CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 66-29

INVESTIGATION OF A COPPER-NICKEL ORE FROM AXIS LAKE AREA OF NORTHERN SASKATCHEWAN FOR PLACID OIL COMPANY, CALGARY, ALBERTA

by

G.I. MATHIEU

MINERAL PROCESSING DIVISION

NOTE: THIS REPORT RELATES ESSENTIALLY TO THE SAMPLES AS RECEIVED. THE REPORT AND ANY CORRESPONDENCE CONNECTED THEREWITH SHALL NOT BE USED IN FULL OR IN PART AS PUBLICITY OR ADVERTISING MATTER.

COPY NO. 6

MARCH 31, 1966

INDUSTRIAL CONFIDENTIAL

Mines Branch Investigation Report IR 66-29

INVESTIGATION OF A COPPER - NICKEL ORE FROM AXIS LAKE AREA OF NORTHERN SASKATCHEWAN FOR PLACID OIL COMPANY, CALGARY, ALBERTA

by

G. I. Mathieu

- - -

SUMMARY OF RESULTS

Head analysis of the ore samples submitted:

Element	Sample No. 1	Sample No. 2	Sample No. 3
Cu, per cent	0.28	0.14	0.07
Ni, per cent	0.50	0.28	0.16
Ag, oz/ton	0.11	0.05	0.01

None of the samples submitted contained gold, platinum, cobalt, molybdenum or vanadium in quantities of economic interest. Only Sample No. 1 was of high enough grade to warrant investigation.

Chalcopyrite and pentlandite presented a rather fine structure and were intimately associated with pyrrhotite. High intensity magnetic separation showed that about 35% of the copper and 70% of the nickel were still attached to this mineral at minus 200 mesh. Selective flotation tests made it evident that the chalcopyrite and pentlandite unassociated with pyrrhotite could not be recovered in separate concentrates of acceptable grade.

A test procedure consisting of bulk flotation of a relatively rich copper - nickel concentrate followed by scavenger flotation of the residual copper and nickel values associated with pyrrhotite produced a copper - nickel bulk concentrate assaying 7.7% Cu and 5.9% Ni with recoveries of 60% and 32% respectively. Extraction of the values in the scavenger concentrate by sulphating roast and leaching increased the overall recovery to 79% of the copper and to 70% of the nickel.

* Scientific Officer, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada

CONTENTS

LIST OF TABLES

INTRODUCTION

Property

Placid Oil Company carried out a drilling program on a property, near Axis Lake, 15 miles northwest of Stoney Rapids, northern Saskatchewan. Twelve holes drilled on a base metal occurence indicated a zone containing copper, nickel, cobalt, vanadium, gold and silver. To aid in assessing the value of this deposit, the company requested a mineral processing investigation.

Shipments

Three cartons containing 200 lb of drill core were received on August 9, 1965. A second shipment consisting of assay pulps and weighing 37 lb was received on August 7. Each shipment contained different samples designated as follows:

TABLE 1

Weight and Designation of Ore Samples

Designation	Weight lb	
	Drill Core	Assay Pulp
Sample No. 1	78	nil
Sample No. 2	40	24
Sample No. 3	84	13

Purpose of Investigation

The investigation was aimed at concentrating the copper and nickel minerals, with possible recovery of other metals such as cobalt, molybdenum, vanadium, gold, silver and platinum, if present in quantities of economic interest. The company requested that the results of the investigation be sent to Mr. C.A. Luverne Hogg, Consultant Geologist, 2501 Wascana Street, Regina, Saskatchewan.

Sampling and Analysis

A few selected pieces were cut from each split drill core for mineralogical examination. The remainder of each sample was then crushed to minus 10 mesh from which head samples were riffled out for chemical and semi-quantitive spectrographic analysis.

TABLE 2

Chemical Analysis* of Head Samples

Element	Sample No. 1	Sample No. 2	Sample No. 3
Copper (Cu)	0.28%	0.14%	0.07%
Nickel (Ni)	0.50%	0.28%	0.16%
Iron (Fe)	16.96%	8.46%	5.04%
Cobalt (Co)	0.04%	0.01%	0.01%
Insoluble	53.9 %	65.1 %	67.9 %
Gold (Au)	0.003 oz/ton	0.002 oz/ton	0.001 oz/ton
Silver (Ag)	0.11 oz/ton	0.05 oz/ton	0.04 oz/ton
Platinum (Pt)	ND	ND	ND

*From Internal Report MS-AC 65-1178.
ND: None detected

Spectrographic analysis on portions of the head samples indicated presence of the following elements in approximate order of abundance.

