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CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS
MINES BRANCH

DEVELOPMENT OF THE PORT RADIUM LEACHING
PROCESS FOR RECOVERY OF URANIUM

RADIOACTIVITY DIVISION

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DEVELOPMENT OF THE PORT RADIUM LEACHING PROCESS FOR
RECOVERY OF URANIUM*

By the Staff

Radioactivity Division

INTRODUCTION**

The mines at Port Radium have been in operation for over 20 years with a short break during the early part of the war. The ore in the upper levels was rich and the pitchblende heavy; concentration by jigs and tables could be carried out with fair success in spite of the dilution of the concentrate by many heavy metallic minerals. Thus the Port Radium mill was for many years the only plant in Canada that recovered values by gravity concentration only.

As the mining operation reached lower levels the character of the ore changed. More of the pitchblende occurred in a finely disseminated form which was difficult or impossible to recover by the existing gravity plant. This situation existed in 1946 when a small group was assembled at the Bureau of Mines to work under the direction of Dr. C.S. Parsons towards better recovery of the uranium. This was the origin of the "Eldorado Project", which later grew to the Radioactivity Division of the Mines Branch. Port Radium was at that time the only producing uranium mine in Canada.

* Presented at the Annual Western Meeting, 1955, of the Canadian Institute of Mining and Metallurgy, by permission of the Acting Deputy Minister, Department of Mines and Technical Surveys, Ottawa.

** Introduction prepared by A. Thunaes, now Director of Research and Development, Eldorado Mining and Refining Ltd.

A systematic study was made to improve the methods of gravity concentration and modern equipment was installed. Soon it became apparent that even the most efficient of gravity concentration methods would fall far short of a really satisfactory recovery.

Flotation of Port Radium's pitchblende was tried through an extensive series of tests, but efficient concentration by this method was not found feasible.

By 1947 it was clear that leaching methods must be used to obtain a satisfactory recovery of uranium and the research work was organized along these lines.

At this time, as later, there was close co-operation between the Canadian group and similar groups working on treatment of uranium ores in Great Britain and the United States. Professor A.M. Gaudin who directed uranium ore research at the U.S.A.E.C. laboratories in Watertown, Mass., was also a consultant to the Canadian group in Ottawa until 1948, and his advice was of great value to the progress of the work.

It was known by 1947 that uranium could be extracted from pitchblende ores by leaching with sulphuric acid when certain oxidants were present during the leach. The methods then available for recovery of uranium from leach solutions were of some value for "clean" ores but unsuitable for use with the "dirty" solutions resulting from leaching Port Radium ore. Iron, aluminum, arsenic, magnesium, manganese, copper, nickel and cobalt were dissolved in appreciable quantities during leaching.

The testwork on leaching soon established that good extraction of uranium could readily be obtained but that the cost of reagents was the main barrier to economic success. The leaching of the total ore, while feasible, was not attractive due to excessive reagent requirements. The decision was made to retain the gravity mill and to leach the gravity tailings which contained somewhat less impurities.

The location of the plant necessitated the use of an oxidizing reagent that provided a maximum amount of oxidizing power per pound and per dollar cost. Similarly, the reagents used for precipitation must be selective, reasonably cheap and the quantity used per ton ore should be low.

The acidity must be kept near the minimum consistent with good extraction in order to avoid excessive reagent cost, but too low an acidity in the final leachers might promote reprecipitation of uranium, and pH control within a narrow range was required for the complete leaching circuit.

Several methods were tried for precipitating uranium from the acid solutions. The method finally selected was very simple in principle and appeared particularly well suited for the impure solution and for the remote locality.

How the details of the leaching and precipitation processes were developed is explained in more detail later.

The development of analytical methods for uranium is a very important chapter in the history of uranium research. As late as 1948 we did not have reliable methods for determination of uranium in low-grade residues or barren solutions and the known methods were

time consuming.

The greatest advance in the field of uranium analysis was the development of fluorimetric methods, these became available in 1949 and resulted in a great speed up in metallurgical test work. Simultaneous advances were made in the radiometric analysis and in methods for mineralogical study of uranium compounds.

