

SUMMARY REPORT

FOR PERIOD ENDING AUGUST 15, 1949

Introduction

Samples of ore concentrate, calcine and speiss were brought to Department of Mining and Metallurgy early in June 1949 by Messrs. J. M. Taylor and R. R. Taylor. These samples were residual from investigations carried out at U.B.C. during the period 1942-1944. This earlier work included reports on the geology, concentration and pyrometallurgy of the Little Gem Ore and may be found in the following references:

1. Investigation of Treatment Methods for Cobalt Ore from Gem Mine. - Raymond R. Taylor - M.A. Sc Thesis 1941 - U.B.C.
2. Investigations into Gold and Cobalt Recovery from Little Gem Ore. - Farmer and Bell - 1V Year Problem - 1942.
3. B.C. War Metals Board - Project #RM-8 - General Summary Report on Treatment of Cobalt Ores - 1943.
4. Metallurgy of Low Grade Cobalt Ores from Little Gem Mine. - Currie and Goodman - 1V Yr. Problem - 1944.
5. Cobalt Extraction from Little Gem Ore by Ammonia Leaching. - Robert Johnson - 1V Year Problem - 1948.

Recent developments in the pressure leaching of nickel ores in ammoniacal solutions have suggested that the Little Gem cobalt ore might be treated by a similar process.

The renewed interest in the treatment of this ore has been due, in part, to the discovery of economic quantities of uranium oxide in the deposit. Any method of treatment for cobalt and gold extraction should also concentrate the radioactive constituents and make them available for subsequent separation.

Robert McIntosh, a graduate chemical engineer carried out preliminary tests and constructed necessary equipment during the month

of June. On July 1st a second chemical engineer was employed on this project - Brian Carrigan. These men have been employed continuously on the investigation to the date of writing. A graduate metallurgist, Walter K. Congreve has given valuable assistance and a considerable part of his time to the instruction of McIntosh and Carrigan in the necessary metallurgical techniques and has carried out numerous pressure leach tests to assist the progress of the investigation.

Materials Used

	Co	Fe	As	S	Au oz/Ton
Mixed Ore Concentrate (from ref. 4 above)	2.41	23.5	32.5	12.1	1.8
CALCINE (from ref. #1)	7.7	32.4	6.2	1.26	-
CALCINE SPEISS (from ref. #1)	28.1	40.2	4.3	-	-
New Ore concentrate containing Uranium (identified as GM200)	6.55	22.0	44.7	8.5	0.96

TEST PROCEDURES

The following methods of treatment were investigated:

1. Ammonia leaching of ore, calcine and speiss over a wide range of pressure and temperature.
2. Caustic leach under oxygen pressure over a range of temperature.
3. Ferric and ferrous sulphate leach under oxygen pressure over a range of temperature.
4. Acid leach under oxygen pressure over a range of temperature.

NOTE: The detailed procedures and results for individual tests are

available in the attached progress reports and will not be included in this report.

All high pressure tests were carried out in electrically heated steel autoclave vessels designed and constructed at U.B.C.

RESULTS

1. Ammonia Leach Tests:

(a) A series of tests were made under oxygen pressure of 30 p.s.i. and at temperatures ranging from 70°F. to 140°F. Under these conditions no cobalt could be recovered from the calcined ore but up to 7% of the cobalt could be extracted from the untreated ore.

A maximum recovery of 5% of the cobalt was obtained from tests on the calcine speiss.

(b) A series of tests were carried out under oxygen pressure of 200 p.s.i. and at temperatures ranging from 200 to 350°F. Recoveries of cobalt ranged from 5 to 64%. The best results were obtained when ammonium carbonate was added to the leach solution and when the ore was ground in a solution of ammonia.

Cobalt recovery from calcined ore and speiss was lower than from the ore concentrate.

Tests at Oxygen pressures of 475 - 500 p.s.i. gave no recovery of Cobalt.

2. Sodium Hydroxide Leach Tests:

A series of leach tests were carried out in 12 Norman and 18 N. sodium hydroxide solution at oxygen pressure ranging from 200 to 480 p.s.i. and temperatures from 200°F. to 500°F.

Cobalt recovery in the leach solution ranged from 0 to 54% with best results obtained at a pressure of 200 p.s.i. and temperature of 280°F.