TABLE 3

Spectrographic Analysis* of Head Samples

I	- Si, Fe, Al, Mg, Ca	(> 1.0%)
II	- Ti, Mn, Na, Ni, Cu	(1.0 - 0.1%)
III	- Cr, Co, V, Mo	(0.1 - 0.01%)
IV	- Sn, Mo, Au, Ag	(< 0.01%)

*From Internal Report MS-AC 65-1102

Mineralogical Examination*

A few hand specimens from each split drill core and representative portions of head samples (-10 mesh) were sent to the Mineralogy Section of the Mineral Sciences Division for examination.

The crushed head samples were screened and the -65 +150 mesh portion was separated into two density fractions by means of a heavy liquid with a specific gravity of 3.33. Nine polished sections and one thin section were prepared from the drill cores, and two polished sections were prepared from each -65 +150 mesh fraction of the head samples. The minerals were identified by microscopic and X-ray diffraction studies, and the textural relationships of the minerals were determined microscopically.

* From Mines Branch Investigation Report IR-65-109

Pyrrhotite is the most abundant ore mineral. It occurs as rounded or irregular grains and as disseminations in the gangue. (Figure 1). The grains vary in size from about 5 to over 3000 microns and contain inclusions of chalcopyrite and pentlandite. It is possible that the pyrrhotite may contain some nickel in solid solution as the small laths of pentlandite may have exsolved from a nickel-rich pyrrhotite. Most of the pyrrhotite appears to be of the monoclinic variety, but there may be some of the hexagonal variety present.

Chalcopyrite occurs as irregular masses adjacent to pyrrhotite grains or as disseminations in gangue (Figure 1), but also sometimes forms intergrowths with pentlandite. The chalcopyrite varies from about 5 to 1000 microns in size. Pyrite is often bordered by chalcopyrite. (Figure 2)

The pentlandite varies in size from about 5 to 300 microns, and generally occurs as inclusions within larger pyrrhotite masses. These inclusions frequently occur at grain boundaries or along fractures, some of which are filled with gangue minerals (Figure 3). Also occurring are smaller grains of pentlandite (about 10 x 2 microns) which appear as elongated flame-like inclusions in the pyrrhotite (Figure 4).

Marcasite, when present, generally replaces pyrrhotite and pyrite. It is most frequently seen in the sections from Sample No. 2.

Safflorite was the only cobalt-bearing mineral identified. Only one grain, closely associated with niccolite, was observed.

Estimates of the proportions of ore and gangue minerals, and of the liberation characteristics of the ore minerals in the -65 +150 mesh fractions were made by means of microscopic examination of polished sections prepared from the products of heavy-liquid separations. The liberation of ore minerals from gangue varies between 40 and 70%. However, less than half of the chalcopyrite is liberated at that size and the proportion of pentlandite liberated was negligible.

