88	+293	3.65	4.97	0.37	5.34	1.46	0.007	-	99.8	1457	18.62	9.50	
7		390	1.70	+325	3.08	4.86	0.64	5.50	1.79	0.083	-	97.4	1169	14.60	10.4	
9		400	1.58	+315	3.16	4.94	0.85	5.79	1.84	0.25	2.66	92.1	1164	9.76	11.0	
10		400	1.62	+298	3.29	5.16	0.80	5.96	1.81	0.11		96.7	1272	15.83	6.45	
11		400	1.50	+335	3.36	4.58	0.95	5.53	1.65	0.060		98.2	1320	15.36	9.65	
12		350	1.62	+345	3.88	5.85	1.68	7.53	1.94	0.005	2.94	99.9	1357	8.32	13.35	
14		400	1.72	+320	3.21	5.42	0.95	6.37	1.98	0.15	4.60	95.4	1224	20.90	8.60	
87-2		380	1.70	+278	6.13	5.38	0.81	6.19	1.01	0.012	4.20	99.8	2325	30.45	7.90	
3		385	1.72	+275	6.70	4.64	0.45	5.09	0.76	0.014	3.76	99.8	2575	32.83	9.30	
4	390	1.78	+230	6.73	4.80	0.45	5.25	0.78	0.074	4.14	99.8	2595	30.28	8.13		
5	405	1.80	+270	6.79	5.26	0.48	5.74	0.85	0.027	3.76	99.6	2740	40.11	5.09		
87-7	Neutralization with CX-17 MgO	385	1.71	+295	7.02	4.92	0.47	5.39	0.77	0.10	4.04	98.6	2662	40.38	5.68	
8		380	1.78	+310	6.06	5.30	0.53	5.83	0.96	0.028	3.86	99.5	2293	36.46	8.09	
85-9		360	1.75	-	2.92	4.60	0.44	5.04	1.73	0.002	-	99.9	1050	19.36	18.0	
10		360	1.90	-	2.56	4.60	0.42	5.02	1.96	0.004	2.00	99.8	920	14.06	21.0	

* The electro-motive force in millivolts was measured by a platinum-calomel electrode assembly.
A positive reading indicates an oxidizing potential, a negative reading a reducing potential.

0.5 g P_2O_5 /g U_3O_8 plus 0.2 g/l excess. In the first four tests the presence of phosphoric acid during reduction resulted in a slow reduction rate. This was avoided, in Test 87-6, by adding the phosphoric acid after the reduction was completed.

(3) Iron Reduction-Magnesia Neutralization

This method, as used in Test 86-13, involves the use of metallic iron filings to reduce the ferric iron in the solutions prior to adding dry MgO "90" powder to pH 6 for precipitation. There was no pH control employed during reduction. The amount of iron added was equivalent to 0.5 times the ferric iron in solution plus 2 g/l excess. The unreacted iron was not removed.

DISCUSSION OF RESULTS

The highest grade product produced by these methods, was the precipitate produced by the aluminum reduction method. This precipitate is also the only product of those made by direct precipitation in this study which is acceptable to the Port Hope refinery of Eldorado Mining and Refining Ltd. at the present time (See Appendix 2). However, the high cost (20 cents-30 cents/lb U_3O_8) of this method compared to that of ion exchange (12 cents/lb U_3O_8) makes the process unattractive. The low-grade precipitates produced in Tests 85-4 and 86-1 were thought to be due to incomplete reduction of the solution.

The test work indicates that the CX-17 magnesia is more reactive than the MgO "90" magnesia. When neutralizing similar solutions, the MgO "90" consumption is about two and a half times that of the CX-17.

It was also shown by the test work that the reactivity of both the MgO "90" and the CX-17 is greatly improved if the reagent is added as a slurry rather than dry.

REFERENCES

1. H.H. McCreedy and W.A. Gow - Pilot Plant Tests on Ore from the Kitts Property of British Newfoundland Exploration Limited, Newfoundland. Mines Branch Investigation Report IR 58-139, Department of Mines and Technical Surveys, Ottawa, August 8, 1958. (Industrial Confidential)
2. A.J. Gilmore and W.A. Gow - Ion Exchange Treatment of Solution Produced in Acid Leaching of Uranium Ore from the Kitts Property of British Newfoundland Exploration Limited, Newfoundland. Mines Branch Investigation Report IR 58-163, Department of Mines and Technical Surveys, Ottawa, August 28, 1958. (Industrial Confidential)
3. V. McNamara, R. Simard, and W.A. Gow - Solvent Extraction Treatment of Solution Produced in Acid Leaching of Uranium Ore from the Kitts Property of British Newfoundland Exploration Limited, Newfoundland. Mines Branch Investigation Report IR 58-169, Department of Mines and Technical Surveys, Ottawa, September 30, 1958. (Industrial Confidential)