Most of the arsenic and sulphur are removed by the caustic leach.

COMMENTS
The cobalt is recovered in a deep blue solution. When this solution is diluted with water the cobalt separates as a brown oxide.

pressure shows little promise. Tests on speiss were stopped when it was found that the sample speiss was an insoluble cobalt ferrite. The formation of cobalt oxide and if higher temperatures and pressures are used the cobalt is completely oxidized and remains in the residue.

Gold remained in the oxidized residue and was almost completely recovered with moderate consumption of cyanide.

3. FERRIC AND FERROUS SULPHATE LEACH TESTS:

Ferric Sulphate:

(a) Leach tests on ore, calcine, and oxidized residue from the caustic leach gave no recovery of cobalt. These tests were carried out at normal pressures and temperatures from 70 to 260°F.

Ferrous Sulphate:

(b) Leach tests on oxidized residues from the caustic leach and on the raw ore concentrate resulted in extraction of 70% of the cobalt.

The oxidation of the ore proceeded rapidly under these conditions and most of the iron and arsenic remained in the residue. High oxygen (300 p.s.i.) was necessary since a test at normal air pressure gave no recovery of cobalt.

A similar test was made using only water as the leach solution. Under these conditions the sulphide in the ore was oxidized to sulphate and 68% of the cobalt was recovered from the solution.

4. ACID LEACH TESTS:

A series of tests has been started using dilute sulphuric acid as the leach solution.

Tests on oxidized residues from the caustic treatment give cobalt recoveries as high as 60%.

Tests on ore concentrate at low pressures (under 30 p.s.i.) gave low recovery. Further runs are being carried out under high oxygen pressure at the date of writing.

SUMMARY

Direct ammonia leaching of ore or calcine with or without oxygen pressure shows little promise. Tests on speiss were stopped when it was found that the sample speiss was an insoluble cobalt ferrite rather than cobalt arsenide.

Caustic leaching of the ore concentrate offers a possible method of

treatment. A counterflow process could be developed in which the cobalt is removed in the intermediate blue solution stage. However the extraction by caustic is slow and very close control of leaching conditions would be necessary.

A ferrous sulphate or water leach under high oxygen pressure shows considerable promise and further tests are in progress in this direction, to determine the optimum conditions of temperature and pressure.

Results of direct acid leach tests are still incomplete.

The following development has been suggested and a few preliminary tests indicate that high cobalt recoveries may be possible:

The oxidized residue from caustic leaching (or an ore calcine) may be leached in dilute ammonia solution under hydrogen pressure. Under THESE conditions the oxides of cobalt are reduced and the cobalt dissolves in the ammonia.

Submitted by:

W. M. Armstrong.

Conclusion:

This report indicates that the use of high oxygen pressure in the leaching of cobalt ores is a promising method for increasing the extraction of cobalt. Further tests are being conducted to determine the optimum conditions of temperature and pressure. The use of dilute ammonia solution under hydrogen pressure for the leaching of oxidized residue from caustic leaching (or an ore calcine) is also a promising method for increasing the extraction of cobalt. These conditions reduce the oxides of cobalt and the cobalt dissolves in the ammonia.

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PROGRESS REPORT NO. 1

O

July 18, 1949

P

Little Gem

Y

The following bottle leaches have been tried:

1. 10 gms of ore concentrate + 500 ml. of concentrate NH_4OH for 24 hours. - No cobalt.
2. 10 gms of finely ground ore concentrate + 500 ml. of concentrate NH_4OH for 24 hrs. The solution was saturated with O_2 four times - No cobalt.
3. 10 gms of ore concentrate which had undergone 18 N. NaOH leach for $5\frac{1}{2}$ hours at 300°F . under 200 p.s.i. O_2 . This concentrate was well oxidized from this treatment. This sample was leached in NH_4OH for 24 hours - No cobalt.
4. 20 gms of tailings which had been subjected to the same conditions as #3 above was leached for 48 hours in 250 cc of H_2O + 20 gms of $\text{Fe}_2(\text{SO}_4)_3$ - Trace of cobalt.
5. 20 gms of calcine speiss containing 2.9% As, 28.2% Co and 40.5% Fe was leached for 48 hours with 250 cc of H_2O + 20 gms of $\text{Fe}_2(\text{SO}_4)_3$ - No cobalt.
6. 20 gms of calcine containing 7.7% Co, 6.2% As, 1.26% S, and 1.9% CaO was leached for 48 hours in 250 ccts of H_2O plus 20 gms of $\text{Fe}_2(\text{SO}_4)_3$ - No cobalt.