The laboratory-scale batch-leaching tests were completed in 1948. These tests had included separate leaching of flotation concentrate (sulphides and arsenides) and of flotation tailing, leaching of gravity tailings and leaching of mill feed. The leaching of gravity tailings was chosen as the most suitable system and preparations were made for pilot-plant operation on a small scale in the Mines Branch Laboratories.

The Mines Branch pilot-plant run was completed in 1949; the feed to this plant consisted of current gravity tailings plus reclaimed tailings from earlier mill operation. The feed rate was 1000 lbs/day. This pilot plant confirmed the batch-test results and gave valuable information for design of a leaching plant.

A larger pilot plant (8 tons/day capacity) was constructed at Port Radium and operated by Mines Branch and Eldorado staff during the summer and fall of 1950. The purpose of this plant was to test the behaviour of the ore from day to day, to train control operators and obtain design data for a full-scale plant.

The present Port Radium leaching plant was designed in 1950-51; the construction was started in the summer of 1951 and completed in May 1952.

At the start of its operation the Port Radium leaching plant had an efficient process for sulphuric acid leaching of uranium, in spite of the complex ore. This does not mean that there were no operating difficulties to iron out, particularly in filtration of the acid pulp. The gradual improvements made during operation and the problems specific to the location of the plant will be described in another paper.*

The gravity mill, incidentally, is still part of the operation. Having been destroyed by fire in late 1951 the mill was rebuilt and ready for operation at the same time as was the leaching plant. A considerable part of the leach plant feed is obtained by reclamation of stored tailing through an all-year dredging operation.

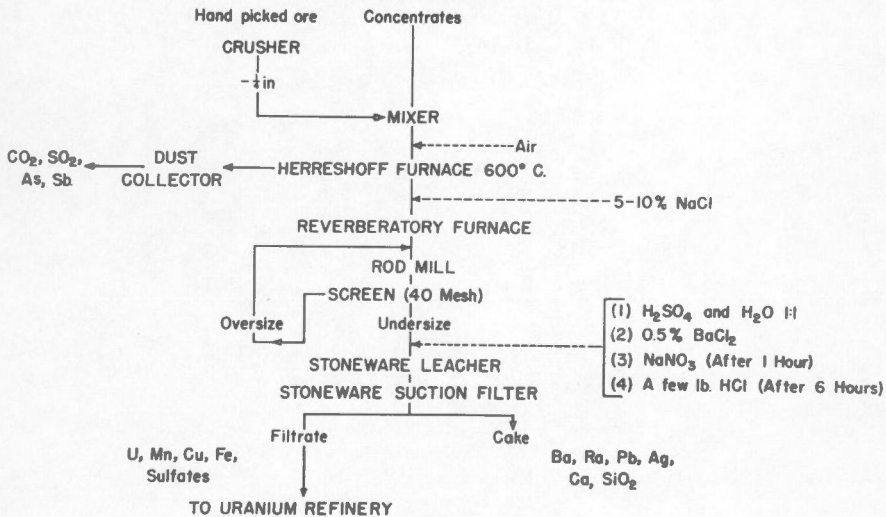
* Leaching of Uranium from Gravity Mill Tailings at Port Radium, N. W. T., by D.F. Lillie and R. Tremblay

CHEMISTRY OF LEACHING *

The leaching process at Port Radium is based upon extraction of uranium by treatment of the ore with weak unheated sulphuric acid solutions in the presence of an oxidizing agent.

The treatment of uranium ore with hot strong acids is not new, and, in the days before uranium acquired its present importance, it was in use at the Port Hope refinery for treatment of pitchblende concentrates from Great Bear Lake. Processing of these concentrates involved roasting to remove arsenic, etc., followed by a hot digestion with sulphuric acid of about 50% strength, using sodium nitrate as oxidising agent. The process in use at the time is illustrated in Figure 1.

PORT HOPE (ONTARIO) REFINERY. Flow Sheet



E F MACTAGGART - THE INDUSTRIAL CHEMIST - VOL 18 P 421-426 (1942)

Figure 1

* By E.A. Brown

The same sort of treatment would certainly leach uranium from the Port Radium gravity tailings, but would simply be too costly to use on low-grade material. The grade of these tailings would not bear the expense of roasting, high reagent concentrations and hot leaching.

However, close attention to the effects of other constituents of the ore and careful study of leaching conditions has made it possible to operate on the same basic principle and to treat the Port Radium gravity tailings successfully without prior roasting, and leaching only with unheated weak sulphuric acid solution plus oxidizing agent rather than a hot stronger acid digestion.