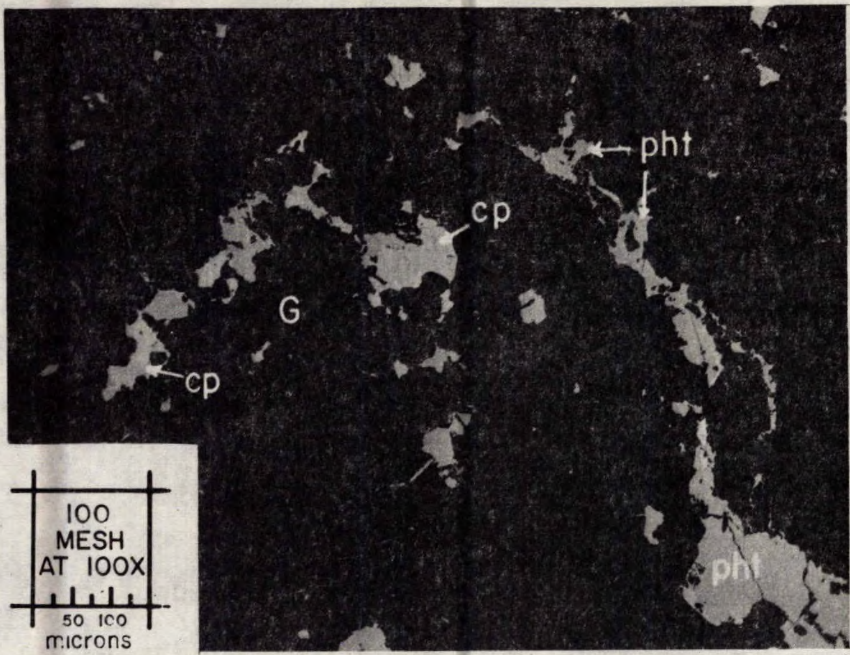

Figure 1 - Photomicrograph of polished section showing disseminations and veins of pyrrhotite (pht) as well as disseminations of chalcopyrite (cp) in gangue (G).

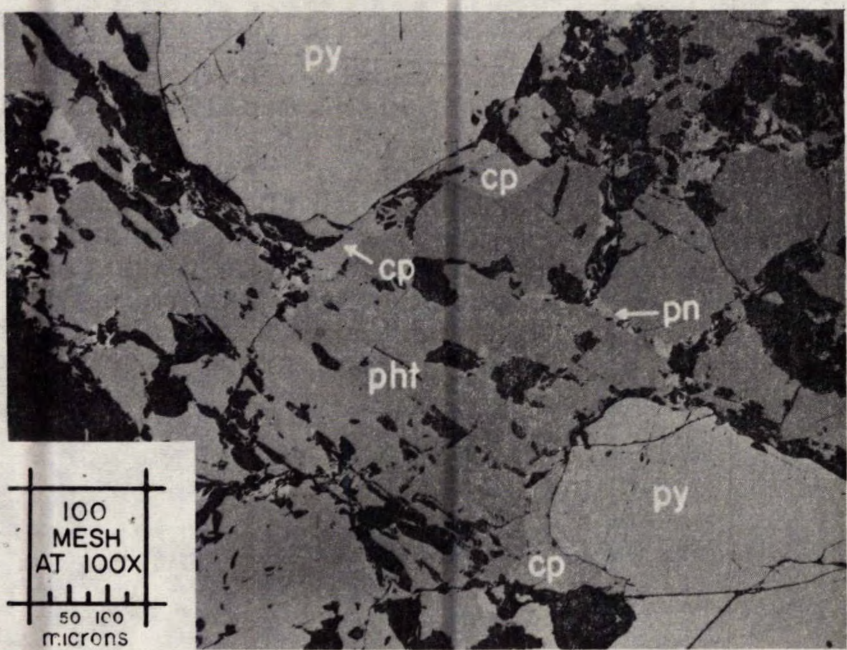

Figure 2 - Photomicrograph of polished section showing chalcopyrite (cp) rimming pyrite (py) crystals. Pentlandite (pn) is shown at grain boundaries of pyrrhotite (pht) grains. Black areas are pits and gangue.

