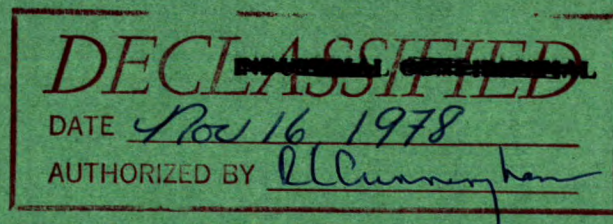


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CANADA



DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 63-102

**FLOTATION OF A COMPLEX COPPER-NICKEL  
ORE FROM RAGLAN NICKEL MINES  
LIMITED, UNGAVA AREA, QUEBEC**

by

**G. I. MATHIEU**

**MINERAL PROCESSING DIVISION**

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FLOTATION OF A COMPLEX COPPER-NICKEL ORE  
FROM RAGLAN NICKEL MINES LIMITED,  
UNGAVA AREA, QUEBEC

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SUMMARY OF RESULTS

The sample of copper-nickel ore submitted contained 0.62% Cu and 1.53% Ni. The chalcopyrite and the nickel minerals presented a rather fine structure and were intimately associated with pyrrhotite and gangue.

A test run on a Jones high intensity wet magnetic separator indicated that about 35% of the copper and 70% of the nickel minerals were attached to the pyrrhotite at minus 100 mesh.

Attempts were made to float successively the liberated chalcopyrite, the free pentlandite, and the pyrrhotite with associated copper and nickel minerals. This procedure resulted in the production of the following concentrates:

	<u>Grade</u>	<u>Recovery</u>
1) Copper concentrates:	13.5 - 19.9% Cu	30 - 42% Cu
2) Nickel concentrates:	8.7 - 8.9% Ni	35 - 42% Ni
3) Pyrrhotite concentrates:	0.34 - 0.35% Cu 1.30 - 1.35% Ni	17 - 21% Cu 29 - 30% Ni

About two-thirds of the copper and nickel contained in the pyrrhotite concentrates were extracted by sulphatizing roast followed by water leaching.

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## INTRODUCTION

Raglan Nickel Mines Limited holds several properties in the Ungava Area of the Province of Quebec known as New Quebec. The largest of these is a concession covering the Cross Lake tract, or Block M-89, and has an area of 76 square miles, located about 38 miles south of Deception Bay on Hudson Strait.

Exploration work on the properties by diamond drilling has indicated a tonnage potential of 10 million tons grading 1.5% nickel and 0.7% copper.

### Shipment

A 125 lb sample of ore crushed to  $-3/4$  in. and identified as 'Composite No 1 and No 3' was received at the Mines Branch on September 25, 1962. This had been shipped by Mr. R.J.C. Tait, Manager of Lakefield Research of Canada Limited, at the request of Dr. Griffis of Watts, Griffis and McOuat Limited, 159 Bay Street, Toronto, Ontario. The sample was said to have come originally from the company's Cross Lake property.

### Purpose of Investigation

Dr. A. T. Griffis requested an investigation with the aim of improving grades and recoveries of copper and nickel concentrates.

### Sampling and Analysis

All the material received was crushed from  $-3/4$  in. to  $-10$  mesh, except a few selected pieces which were picked out for mineralogical examination. A head sample was riffled out by conventional methods for chemical analysis and microscopic examination.

The chemical analysis gave the following results.

TABLE 1

#### Chemical Analysis\* of Head Sample

Copper (Cu)	0.62%
Nickel (Ni)	1.53%
Iron (total Fe)	28.7%
Iron (soluble Fe)	28.0%
Sulphur (S)	15.6%
Insoluble	29.9%

\*From Internal Report MS-AC-62-1174

A spectrographic analysis on a portion of the head sample indicated the elements present in the following approximate order of decreasing abundance.

TABLE 2

Spectrographic Analysis\* of Head Sample

I	-	Fe, Si, Mg
II	-	Al, Ni, Ca, Cu
III	-	Cr, Ti, Mn
IV	-	Co, V

\*From Internal Report MS-AC-62-1070

MINERALOGICAL EXAMINATION

A few hand specimens and a portion of the -10 m head sample were submitted to the Mineralogical Section of the Mineral Sciences Division for microscopic examination.