The purpose of this paper is to present the major points in the chemistry of this leaching process as used on Port Radium tailings.

Important Impurities in the Ore

Among the constituents of the ore apart from uranium, are arsenic and phosphorus, both of which have a marked influence on the chemical conditions required for good leaching.

Both arsenate and phosphate will precipitate uranium quite readily in weakly acid solutions. This was illustrated by the addition of arsenate and phosphate to pure uranyl sulphate solutions.

Tolerance of Uranyl Sulphate Solution for Arsenate

To portions of uranyl sulphate solution containing the equivalent of 1.25 gm U_3O_8 per litre was added sulphuric acid to bring the pH to the initial values shown below. Sodium arsenate solution, containing 18.9 grams arsenic per litre was added until a noticeable precipitate formed, and the pH at which this occurred was noted. The amount of arsenic added is expressed in equivalents, an equivalent being

the amount required to form $\text{UO}_2 \text{HASO}_4$ with the uranium in the sample.

Table 1

Tolerance of Uranyl Sulphate Solution for Arsenate

pH of uranium solution		Arsenic added to precipitation point, (equivalents)
Initial	At precipitation point	
1.0	2.1	25.8
1.5	2.0	4.5
2.0	2.15	0.45

Tolerance of Uranyl Sulphate Solution for Phosphate

The effect of phosphate in solution is similar to the effect of arsenate. This is illustrated by the results of the following tests, which were carried out in a somewhat different manner than the arsenate tests.

To samples of uranyl sulphate solution of pH 0.9, containing the equivalent of 1 gram U_3O_8 per liter, were added varying amounts of phosphoric acid solution pH 1.7 and containing 2.53 gm P_2O_5 per liter. Sodium hydroxide was added when necessary to adjust the pH to the values shown in Table 2. The amount of P_2O_5 added is expressed in equivalents, an equivalent being the amount corresponding to formation of $\text{UO}_2 \text{HPO}_4$ with the uranium in the samples.

For minimum acid consumption, the ore should be leached at as high a pH as possible. The data in Table 2 show, however, that if soluble arsenates and phosphates are present, the pH during leaching should be kept below a value of 2 in order to prevent reprecipitation of dissolved uranium.

Table 2

Tolerance of Uranyl Sulphate for Phosphate		
pH	Equivalents of P ₂ O ₅ added	Precipitation
1.25	37.5	none
1.7	1.5	none in 24 hours
1.8	1.5	none in 24 hours
1.9	1.5	after 2 hours
1.9 (diluted 2X)	1.5	none in 5 hours
2.0	3	immediate
2.0	1.5	after 2 hours
2.0	1	none in 7 hours
2.1	2	immediate
2.1	1.5	immediate

Need for Oxidizing Agent

In pitchblende a large proportion (perhaps 1/3 or more) of the uranium is present in the uranous or tetravalent state, corresponding to the oxide UO₂, while the remainder is present in the uranyl or hexavalent state, corresponding to the oxide UO₃. Pure uranyl oxide UO₃ will dissolve completely in dilute sulphuric acid alone. Uranous oxide UO₂ on the other hand does not dissolve at a useful rate unless an oxidizing agent is present to oxidize the uranium from the uranous to the uranyl form.

This is illustrated by leaching tests on an essentially pure sample of UO₂. In each test 1 gram of UO₂ was agitated with 20 cc of 5% H₂SO₄ for 24 hours at room temperature:

Table 3

Effects of Chlorate and Ferric Iron on
Extraction of Uranium from UO_2 .

	Oxidizing agent added (% of theoretical)*		Uranium dissolved (% of total)
	$NaClO_3$	$Fe_2(SO_4)_3$	
Test 1	nil	nil	1.3
Test 2	nil	150	89.6
Test 3	150	nil	4.5
Test 4	100	10	100.0

* based on chemical equations listed in Table 4.

An interesting point apparent here is the marked influence of a small amount of iron on the effectiveness of the chlorate. It appears that the uranium is actually oxidized by reaction with the ferric iron and that the chlorate maintains the oxidation of the iron.