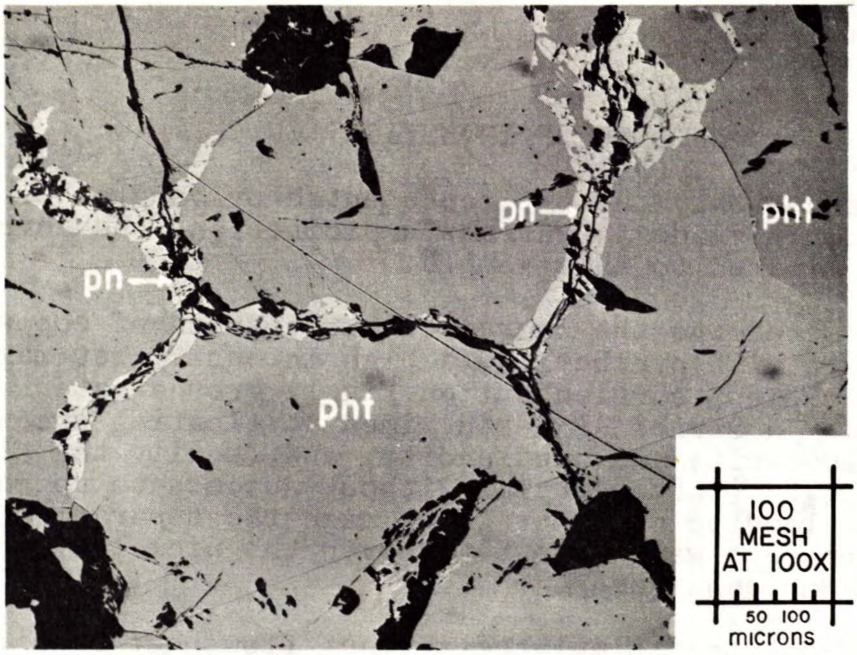

Figure 3 - Photomicrograph of polished section showing pentlandite (pn) at grain boundaries of pyrrhotite (pht). Black areas are pits and gangue.

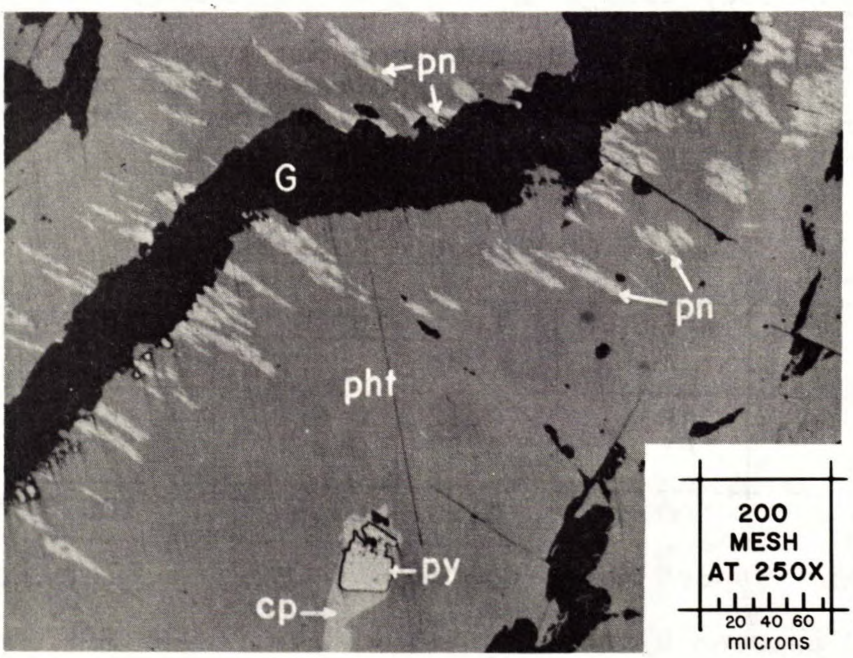

Figure 4 - Photomicrograph of polished section showing "flames" of pentlandite (pn) in pyrrhotite (pht). Also shown is a corroded pyrite (py) crystal surrounded by chalcopyrite (cp) and vein of gangue (G) minerals. Other black areas are pits.

OUTLINE OF INVESTIGATION

Because only the ore represented by Sample No. 1 may be of high enough grade to warrant exploitation, the investigative testwork was limited to this sample.

To determine the degree of association of copper and nickel minerals with pyrrhotite, a high intensity wet magnetic separation was first carried out on finely ground material. This was followed by a series of tests aimed at floating successively the free chalcopyrite and pentlandite, and finally the nickeliferous pyrrhotite. After trying without success to upgrade the so-called pentlandite concentrate by magnetic separation and cleaner flotation, attempts were made to recover the nickel by a sulphating roast and leach technique.

A final test simulated a plant flowsheet consisting of bulk flotation of a copper-nickel final concentrate and scavenger flotation of the residual values with their extraction by roasting and leaching.

DETAILS OF INVESTIGATION

Magnetic Separation, Test 1

A 1000 g sample was cut from the crushed drill core, Sample No. 1, and ground to minus 200 mesh. This product was fed to a Jones high intensity magnetic separator set at 5 amperes.