Method of Examination

Four polished sections were prepared from the hand specimens and the minerals identified by means of microscopic and X-ray diffraction study.

Results of Examination\*

"The sample consists of disseminated metallic minerals in a serpentine-chlorite rock. The metallic minerals are pyrrhotite (FeS), pentlandite ( (Fe, Ni) S), chalcopyrite (CuFeS<sub>2</sub>) and violarite ( (Ni, Fe)<sub>3</sub>S<sub>4</sub>). The non-metallic minerals are serpentine, chlorite, amphibole, feldspar and spinel.

Pyrrhotite is the principal metallic mineral. It occurs as anhedral grains that range from below 1 micron to about 500 microns in diameter, and contains inclusions of pentlandite and spinel. Some of the pyrrhotite grains occur as minute angular fragments that appear to be parts of larger grains that have been intensely sheared, fractured and recemented by gangue minerals (see Figure 1).

---

\*From Investigation Report IR-62-107 by W. Petruk, December 10, 1962.

The pentlandite occurs as (a) irregular grains intergrown with pyrrhotite and gangue (see Figure 2), and (b) elongated inclusions in pyrrhotite (see Figure 3). The inclusions range from below 1 micron to 20 microns in width and up to 50 microns in length. They were likely exsolved from pyrrhotite. The irregular grains of pentlandite range from about 1 to 250 microns in diameter, and the larger ones are partially altered to violarite (see Figure 4). The alteration proceeds into pentlandite from fractures and from pyrrhotite-pentlandite boundaries.

A small quantity of chalcopyrite occurs as irregular grains ranging from about 5 to 200 microns in diameter. It is commonly intergrown with pyrrhotite and gangue minerals.

A few grains of spinel are present in the ore. The variety could not be identified, but X-ray studies suggest that it is not magnetite but may be a magnesium-aluminum spinel."

#### Remarks

The minerals that may be of economic significance are pentlandite, violarite and chalcopyrite. These minerals are very finely intergrown with pyrrhotite and gangue, and complete liberation cannot be expected even after extremely fine grinding.

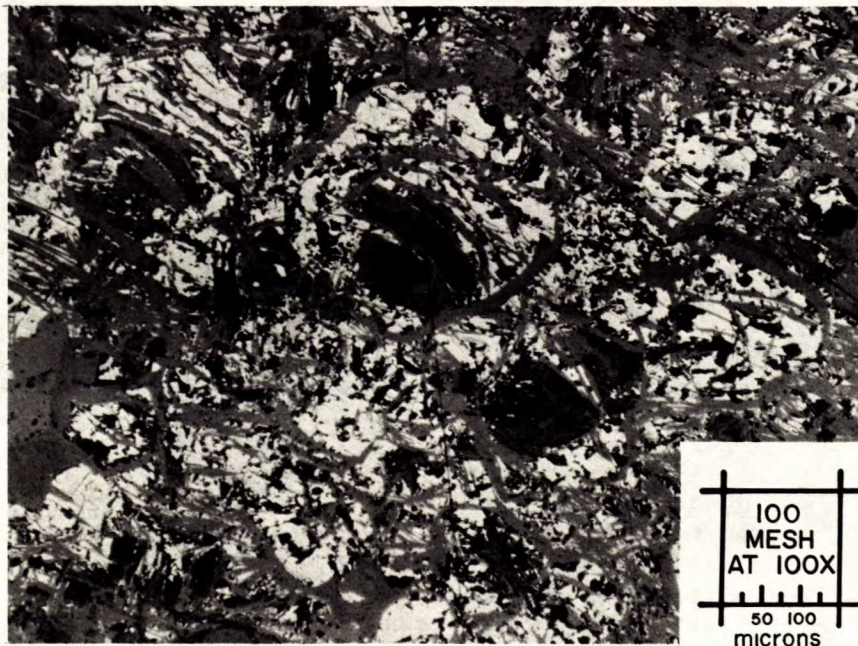


Figure 1 - Photomicrograph of a polished section showing pyrrhotite (white) that appears to have been fractured and recemented by gangue minerals (grey).