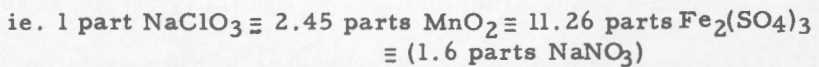
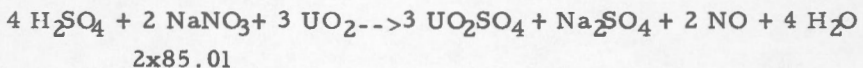
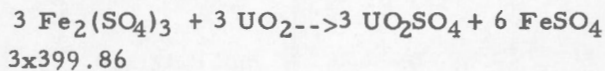
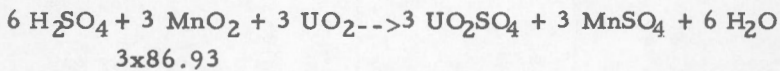
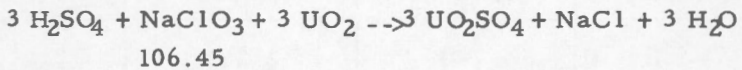
An oxidizing agent is also required to keep the uranium in an oxidized condition once it has been taken into solution. If an oxidizing condition is not maintained then dissolved uranium may be reduced from hexavalent back to the tetravalent or uranous state by reducing agents such as metallic iron introduced in grinding the ore. When arsenate and phosphate are present, these can reprecipitate dissolved uranium as insoluble uranous arsenate and phosphate unless an oxidizing condition is maintained. Uranous arsenate and phosphate are much less soluble than uranyl arsenate and phosphate and will precipitate practically completely at pH values where hexavalent uranium remains soluble.

The difference between the solubilities of uranous and uranyl arsenates and phosphates is the basis of the process used for recovery

of uranium from Port Radium leach liquors. The precipitation process is described in more detail later.

There are several oxidizing agents which may be employed in acid leaching uranium ores. Sodium chlorate was chosen for Port Radium because it is one of the more powerful reagents and will readily maintain the oxidizing condition that is required in the presence of arsenates and phosphates. On a weight for weight basis it is also one of the most concentrated reagents available for this purpose. This of course is an important consideration in the cost of reagents delivered to remote localities.

Table 4
Oxidizing Agents



These equations indicate that in dissolving the same amount of UO_2 the oxidation reaction with sodium chlorate consumes only half as much acid as the oxidation reaction using MnO_2 . The oxidizing reaction using ferric sulphate should consume no acid, but the amount of ferric sulphate required more than offsets the acid consumed by the chlorate reaction. An equation is shown for the sodium nitrate as an oxidizing

agent but this reaction does not take place without heating, as will be evident later.

Leaching of a sample of Port Radium ore without pH control and without oxidizing agent illustrates what happens under such conditions:

Table 5

Effect of Leaching Port Radium Ore
without pH Control or Oxidizing Agent

Sample E71

Leached with 2 parts 5% H₂SO₄ to 1 part ore (200 lb H₂SO₄/ton ore).

No oxidizing agent.

pH not controlled and allowed to rise.

Leach time hrs	Leach pH value	Leach liquor composition		% extraction of U ₃ O ₈
		As lb/ton ore	U ₃ O ₈ lb/ton ore	
1	1.1	1.0	2.26	43.7
4	2.0	1.0	1.46	34.8
16	3.25	1.0	0.04	0.84
24	3.5	0.4	not detected	nil

Some of the uranium dissolved initially but began to reprecipitate at once even at pH values below 2 because of the lack of an oxidizing condition. With continued leaching, the pH continued to rise to a point where uranyl arsenates and phosphates also became insoluble, until ultimately all the uranium initially dissolved was reprecipitated.

In leaching a sample of the ore at room temperature with pH controlled at 1.5 but without oxidizing agent, close to 60% of the uranium was extracted:

Table 6

Effect of Oxidizing Agent in Leaching Port Radium Ore
at Controlled pH

Assays of ore sample:

U ₃ O ₈	As	P ₂ O ₅	CO ₂	Fe	S
0.22%	0.24	1.14	3.41	4.55	1.01

Grind- 67% minus 200 mesh.