TABLE 4

Results of Magnetic Separation

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
Concentrate	38.6	0.22	0.82	33.9	69.2
Tailing	61.4	0.27	0.23	66.1	30.8
Feed (calcd)	100.0	0.26	0.46	100.0	100.0

* From Internal Report MS-AC 65-1189.

It is seen by this test that 34% of the copper and 69% of the nickel were still associated with pyrrhotite at minus 200 mesh.

Selective Flotation

This series of tests was done to determine if:

(i) the chalcopyrite and the pentlandite not associated with pyrrhotite could be recovered successively by selective flotation and;

(ii) most of the remaining copper and nickel values could be floated subsequently along with pyrrhotite.

Tests 2, 3 and 4 - Effect of Alkalis

Lots of 2000 g of ore were ground to 70% minus 200 mesh and floated using the procedure shown in Table 5.

TABLE 5

Reagents and Conditions of Selective Flotation

Operation	Time min	Reagents	lb/ton	pH
Conditioning	5	Xanthate Z - 6 Test 2: no alkali Test 3: lime Test 4: soda ash	0.04 - 1.00 3.00	 8.1 9.9 9.7
Copper flotation	4	Dowfroth 250	0.02	8.0 - 9.5
Nickel flotation	5	Xanthate Z - 6 Dowfroth 250	0.04 0.02	7.9 - 9.1
Pyrrhotite flotation	16	At start: Copper sulphate Xanthate Z - 6 Dowfroth 250 After 8 min: Copper sulphate Xanthate Z - 6 Dowfroth 250	 1.00 0.10 0.02 0.50 0.05 0.02	 7.6 - 9.0 7.6 - 8.8

Note: The higher pH readings correspond to the tests with lime and soda ash.

TABLE 6

Results of Selective Flotation with Alkalis

Test	Product	Weight %	Analysis* %		Distribution %	
			Cu	Ni	Cu	Ni
2 no alkali	Copper conc	3.0	5.63	5.24	59.5	32.5
	Nickel conc	8.0	0.60	1.80	16.9	29.8
	Pyrrhotite conc	16.1	0.19	0.77	10.9	25.7
	Flotation tailing	72.9	0.05	0.08	12.7	12.0
	Feed (calcd)	100.0	0.28	0.48	100.0	100.0
3 lime	Copper conc	2.2	6.42	5.12	50.3	21.0
	Nickel conc	7.7	0.65	2.15	17.9	31.0
	Pyrrhotite conc	11.5	0.23	1.14	9.4	24.5
	Flotation tailing	78.6	0.08	0.16	22.4	23.5
	Feed (calcd)	100.0	0.28	0.54	100.0	100.0
4 soda ash	Copper conc	3.3	5.69	4.67	66.8	31.3
	Nickel conc	5.0	0.49	2.29	8.7	23.9
	Pyrrhotite conc	8.6	0.22	1.11	6.7	19.4
	Flotation tailing	83.1	0.06	0.15	17.8	25.4
	Feed (calcd)	100.0	0.28	0.49	100.0	100.0

* From Internal Reports MS-AC 65-1166 and 1189.

Tests 5, 6 and 7 - Effect of Aeration

These tests were similar to Tests 2, 3 and 4, respectively, except that the conditioning stage was replaced by a 30 minutes aeration period.

TABLE 7

Results of Selective Flotation after Aeration

Test	Product	Weight %	Analysis* %		Distribution %	
			Cu	Ni	Cu	Ni
5 no alkali	Copper conc	2.9	6.98	4.43	63.9	24.3
	Nickel conc	5.8	0.52	2.49	9.5	27.4
	Pyrrhotite conc	7.3	0.35	1.31	8.0	18.1
	Flotation tailing	84.0	0.07	0.19	18.6	30.2
	Feed (calcd)	100.0	0.32	0.53	100.0	100.0
6 lime	Copper conc	2.3	8.55	5.05	61.8	23.3
	Nickel conc	5.9	0.62	2.19	11.5	26.0
	Pyrrhotite conc	10.2	0.27	1.03	8.7	21.1
	Flotation tailing	81.6	0.07	0.18	18.0	29.6
	Feed (calcd)	100.0	0.32	0.50	100.0	100.0
7 soda ash	Copper conc	3.6	5.97	4.38	71.4	31.0
	Nickel conc	5.1	0.34	2.39	5.7	24.0
	Pyrrhotite conc	7.8	0.24	1.11	6.2	17.0
	Flotation tailing	83.5	0.06	0.17	16.7	28.0
	Feed (calcd)	100.0	0.30	0.51	100.0	100.0

* From Internal Report MS-AC 65-1208.