Conclusions

While no definite conclusions can be drawn, the above results do indicate that a high temperature leach with $\text{Fe}_2(\text{SO}_4)_3$ on the tailings from the caustic leach might produce some cobalt in a shorter time. The following NH_4OH low pressure (30 p.s.i.) O_2 leaches have been tried:

1. 200 gms of ore concentrate + 1500 ml. of NH_4OH was leached for 5 hours at a temperature of 20°C . Approximately 1.5% cobalt was recovered.
2. 30 gms of ore concentrate which had been Ag distilled for 4 hours at 700°F in a N_2 atmosphere was leached with 1500 c.c. of NH_4OH for 5-3/4 hours at 20°C . - No cobalt was recovered.
3. 30 gms of a calcine containing 8.04% Co, 4.0% As, 0.55% S was leached with 1500 c.c. of NH_4OH for 4 1/2 hours. No cobalt was recovered.
4. 30 gms. of a finely ground calcine containing 7.7% Co, 6.2% As, and 1.26% S was leached with 1500 c.c. of NH_4OH for 5 hours at room temperature. - No cobalt was recovered.
5. 30 gms of finely ground ore concentrate was leached with 1500 c.c. of NH_4OH + 100 gms of $(\text{NH}_4)_2\text{CO}_3$ for 2 1/2 hours. The maximum temperature reached during this leach was 139°F . No cobalt was recovered.
6. 30 gms of ore concentrate which had been finely ground in a pebble mill for 4 hours was leached for 4 1/2 hours with 1500 c.c. of NH_4OH + 50 gms of $(\text{NH}_4)_2\text{CO}_3$ - Recovered 7.0% cobalt.
7. 30 gms of finely ground calcine speiss containing 4.3% As, 28.1% Co, and 40.2% Fe was leached as above for approximately 8 hours at a maximum temperature of 105°F . Recovered 2.2% cobalt.
8. 30 gms of finely ground calcine speiss was leached with 1500 c.c. of NH_4OH + 100 gms of $(\text{NH}_4)_2\text{CO}_3$ under O_2 pressure with agitation for 5 1/2 hours. It was allowed to stand in the solution for 39 hours. The maximum temperature reached during agitation was 113°F . - There was 4.68% recovery of cobalt achieved.
9. 30 gms of finely ground calcine speiss was leached with NH_4Cl in

- 1500 c.c. of NH_4OH for 3 hrs. It was allowed to stand for 15 hrs. - Slight trace of cobalt in the solution.
10. 30 gms of calcine speiss + 100 gms of NH_4Cl was leached for 5 hours at room temperature in 1500 c.c. NH_4OH - Trace of cobalt in the solution.
11. 30 gms of calcine speiss + 100 gms of NH_4Ac was leached for 5 hours in 1500 c.c. NH_4OH . Trace of cobalt found in the solution.
12. 65 gms of residue from an ore concentrate which had been subjected to 18 N. NaOH at 200 p.s.i. in O_2 at 300°F for $1\frac{1}{2}$ hours. The leach was carried out for 6 hours in 2000 c.c. of NH_4OH with a maximum temperature of 53°C . No cobalt was found in the solution.
13. 30 gms of residue which had been subjected to same conditions as #12 above for $5\frac{1}{2}$ hours was leached in 1500 c.c. of NH_4OH for 5 hours. - No cobalt was found in the solution.

Conclusions

1. It was thought that the iron may be tying up the cobalt in such form that the NH_4OH leach had no effect on the ore. To overcome this, NH_4Ac was tried in the hope that it would tie up the iron as an insoluble basic acetate and thereby free the cobalt. No success was achieved at room temperature but since the basic acetate forms much better at higher temperatures better results may be achieved at higher temperatures.
2. From the results obtained it seems that the presence of $(\text{NH}_4)_2\text{CO}_3$ in the NH_4OH will produce the best results. Also, it should be noted, the best results are obtained on the raw ore.
3. For the present, it has been decided to discontinue all low pressure, low temperature leaches until we obtain better recovery at point where we obtain our maximum recovery of cobalt. This is confirmed by higher pressures and temperatures. The high pressures unit has now been adapted for the use of ammonia.