Leach time hours	Test 2-587		Test 2-584		Test 2-585	
	Acid only, no oxidizing agent		NaNO ₃ 9.6 lb/ton ore		NaClO ₃ 6 lb/ton ore	
	Residue % U ₃ O ₈	% Extr. U ₃ O ₈	Residue % U ₃ O ₈	% Extr. U ₃ O ₈	Residue % U ₃ O ₈	% Extr. U ₃ O ₈
6	0.10	54.5	0.091	58.6	0.030	86.3
12	0.093	57.7	0.092	58.2	0.020	90.9
18	0.091	58.6	0.089	59.5	0.015	93.2
24 3/4	0.092	58.2	0.081	63.2	0.014	93.6

Leaching another portion of the same sample under the same conditions, but with the addition of sodium nitrate, gave much the same result as no oxidizing agent since sodium nitrate is not effective as an oxidizing agent without heating. However with pH control and with the addition of sodium chlorate as oxidizing agent, 93.6% of the uranium was extracted. The 6 lb of sodium chlorate added would be the theoretical equivalent of 9.6 lb of sodium nitrate if the nitrate did react in the cold according to the equations shown in Table 4.

When there is a relatively high content of soluble arsenic and phosphorus in the leach feed, the presence of ferric iron is beneficial for an additional reason. It appears that by its ability to

"complex" arsenate and phosphate, the presence of ferric iron can be utilized to minimize reprecipitation of uranyl arsenates and phosphates, thus improving extraction.

Leaching tests to illustrate this point were carried out on a sample having the following head assays:

	<u>U₃O₈</u>	<u>CO₂</u>	<u>As</u>	<u>P₂O₅</u>
%	0.58	2.72	1.56	1.02

In each case the leach feed had the following screen analysis:

	<u>+100 mesh</u>	<u>+325 mesh</u>	<u>-325 mesh</u>
%	2.9	34.6	62.5

The total amount of oxidizing agent used in each test was equivalent to 14 lb NaClO₃ per ton ore, but over the series of tests ferric sulphate in increasing amounts was substituted for equivalent amounts of NaClO₃. All leaches were for 24 hours, at 20°C, at a controlled pH value of 1.35 and at an initial pulp density of 3 parts solution to 4 parts ore.

The leach liquors obtained were analysed for ferric iron, arsenic and phosphate. The total ferric iron less the amount necessary to form FeAsO₄ and FePO₄ with the arsenate and phosphate present was designated "free" ferric iron.

There was an evident correlation between the concentration of "free" ferric iron in the leach liquor and percentage extraction of uranium. Maximum uranium extraction resulted when the "free" ferric iron concentration reached a level about 4 gm/l, as shown in Table 7 and Figure 2.

Table 7

Effect of "Free" Ferric Iron on Extraction

"Free" ferric iron in leach liquor gm/l	% Extraction	
	As	U ₃ O ₈
1.43	8.6	44.2
2.10	7.8	52.3
2.06	8.6	67.6
2.72	10.8	81.2
3.35	9.5	86.8
4.37	14.7	87.5
6.02	15.8	89.6
10.88	24.4	90.0