Test 8 and 9 - Effect of Reagent Z - 200

The flotation procedure described in Table 5 was followed, except that the conditioning stage was done with 0.02 lb Z - 6/ton and 0.03 lb Z - 200/ton in Test 8, and with 0.06 lb Z - 200/ton in Test 9. Both tests were carried out at a natural pH of 8.0.

TABLE 8

Results of Selective Flotation with Z - 200

Test	Product	Weight %	Analysis* %		Distribution %	
			Cu	Ni	Cu	Ni
8 Z - 6 and Z - 200	Copper conc	2.5	7.08	5.06	60.0	24.1
	Nickel conc	10.3	0.44	2.01	15.4	39.4
	Pyrrhotite conc	12.7	0.22	1.04	9.5	25.2
	Flotation tailing	74.5	0.06	0.08	15.1	11.3
	Feed (calcd)	100.0	0.29	0.53	100.0	100.0
9 Z - 200	Copper conc	2.0	9.23	5.25	60.1	21.5
	Nickel conc	11.5	0.50	1.84	18.7	43.5
	Pyrrhotite conc	11.8	0.17	0.88	6.5	21.3
	Flotation tailing	74.7	0.06	0.09	14.7	13.7
	Feed (calcd)	100.0	0.31	0.49	100.0	100.0

* From Internal Reports 65-1207 and 66-38, 49 and 73.

Selective Flotation and Magnetic Separation (Test 10)

In an attempt to upgrade both the copper and the nickel concentrates, selective flotation was supplemented by cleaning stages and by magnetic separation. The latter technique was to remove pyrrhotite reporting in the nickel concentrate. Details of procedure are shown in Table 5.

TABLE 9

Conditions of Grinding, Flotation and Magnetic Separation

Operation	Time min	Reagents	lb/ton	pH
Grinding (70% - 200 m)	30			
Conditioning	10	Reagent Z - 200	0.06	8.2
Cu rougher flotation	4	Dowfroth 250	0.01	8.1
Cu cleaner flotation	2	Reagent Z - 200	0.01	8.1
Cu recleaner flotation	2	Reagent Z - 200	0.005	7.9
Ni rougher flotation	5	Xanthate Z - 6 Dowfroth 250	0.04 0.02	7.8
Regrinding Ni conc (86% - 325 m) Magnetic separation*	10			
Ni cleaner flotation	3	Xanthate Z - 6	0.01	8.0
Ni recleaner flotation	2	Xanthate Z - 6	0.005	8.1
Pyrrhotite flotation	16	At start: Copper sulphate Xanthate Z - 6 Dowfroth 250 After 8 min: Copper sulphate Xanthate Z - 6 Dowfroth 250	 1.00 0.10 0.02 0.50 0.05 0.02	 7.7 7.8

* On Jones high intensity wet magnetic separator set at 4 amperes.

TABLE 10

Results of Selective Flotation and Magnetic Separation

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
Cu recl conc	1.4	12.59	6.85	56.6	18.3
Cu recl + cl tailing	0.5	0.84	3.31	1.4	3.2
Ni recl conc	3.7	0.88	2.58	10.5	18.3
Ni recl + cl tailing	2.2	0.38	1.33	2.7	5.6
Pyrrhotite conc (mag sep)	3.9	0.41	2.08	5.1	15.5
Pyrrhotite conc (flotation)	14.0	0.21	0.98	9.4	26.3
Flotation tailing	74.3	0.06	0.09	14.3	12.8
Feed (calcd)	100.0	0.31	0.52	100.0	100.0

* From Internal Reports MS-AC 66-49 and 73.

Despite the use of a more selective promoter in the copper flotation, appreciable nickel was carried into the concentrate. This was due partly to the intimate association of pentlandite with chalcopyrite, and partly to their similar flotation characteristics.