High Pressure NH₄OH Leach

1. 100 gms of finely ground ore concentrate + 500 c.c. of NH₄OH was leached for 3 hrs. at 200 p.s.i. and 221°F. There was a 15% cobalt recovery.
2. 50 gms of ore concentrate in 500 c.c. of NH₄OH was leached for 8 hours at 200 p.s.i. and 210°F. There was a 4.38% recovery of cobalt.
3. 50 gms of finely ground ore concentrate + 50 gms of (NH₄)₂CO₃ in 500 c.c. of NH₄OH for 3 hours at 200 p.s.i. and 210°F. there was a 21.9% recovery of cobalt.
4. 50 gms of composite ore with 50 gms of CaO was leached in 500 c.c. of NH₄OH with a pressure of 475 p.s.i. at temperatures of 250°F and 350°F. There was no trace of oxidation of the residues and no trace of cobalt in either solution.
5. 103 gms of finely ground concentrate which had undergone a pressure of 200 p.s.i. at 221°F in 500 c.c. of NH₄OH. This residue was leached for 2½ hours in 500 c.c. of NH₄OH at 200 p.s.i. at 210°F. There was no trace of cobalt in the solution.
6. 50 gms of calcine containing 7.70% Co, 6.2% As, 1.26% S and 1.90% CaO was leached in 500 c.c. of NH₄OH under a pressure of 200 p.s.i. and a temperature of 210°F for 2 hours. No cobalt was found in the solution.

Conclusions

1. As in the low pressure leach best results were obtained on the raw ore. The effect of (NH₄)₂CO₃ on the leaching at these temperatures and pressures is still undetermined because of the insufficient number of runs.
2. It is possible, as in the caustic leach, that there is an optimum point where we obtain our maximum recovery of cobalt. This is confirmed by a repeat leaching on the residue, which appeared completely oxidized, with no recovery of cobalt.
3. It was thought that the cobalt may be precipitated out of

the ammoniacal solution as a cobalt arsenate. Lime was therefore added in the hope that the arsenates would precipitate preferentially as a calcium arsenate. From the results above it is hard to determine what did happen.

4. In order to try and establish to some extent the form that the cobalt assumes in the residue of these leaches a number of acid leaches will be carried out. This is based on the information that the cobalt arsenates are soluble only in concentrated acids.

High Pressure NaOH Leach

1. 100 gms of ore concentrate + 500 c.c. of 18 N. NaOH for 1 hour at 60°F with a pressure of approximately 300 p.s.i. - there was 23.6% cobalt recovered in the blue solution.

2. 150 gms of ore concentrate in 750 c.c. of 18 N NaOH was subjected to a pressure of 200 p.s.i. at a temperature of 300°F for 2 hours - 34% cobalt was recovered. The solution was deep blue.

3. 100 gms of ore concentrate leached in 500 c.c. of 18 N. NaOH at 300°F under 200 p.s.i. for 5½ hours. No cobalt was found in the solution. Colorless solution.

4. 100 gms of ore concentrate + 500 c.c. of 18 N NaOH at 200 p.s.i. and 300°F for 3 hours. No cobalt was recovered in the solution. Colorless solution.

Conclusions

1. The blue solution formed is probably the cobaltite ion. Dilution results in the separation of a brown oxide of cobalt leaving a colorless solution.

2. This blue solution is evidently an intermediate product in the oxidation of the ore. It is possible that two or more simultaneous reactions are occurring which will set an unknown limitation on this method of recovering the cobalt. A low pressure caustic leach will be carried

out using 30 p.s.i. O_2 pressure in an attempt to find out if there is a definite optimum point.

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Brian Carrigan

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Robert McIntosh.

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PROGRESS REPORT No. 2.

LITTLE GEM

Analysis of ores: (%)	Co	Fe	As	S	Au (oz./Ton)
Ore Concentrate	2.41	23.5	32.5	12.1	1.8
GM-200	6.55	22.0	44.7	8.5	0.96

CONCLUSIONS
 NaOH Leach under Oxygen pressure

The GM-200 used in the latest sample from the Little Gem area. This ore which is higher in cobalt and arsenic, is more difficult to oxidize. It is interesting to note that since the blue solution has been obtained with No Co in the solution.