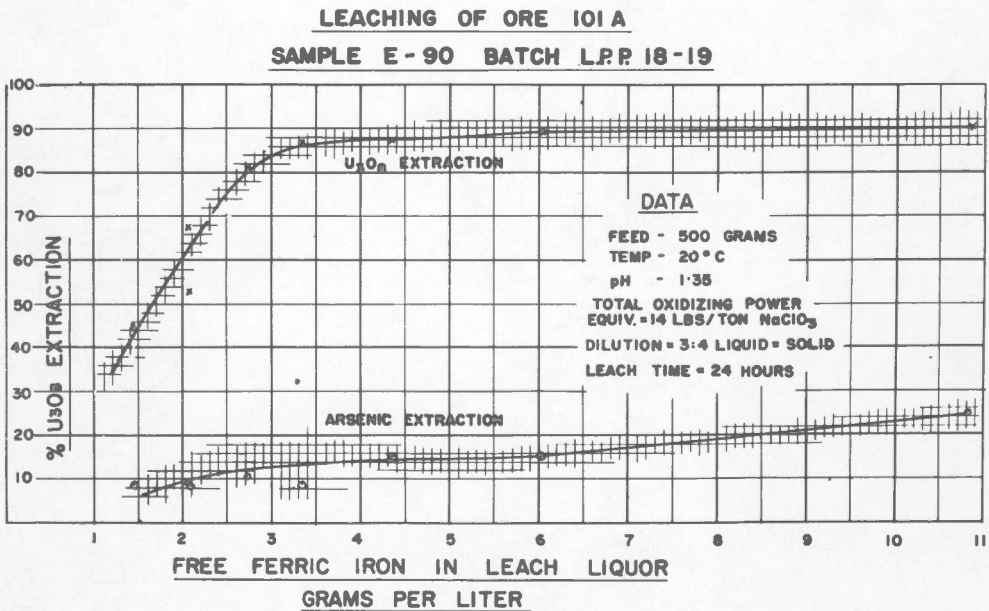


Figure 2 - Graph showing Effect of "Free" Ferric Iron on Extraction

The indicated increase in arsenic extraction is no doubt due to effect of increasing ferric iron in preventing reprecipitation of the dissolved arsenic, which could otherwise reduce uranium extraction by reprecipitating uranyl arsenate.

CHEMISTRY OF PRECIPITATION *

When the sulphuric-acid leach process was developed for Port Radium ore, the problem of uranium recovery from solution was investigated at the same time with the purpose of making a high grade product by simple and economic means. The better known procedure of precipitation of uranium as the hydroxide was first studied but proved unsuitable due to the high ferric iron content of the solution. Reasonable grades could be obtained only by retreating the first precipitate or by neutralizing in two stages to separate the bulk of the iron from the final product. In the course of this investigation it was found that reduction of the ferric iron in solution by aluminum powder would bring down the uranium selectively as a mixture of uranous arsenate and phosphate, these two ions being present in the natural liquor. Further test work showed that this reduction could be a simple process that would produce a 40 to 50% U_3O_8 precipitate at a recovery of 98-99% from solutions assaying 1 to 2 gm U_3O_8 / liter. The remainder of this paper is devoted to the chemistry of the aluminum reduction process as applied to Port Radium leach solutions.

Chemistry

Pregnant solution from the leach circuit after clarification is a complex weak sulphuric acid solution of pH 1.5 to 1.8 assaying typically as follows:

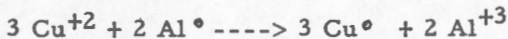
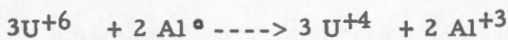
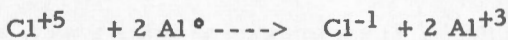
U_3O_8	-	1.5-2.0 gm/l
Ferric iron	-	3.0-5.0 "

* By R. Simard

Ferrous iron	-	0.0-0.5 gm/l
NaClO ₃	-	0.0-0.3 "
As ⁺⁵	-	0.75-1.0 "
P ₂ O ₅	-	0.20-0.25 "
Cu ⁺²	-	0.10-0.50 "
Co ⁺²	-	0.20-0.30 "
F ⁻¹	-	1.0 - 1.0 "
Cl ⁻¹	-	0.5 - 1.0 "

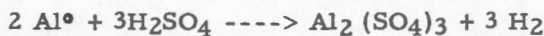
The oxidation-reduction potential is in the order of + 400 millivolts as measured by a platinum-calomel electrode assembly.

Upon the addition of a reducing agent such as aluminum powder the chlorate, ferric, cupric and uranyl ions are reduced to chloride, ferrous, copper metal and uranous ions as represented below:



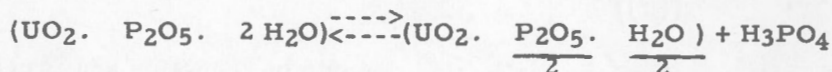
Bismuth which is sometimes present in the solution will also reduce to the metallic form.

Part of the aluminum powder is also dissolved by the acid, producing hydrogen according to the reaction:



In the course of this reduction the uranous ion precipitates as the arsenate or phosphate. The compounds have been tentatively identified as a mixture of two acid salts having one and two mols of

arsenate or phosphate per mol of UO_2 . The proportion of each is dependent on the final pH of the barren solution and the equilibrium may be represented as:



Controlling Factors

The factors affecting the rate of reduction are mainly pH, temperature, and aluminum concentration. To obtain effective reduction with a minimum of reducing agent it was found that a pH range of 1.5 to 2.0 was optimum. The pregnant is thus maintained at pH 1.5 to 1.75 for the first 4 to 6 hours with concentrated sulphuric acid. Most of the aluminum is consumed at that time and near-complete precipitation is obtained in 6 to 12 hours. No control is required after this stage, so that pH will rise to 2.0 or higher.