Although considerable pyrrhotite was removed by magnetic separation, and further pyrrhotite and gangue eliminated by cleaner flotation, the final nickel concentrate was still short of acceptable grade. Failure of this technique seems due to the fine inclusions of much of the pentlandite in pyrrhotite and, to a lesser extent, in gangue minerals.

Thus, the only possible method for processing this ore would be:

(a) Bulk flotation to produce a relatively rich copper-nickel concentrate;

(b) Scavenger flotation to recover residual copper and nickel values with pyrrhotite;

(c) Roasting and leaching of the scavenger concentrate.

Roasting and Leaching (Test 11)

This was a preliminary test aimed at recovering the copper and nickel values associated with pyrrhotite. The objective was to convert the metal sulphides to the form of water soluble sulphates by the technique described below. The test was made on a few products from Test 10.

Lots of 50 g were pulped with a saturated solution of sodium sulphate. Each pulp was filtered and the filter cake dried. This procedure insured an homogeneous distribution of about 2 g of salt through the sample. The sulphating roast was then carried out over a period of three hours at gradually increasing temperature from 200°C to 650°C. After allowing ten minutes to lower the temperature of the roasted products to about 200°C, the calcine was leached with 100 ml of water at approximately 60°C for one hour. The residue was filtered, washed and analysed for copper and nickel with the following results.

TABLE 11

Results of Roasting and Leaching

Product Used	Weight Lost %	Residue Analysis* %		Extraction** %	
		Cu	Ni	Cu	Ni
Ni recl conc	36.4	0.10	0.89	88.6	65.5
Ni recl + cl tailing	21.5	0.12	0.52	68.4	60.2
Pyrrhotite conc (mag sep)	34.6	0.03	0.53	92.7	74.5
Pyrrhotite conc (flotation)	27.0	0.04	0.22	80.1	77.6
Weighed average (calcd)	29.1	0.06	0.40	82.4	73.6

* From Internal Report MS-AC 66-100

** Calculated by difference

Note: The residue analyses are expressed in terms of original products.

Simulation of Plant Flowsheet (Test 12)

This test was an attempt to simulate a plant flowsheet for the treatment of the Axis Lake ore. As illustrated by Figure 5, the proposed procedure included flotation of a copper-nickel concentrate followed by scavenger flotation of the residual values and their extraction by sulphating roast and leach.

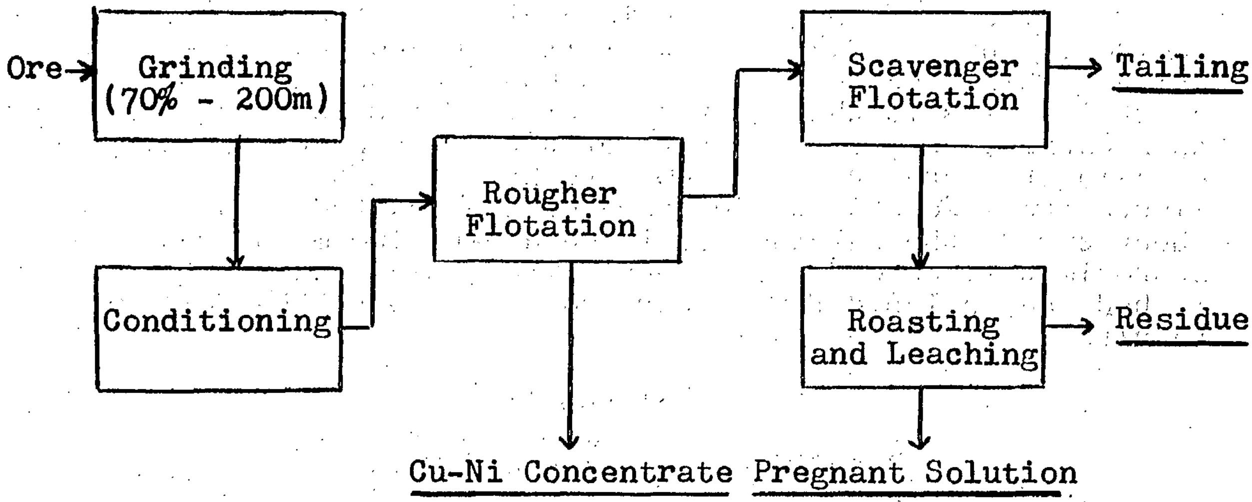

Figure 5 - Proposed Flowsheet

The details of the rougher and scavenger flotation are shown in Table 12. Roasting and leaching were carried out using the technique described in Test 11.