1. 50 gms of ore concentrate + 750 ml. of 18N NaOH for 2½ hrs. at a pressure of 480 p.s.i. and 450°F.

2. 100 gms of ore concentrate + 500 ml of 12 N. NaOH at 200 p.s.i. and 270°F for 5 hrs.

Residue appeared completely oxidized. No Co in the solution.

3. 100 gms of ore concentrate + 500 ml of 12 N. NaOH for 5 hrs. at 200 p.s.i. and 220°F.

Residue appeared completely oxidized.

4. 100 gms of GM-200 + 500 ml of 18 N. NaOH for 3 hrs. at 200 p.s.i. and 220°F.

Very little oxidation with no Co in the solution.

5. 100 gms of GM-200 + 500 ml of 18 N. NaOH for 3 hrs. at 200 p.s.i. and 300°F.

Slightly oxidized residue - 14% Co recovered.

6. 100 gms of GM-200 + 500 ml of 18 N. NaOH for 3 hrs. at 200 p.s.i. and 320°F. Partial oxidation of residue with 53% of Co recovered in the solution.

7. 100 gms of GM-200 + 500 ml of 18 N. NaOH for 4 hrs. at 200 p.s.i. and 280°F. The solution was blue. The residue was then leached

twice more for 1 hr. at 200 p.s.i. and 280°F. Both solutions were blue.

54% Co was recovered.

8. 100 gms of GM-200 + 500 ml of 18 N. NaOH for 3 hrs. at 200 p.s.i. and 350°F. The solution was dark blue. The residue was leached three more times with 18 N. NaOH for 1½ hrs. at 200 p.s.i. and 350°F. Each solution was a dark blue.
- 54% Co was recovered.

CONCLUSIONS

The GM-200 head is the latest sample from the Little Gem Mine. This ore which is higher in Cobalt and Arsenic is much harder to oxidize. It is interesting to note that once the blue solution has been achieved it is easier to obtain a blue solution from the residue. This is probably due to the elimination of large amounts of sulphur and Arsenic in the preliminary leach.

From the results obtained from the caustic leaches on both the GM-200 Head and the old ore concentrate a great deal of difficulty could be expected when leaching on a commercial scale for the following reasons:

First, the optimum point which seems to be a fairly critical point varies with the ore concentrate.

Secondly, after the first leach there is a heavy white crystalline solid formed in the solution if the solution is allowed to cool. This of course would be detrimental in a counter flow multiple stage process which would seem to be necessary to obtain maximum recovery.

From the results obtained by a portable Gieger counter on the blue solution and residue the uranium seems to remain in the residue.

The Gold remains in the oxidized residue and is almost completely removed with moderate consumption of cyanide.

H₂SO₄ Leach:

1. 17 gms of oxidized ore concentrate + 1000 ml of 10% (by volume)

H₂SO₄ at room temperature under 30 p.s.i. for 4 hrs.

Trace of Co in the solution.

- 16.5 gms of oxidized ore concentrate + 1000 ml of 20% (by volume) H_2SO_4 for $5\frac{1}{2}$ hrs. at room temperature under 30 p.s.i.

This ore had originally been treated with an NH_4OH leach. The total Co recovered by the combined leaches was 60%.

- 50 gms of ore concentrate + 1000 ml of 10% (by volume) H_2SO_4 for 24 hrs. at room temperature under 30 p.s.i. The results are indefinite due to poor agitation and exhaustion of the oxygen sometime during the run.

8.1% Co recovered.

- 50 gms of ore concentrate + 1000 ml of 15% H_2SO_4 agitated for 20 hrs. at room temperature and pressure.

Trace of Co in the solution.

- 50 gms of ore concentrate + 1000 ml of 10% H_2SO_4 for 20 hrs. at room temperature and pressure.

Trace of Co in the solution.

CONCLUSIONS

Results on weak acid leaching are at this time very indefinite.

Better results are expected when leaching is carried on at higher temperatures and pressures.

Ferric Sulphate:

- 20 gms of No. 13 Calcine + 20 gms of $Fe_2(SO_4)_3$ + 250 ml of H_2O was bottle leached for 48 hrs.
No Co recovered.