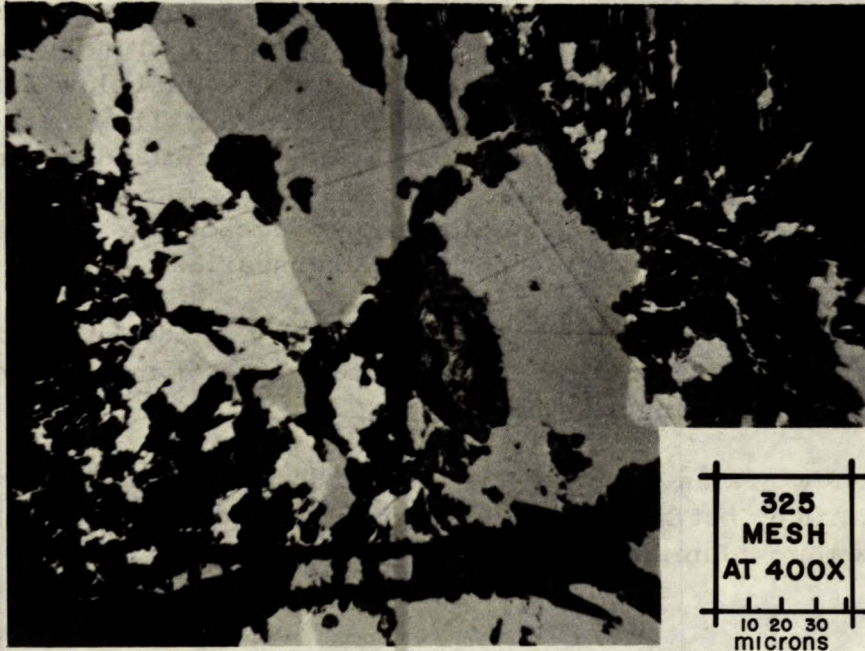


Figure 2 - Photomicrograph of a polished section showing pentlandite (white) intergrown with pyrrhotite (grey). The black areas represent gangue.



Figure 3 - Photomicrograph of a polished section showing minute elongated inclusions of pentlandite (white) in pyrrhotite (grey). The black areas are pits on the polished surface.