VFH/WAG/dm

APPENDIX 1TABLE 5Reagent Information

Reagent		Source	R.A. Division Sample No.	Approximate Cost* cents/lb
Magnesia	Mg(OH) ₂	Dow Chemical of Canada Ltd., Montreal, Que.	3/56-9	6.0
	MgO "90"	Aluminum Company of Canada Ltd., Wakefield, Que.	6/58-12	6.0
	CX-17	Aluminum Company of Canada Ltd., Wakefield, Que.	6/58-25	7.5
Metallic Aluminum Powder	Al	Canadian Bronze Powder Works Ltd., Montreal, Que.	-	50
Phosphoric Acid	H ₃ PO ₄	Regular C.P. Grade	-	10
Metallic Iron Powder	Fe	Belmont Smelting and Refining Works Inc., Brooklyn, N.Y.	3/56-21	10

* Estimated cost of technical grade reagent
delivered to the mine site.

APPENDIX 2CORRESPONDENCE RELATING TO ACCEPTABILITY
OF VARIOUS PRECIPITATESRadioactivity File

30 Lydia Street,
Ottawa, Ontario,
19 June, 1958.

Mr. J. Burger,
Manager,
Eldorado Mining and Refining Limited,
Port Hope, Ontario.

Dear Jack,

We are carrying out pilot plant work on a sample from the British Newfoundland Exploration Ltd., property in Labrador. They have made enquiries to Dick Barrett regarding product acceptability, and he has asked us by phone to send you samples of product, as we have done on some other ores.

We have 4 types of product.

	<u>Approx. U₃O₈ Assay</u>
1. Precipitate from MgO neutralization of the leach solution	10-15%
2. Aluminum reduction-phosphate precipitate	50%
3. Solvent extraction NaOH neutralization precipitate	85%
4. Ion exchange MgO neutralization precipitate from a chloride eluate	70%

What would be the minimum amounts you would have to have for your acceptability tests? We have some of each product on hand now which can be sent to Port Hope on instructions from you.

As a matter of record, which type of product would be more acceptable by the refinery under present circuit conditions. Some information in this regard would be helpful in directing our test work.

Yours very truly

H. W. Smith
Acting Chief, Radioactivity Division.

R. Ennis
Dr. Beavan

ELDORADO MINING AND REFINING LIMITED

Refining Division

Port Hope, Ontario.

June 27, 1958.

Mr. H.W. Smith, Acting Chief,
Radioactivity Division,
Department of Mines and Technical Surveys,
30 Lydia Street,
Ottawa, Ontario.

Dear Harold:

I have for reply your letter dated June 19th requesting information on the precipitate from the British Newfoundland Exploration Ltd.

According to the published price schedule, the precipitate, or feed material, must be amenable to the Port Hope Refinery process and, on this basis, our choice order would be as follows:

	<u>Approx. U₃O₈ Assay</u>
1. Solvent extraction NaOH neutralization precipitate	85%
2. Ion exchange MgO neutralization precipitate from a chloride eluate	70%
3. Aluminum reduction-phosphate precipitate	50%

The precipitate from MgO neutralization of the leach solution analysing 10 to 15% U₃O₈ would not be considered as a suitable product. This is based on the high acid consumption and the dilution effect with subsequent problems associated with digestion. Furthermore, because of the type of product, it would be necessary for us to reduce refinery entry due to the overloading of our extraction circuit with this material. This, of course, would result in reduced production and subsequently higher costs.

Probably the greatest difficulty encountered would be the emulsification tendencies shown with this type of material and this would, of course, render our circuit inoperable.

It should also be pointed out that moisture content of any product must be less than 10% and the total halogen should not exceed 0.2%.

In order to carry out test work at Port Hope, we would require a minimum of five pounds of product.

From past experience it is known that products, other than the No. 1 in your letter, would be suitable for refinery entry, providing they met the moisture and halogen requirements outlined above. This however cannot be completely confirmed until a check is made using the five pounds of material as a basis for study.

I trust that this gives you a general outline of our requirements. Please contact us further if there are any additional questions.

Yours truly,

ELDORADO MINING AND REFINING
LIMITED,

J.C. Burger
Manager.

JCB:js

cc: WMGilchrist
GCBrown