NH_4OH Leach:

- 20 gms of well oxidized ore + 20 gms $Fe_2(SO_4)_3$ + 250 ml of H_2O was bottle leached for 48 hrs.
Trace of Co in the solution.
- 20 gms of No. 1 Calcine + 20 gms of $Fe_2(SO_4)_3$ + 250 ml of H_2O was bottle leached for 48 hrs.
No Co recovered.

4. 35 gms of well oxidized ore + 50 gms of $Fe_2(SO_4)_3$ + 500 ml of H_2O was leached for two hrs. at $260^\circ F$ without oxygen.

Trace of Co in the solution.

Ferrous Sulphate:

1. 100 gms of oxidized ore + 50 gms of $FeSO_4$ + 500 ml of H_2O for 2 hrs. at $400^\circ F$ and 450 p.s.i.

68% Co recovered.

2. 50 gms of ore concentrate + 500 ml of H_2O + 50 gms of $FeSO_4$ for 2 hrs. at $260^\circ F$ without oxygen.

CONCLUSIONS Trace of Co recovered.

3. 50 gms of GM-200 + 750 ML of H_2O + 75 gms of $FeSO_4$ at $410^\circ F$ and 450 p.s.i. for 3 hrs.

56% Co recovered.

H_2O Leach:

1. 100 gms of GM-200 + 750 ml of H_2O for $1\frac{1}{2}$ hrs. at $300^\circ F$ and 300 p.s.i.

68% Co recovered.

CONCLUSIONS

While no definite conclusions can be drawn at this time the results of both the $FeSO_4$ and H_2O leaches certainly warrant further investigation. there is also no reason why these cannot be carried out at a lower temperature and pressure. These conditions of high temperature and pressure were picked to see what would happen.

NH_4OH Leach:

1. 150 gms of ore concentrate + 750 ml of concentrated NH_4OH ground in pebble mill. The residue was leached in NH_4OH at

This is confirmed by the fact that the ore was well oxidized $250^\circ F$ and 250 p.s.i. for 1-3/4 hrs.

64% Co recovered.

2. 50 gms of an oxidized ore concentrate + 500 ml of concentrated

NH₄OH was leached for 2½ hrs. at 200°F under 260 p.s.i. of Hydrogen.

Trace of Co in solution.

3. 100 gms of GM-200 + 500 ml of NH₄OH (1 part NH₄OH to three parts H₂O) for 2½ hrs. at 250°F. and 300 p.s.i. of oxygen.
46% Co recovered.

4. 100 gms of GM-200 + 500 ml of concentrated NH₄OH for 2 hrs. at 200 p.s.i. and 200°F.
10% Co recovered.

CONCLUSIONS

As suggested, an ammonia leach under hydrogen pressure was carried out on an oxidized ore with poor results. Since this is not conclusive more runs will be attempted and the results should be available by the next report. More runs would have been attempted by this time but we have had mechanical difficulty which has since been remedied.

It is possible that we have been conducting these ammonia leaches at too high a concentration. From the results obtained in No. 3 it would seem that we will get better results at lower concentrations.

We believe that in the caustic leaches the oxidation when once started takes place very rapidly and is completed in a very short time. This will explain why there is a critical optimum point where we obtain the cobalt hydroxide before it is converted to the oxide. Since we have also had indications of an optimum point in our ammonia leaches the cobalt is probably first converted to the soluble ammoniacal complex and from there to the oxide. From the results so far it seems that there is a relation between the case of oxidation and the hydroxide ion concentration. This is confirmed by the fact that the ore was more readily oxidized in the water solution than in the ammoniacal solution which in turn was more readily oxidized than in the caustic solution. This does not mean we want the whole of the ore oxidized, but since the cobalt probably

is oxidized into the solution at the same time that the ore is oxidized, we should get better results by obtaining a slower oxidation and stopping it before the cobalt is oxidized to an insoluble oxide.

Sodium Sulphate Leach:

1. 100 gms. of GM-200 + 75 gms of Na_2SO_4 + 500 ml 18 N. NaOH.

Leached for 4 hrs. at 200 p.s.i. and 370°F.

34% Co recovered.

CONCLUSIONS

The Na_2SO_4 does not seem to have any effect on this reaction.