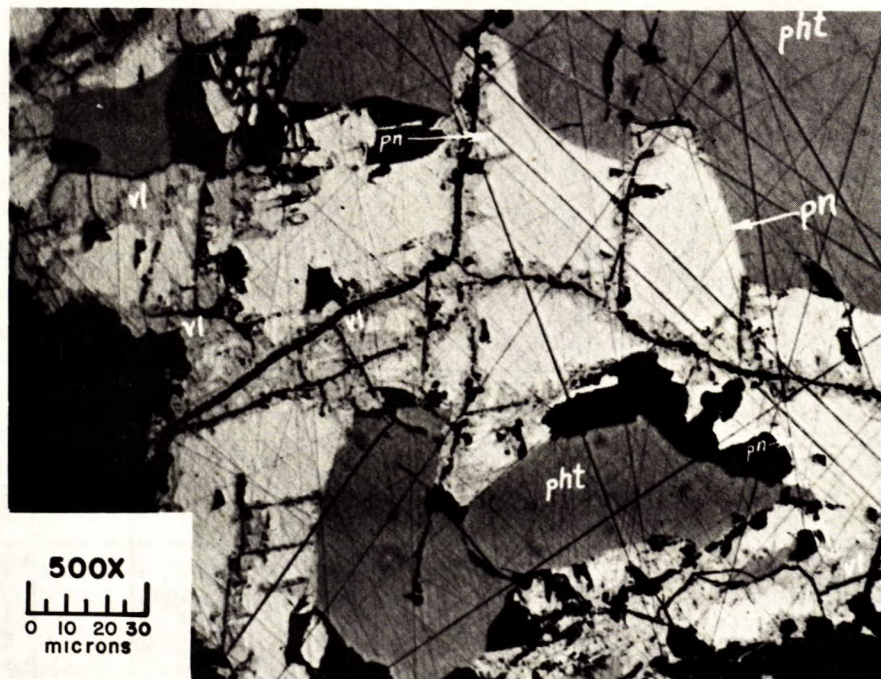


Figure 4 - Photomicrograph of a polished section showing pentlandite (pn) partially altered to violarite (v1). The areas marked (pht) are pyrrhotite and the black areas are pits on the polished surface. The straight lines are scratches on the polished surface and the curved ones are fractures in the minerals.

#### DETAILS OF INVESTIGATION

A test was run using a high intensity wet magnetic separator to determine the degree of association of copper and nickel minerals with pyrrhotite.

A series of tests, each consisting of floating three successive rougher concentrates, was made to study the effect on selectivity of different reagents and treatments.

Selective flotation tests were then carried out to try to produce separate clean chalcopyrite, pentlandite and pyrrhotite concentrates.

As the pyrrhotite concentrates contained a fair amount of copper and nickel minerals finely disseminated throughout pyrrhotite particles, attempts were made to recover these minerals by sulphatizing roast followed by water leaching.

Magnetic Separation

Test 1

A 1000 g sample of ore was stage ground to -100 m and fed to a Jones high intensity wet magnetic separator set at 5 amperes. A pyrrhotite concentrate, a middling and a tailing were produced.

TABLE 3

Results of Magnetic Separation

Product	Weight %	Analysis* %			Distribution %		
		Cu	Ni	Fe	Cu	Ni	Fe
Pyrrhotite conc	58.4	0.38	1.54	36.9	34.4	67.8	76.6
Middling	20.2	0.94	1.09	16.9	29.4	16.6	12.2
Tailing	21.4	1.09	0.97	14.6	36.2	15.6	11.2
Feed (calcd)	100.0	0.65	1.33	28.0	100.0	100.0	100.0

\*From Internal Report MS-AC-63-170

Rougher Flotation

Test 2

A 2000 g sample of ore was ground for 30 min to 78% -200m and floated using the following procedure.

TABLE 4

Reagents and Conditions of Rougher Flotation

Operation	Time min	Reagents lb/ton		pH
No 1 conditioning	5	Xanthate Z-6	0.08	7.2
		Dowfroth 250	0.02	
No 1 flotation	8	Dowfroth 250	0.04	
No 2 conditioning	2	Xanthate Z-6	0.08	6.8
No 2 flotation	8	Dowfroth 250	0.02	
No 3 conditioning	5	Xanthate Z-6	0.08	6.3
		Cu SO <sub>4</sub>	1.0	
No 3 flotation	8	Dowfroth 250	0.02	

TABLE 5

Flotation Results at Natural pH

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
No 1 conc	7.0	3.09	4.80	35.5	22.2
No 2 conc	14.1	1.31	3.25	30.3	30.3
No 3 conc	20.1	0.39	1.50	12.9	19.9
Flotn tailing	58.8	0.22	0.71	21.3	27.6
Feed (calcd)	100.0	0.61	1.51	100.0	100.0
Bulk conc (calcd)	41.2	1.16	2.64	78.7	72.4

\*From Internal Report MS-AC-63-170

Tests 3 and 4

These tests were similar to Test 1 except that lime (Test 3) and soda ash (Test 4) in ratio of 1.0 lb/ton were added to the first conditioning step. In the test with lime, the pH readings were 8.9, 7.8 and 7.4 in the three successive conditioning stages. The pH readings were 7.6, 7.2 and 7.1 in the test with soda ash.