TABLE 12

Reagents and Conditions of Bulk Flotation

Operation	Time min	Reagents	lb/ton	pH
Conditioning	5	Xanthate Z - 6 Reagent Z - 200	0.03 0.02	8.2
Cu-Ni flotation	4	Dowfroth 250	0.02	8.1
Scavenger flotation	24	Copper sulphate* Xanthate Z - 6* Dowfroth 250*	1.00 0.20 0.04	7.8

* By staged additions.

TABLE 13

Results of Bulk Flotation, Roasting and Leaching

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
Cu-Ni conc	2.6	7.70	5.86	59.5	31.5
Pregnant solution	23.5	0.28	0.80	19.5	38.8
Leaching residue		0.08	0.33	5.6	16.0
Flotation tailing	73.9	0.07	0.09	15.4	13.7
Feed (calcd)	100.0	0.34	0.48	100.0	100.0

*From Internal Report MS-AC 66-170.

Note: The weight and the assays of the solution and the residue are expressed in terms of original scavenger concentrate.

As requested by Mr. Hogg, the following minor constituents were determined in the final copper - nickel concentrate.

TABLE 14

Analysis* of Minor Elements in Cu - Ni Concentrate

Oz/ton			%		
Au	Ag	Pt	Co	Mo	V
0.05	3.85	ND**	0.32	0.05	0.04

* From Internal Reports MS-AC 66-420

**ND: none detected

SUMMARY AND CONCLUSIONS

Of three lots of drill core submitted by Placid Oil Company, only Sample No. 1 represent an ore of high enough grade to warrant possible exploitation. The investigation was limited to this sample which contained 0.28% Cu, 0.50% Ni and 0.11 oz Ag/ton.

By mineralogical studies and high intensity magnetic separation, it was found that about 35% of the chalcopyrite and 70% of the pentlandite were finely intergrown with pyrrhotite.

A few selective flotation tests were made in attempt to float successively the chalcopyrite and the pentlandite unassociated with pyrrhotite, and finally the residual values intergrown with pyrrhotite. However, partly because of similar floatability of the copper and nickel minerals, and partly due to lack of liberation, it was not possible to produce separate concentrates of acceptable grade. Even when selective flotation was supplemented by several cleaning stages and by application of magnetic separation to remove pyrrhotite from the nickel concentrate, the results were still unsatisfactory as shown below:

Product	Grade %		Distribution %	
	Cu	Ni	Cu	Ni
Cu recl conc	12.6	6.8	56.6	18.3
Ni recl conc	0.9	2.6	10.5	18.3
Pyrrhotite conc	0.3	1.2	14.5	41.8

In the flotation tests using alkalis either with or without preaeration, no improvement of selectivity was achieved and the nickel overall recovery was decreased.

Although of doubtful economics because of the low metal content, the only process developped for the treatment of the Axis Lake ore consisted of recovering first a relatively rich copper - nickel concentrate, and subsequently the residual values for extraction by sulphating roast and leaching. A test carried out along these lines gave the following results:

Product	Grade		Distribution	
	Cu	Ni	Cu	Ni
Cu - Ni conc	7.7%	5.9%	59.5%	31.5%
Cu - Ni soln	0.2g/l	0.8g/l	19.5%	38.8%

No minor element of economic importance was found in the copper-nickel concentrate, with the exception of 3.8 oz Ag/ton.

The limited work done on roasting and leaching resulted in nickel extraction of about 72%. Further research on the process might improve this result, but is beyond the scope of the present investigation.

ACKNOWLEDGEMENTS

The writer wishes to acknowledge the contribution to this investigation by members of the Mineral Sciences Division, namely, R. McAdam, H. Bart, R. Buckmaster, L. McCorriston, L. Lutes, C. Derry and D. Cumming for the chemical analyses, P. Porteau for the spectrographic analysis and L. Cabri for the mineralogical examination.

GIM/lm