Temperature will affect rate of reduction. Below 20°C the reaction is slowed down and to obtain good precipitation a lower pH must be maintained. Above 30°C the reverse is true and excessive consumption of aluminum will occur unless the pH is controlled at a higher point of 1.75 to 1.85.

Aluminum is added as a fine flake powder produced for the aluminum paint industry, in a dry form or as a paste in varsol. The paste is not readily wetted by the acid solution and a wetting agent is incorporated for this purpose. The amount of reductant required is based on the oxidizing power of the pregnant, expressed as ferric iron or sodium chlorate. These are determined for each batch and the equivalent aluminum calculated. To this is added an empirical excess of 0.5 to 0.6 gm/ liter so

that the total charge will amount to 1.0 to 1.3 gm/liter of pregnant or approximately 3 lb per ton of ore. The batch is then agitated at minimum speed to keep the powder in suspension and avoid air entrainment.

One factor affecting the rate of reduction and precipitation is the concentration of certain ions. Chlorides and fluorides are known to accelerate the attack on the aluminum; this may not be beneficial if it favours the formation of hydrogen alone. But certain cations such as copper, nickel and cobalt have been shown to improve reduction of the uranyl ion and since these are present in the liquor in small concentrations they are beneficial. A concentration of 0.1 gm Cu^{+2} /liter, as is found in the Port Radium solutions, reduced precipitation time threefold when added to a pregnant containing no copper.

The phosphate-arsenate concentration in solution is normally sufficient to precipitate all the uranium present, but for some periods in the plant the required ratio for $\text{As} + \text{P}_2\text{O}_5 / \text{U}_3\text{O}_8$ of 0.5 to 1.0 by weight will not be met and the difference is made up with monocalcium phosphate added directly to the batch. The barren is usually tested for low phosphate-arsenate by adding a drop of phosphoric acid to a clear barren; the appearance of uranous phosphate is conclusive and the monocalcium salt is added to the precipitation tank as required.

As may be seen these various controlling factors may affect the total time required for good recovery. From 6 to 12 hours is normal, but longer periods up to 24 hours are required in some cases. A rapid U_3O_8 determination by the peroxide-colorimetric method is done in the plant to control these conditions.

Recovery

From 98 to 99% of the uranium is recovered from solutions assaying 1 to 2 gm U_3O_8 / liter and barrens of 0.01 to 0.03 gm/ liter are discarded after settling and filtering.

Product

The filtered product is made up largely of uranous arsenate and phosphate with some copper metal and residual aluminum. Minor constituents are silica, bismuth and occluded soluble salts of iron, manganese, magnesium and other cations in the sulphate form.

A typical precipitate dried at 110°C assays:

	U_3O_8	As	P_2O_5	Cu	Al	S/ SO_4	L.O.I.
%	44	10.0	6.0	3.5	4.6	2.50	16.0

SUMMARY

Three of the main factors in the chemistry of leaching gravity tailings at Port Radium are:

- (1) sufficient acidity is maintained to dissolve the uranium and to keep the uranyl arsenates and phosphates in solution. This acidity does not have to be high and the leach pulp does not have to be heated. A pH value of about 1.5 is satisfactory for leaching of the tailings, and gives a leach liquor with suitable acidity for subsequent precipitation operations.
- (2) an oxidizing condition is maintained so that substantially all of the uranium in the ore can be dissolved by the acid, also to keep the dissolved uranium in the uranyl form and prevent the formation of insoluble uranous arsenates and phosphates. The oxidizing agent employed is sodium chlorate, a reagent which is effective in unheated weak acid solution.
- (3) in recovering uranium from the leach solution, the dissolved uranium is converted from the uranyl to the uranous form by treating the solutions with metallic aluminum reducing agent, whereupon the uranium is quantitatively precipitated and recovered as a mixture of insoluble uranous arsenate and phosphate.

**EDMOND CLOUTIER, C.M.G., O.A., D.S.P.,
Queen's Printer and Controller of Stationery, Ottawa.**