TABLE 6

Flotation Results at Low Alkalinity

Test	Product	Weight %	Analysis* %		Distribution %	
			Cu	Ni	Cu	Ni
3 Lime 1 lb/t	No 1 conc	6.4	2.24	3.65	22.5	15.0
	No 2 conc	15.7	1.62	3.85	40.0	38.9
	No 3 conc	23.6	0.48	1.54	17.8	23.4
	Flotn tailing	54.3	0.23	0.65	19.7	22.7
	Feed (calcd)	100.0	0.64	1.55	100.0	100.0
	Bulk conc (calcd)	45.7	1.12	2.63	80.3	77.3
4 Soda ash 1 lb/t	No 1 conc	6.4	3.83	3.75	38.5	15.8
	No 2 conc	20.0	0.94	3.25	29.6	42.8
	No 3 conc	20.8	0.34	1.40	11.1	19.2
	Flotn tailing	52.8	0.25	0.64	20.8	22.2
	Feed (calcd)	100.0	0.64	1.52	100.0	100.0
	Bulk conc (calcd)	47.2	1.07	2.50	79.2	77.8

\*From Internal Report MS-AC-63-173

Tests 5, 6 and 7

This group of tests was carried out using the procedure described in Table 4, except that they were preceded by a 20 min aeration period, without an alkali added in Test 5, with 1.0 lb lime/ton in Test 6 and 1.0 lb soda ash/ton in Test 7.

TABLE 7

Flotation Results After Aeration at Low Alkalinity

Test	Product	Weight %	Analysis* %		Distribution %	
			Cu	Ni	Cu	Ni
5 natural pH	No 1 conc	12.5	3.20	4.01	62.7	34.1
	No 2 conc	10.2	0.47	2.71	7.5	18.8
	No 3 conc	17.7	0.30	1.63	8.3	19.6
	Flotn tailing	59.6	0.23	0.68	21.5	27.5
	Feed (calcd)	100.0	0.64	1.47	100.0	100.0
	Bulk conc (calcd)	40.4	1.24	2.64	78.5	72.5
6 1.0 lb/t lime	No 1 conc	12.3	3.20	5.30	63.8	43.4
	No 2 conc	9.3	0.47	2.40	7.1	14.9
	No 3 conc	20.8	0.31	1.40	10.5	19.4
	Flotn tailing	57.6	0.20	0.58	18.6	22.3
	Feed (calcd)	100.0	0.62	1.50	100.0	100.0
	Bulk conc (calcd)	42.4	1.18	2.75	81.4	77.7
7 1.0 lb/t soda ash	No 1 conc	10.7	3.30	4.97	61.2	34.0
	No 2 conc	8.3	0.47	2.95	6.8	15.6
	No 3 conc	25.0	0.27	1.59	11.7	25.4
	Flotn tailing	56.0	0.21	0.70	20.3	25.0
	Feed (calcd)	100.0	0.58	1.57	100.0	100.0
	Bulk conc (calcd)	44.0	1.04	2.67	79.7	75.0

\*From Internal Report MS-AC-63-176, 186 and 193

In the subsequent rougher flotation tests, the following changes in the procedure as outlined in Table 4 were made:

- 1) The No 1 conditioning period was increased to 20 min and the No 1 flotation was decreased to 5 min to improve the copper grade in the first concentrate and to reduce its nickel content.
- 2) The addition of Dowfroth 250 to the No 1 conditioning stage was discontinued.

- 3) The No 3 flotation period was increased to 15 min to obtain higher copper and nickel overall recoveries. An addition of 0.04 lb Z-6/ton was made after 8 min during this flotation.

Test 8

An addition of 4.0 lb lime/ton was made to the No 1 conditioning stage (20 min in this test). The pH readings were 10.1, 8.9 and 8.0 respectively, during the Nos 1, 2 and 3 conditioning stages.

TABLE 8

Flotation Results at High Alkalinity

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
No 1 conc	3.6	7.70	5.15	44.1	12.1
No 2 conc	18.6	0.99	3.66	29.3	44.5
No 3 conc	32.2	0.28	1.34	14.3	28.2
Flotn tailing	45.6	0.17	0.51	12.3	15.2
Feed (calcd)	100.0	0.63	1.53	100.0	100.0
Bulk conc	54.4	1.01	2.38	87.7	84.8

\*From Internal Report MS-AC-63-903

Test 9

In this test, the Reagent Z-6 was replaced by Z-200 during the No 1 conditioning stage.

TABLE 9

Flotation Results With Reagent Z-200

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
No 1 conc	4.8	6.03	2.25	46.0	7.0
No 2 conc	16.9	0.59	3.73	15.9	40.6
No 3 conc	28.5	0.40	1.80	18.1	33.1
Flotn tailing	49.8	0.25	0.60	20.0	19.3
Feed (calcd)	100.0	0.63	1.55	100.0	100.0
Bulk conc	50.2	1.20	2.50	80.0	80.7

\*From Internal Report MS-AC-63-830

Test 10.

An addition of 4.0 lb lime/ton was made to the No 1 conditioning stage which gave pH readings of 10.0, 9.0 and 8.1, respectively, in the Nos 1, 2 and 3 conditioning stages. Reagent Z-200 and Aero Promoter 404 additions of 0.04 lb/ton were used instead of Z-6 during the same conditioning stage. The addition of Aero Promoter 404 was necessary to assist the flotation of copper at this high pH.

TABLE 10 .

Flotation Results With Reagent Z-200 at High Alkalinity

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
No 1 conc	3.8	7.54	1.22	47.7	2.9
No 2 conc	15.3	0.72	5.50	18.3	53.5
No 3 conc	35.2	0.36	1.30	21.1	29.1
Flotn tailing	45.7	0.17	0.50	12.9	14.5
Feed (calcd)	100.0	0.60	1.57	100.0	100.0
Bulk conc	54.3	0.95	2.48	87.1	85.5

\*From Internal Report MS-AC-851

Test 11

In this test, the pulp was aerated for 20 min with 4.0 lb lime/ton. Even after this treatment, a 20 min conditioning with Z-200 and Aero Promoter 404 additions of 0.04 lb/ton was necessary to float a first concentrate of relatively high copper grade.

TABLE 11

Flotation Results With Reagent Z-200 After Aeration  
at High Alkalinity

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
No 1 conc	2.6	9.50	0.89	39.3	1.5
No 2 conc	17.9	1.02	4.52	29.0	53.9
No 3 conc	28.5	0.34	1.35	15.4	25.6
Flotn tailing	51.0	0.20	0.56	16.3	19.0
Feed (calcd)	100.0	0.63	1.50	100.0	100.0
Bulk conc	49.0	1.08	2.48	83.7	81.0

\*From Internal Report MS-AC-63-1100

A few other rougher flotation tests were run at finer grinds. These gave results inferior to those obtained with the 30 min grinding periods.

Selective Flotation

Test 12

A 2000 g sample of ore was ground for 30 min to 76% -200 m and floated using the following procedure.



TABLE 12

Reagents and Conditions of Selective Flotation

Operation	Time min	Reagents lb/ton		pH
Cu conditioning	25	Reagent Z-200	0.04	10.0
		Aero Promoter 404	0.04	
		Lime	4.0	
		After 20 min: TSP*	0.4	
Cu rougher flotation	5	Dowfroth 250	0.02	
Cu cleaner flotation	4	Reagent Z-200	0.01	9.0
Ni conditioning	2	Xanthate Z-6	0.08	8.9
Ni rougher flotation	8	Dowfroth 250	0.02	
Ni cleaner flotation	5	Xanthate Z-6	0.01	8.8
Pyrrhotite conditioning	5	Xanthate Z-6	0.08	8.1
		Cu SO <sub>4</sub>	1.0	
Pyrrhotite flotation	15	Dowfroth 250	0.02	
		After 8 min:		
		Xanthate Z-6	0.04	

\*Tetra Sodium Pyrophosphate

TABLE 13

Results of Selective Flotation

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
Cu cl conc	1.9	13.54	0.92	42.8	1.1
Cu cl tailing	1.9	1.54	1.54	4.9	1.8
Ni cl conc	7.6	0.95	8.70	12.0	42.0
Ni cl tailing	7.7	0.49	2.38	6.3	11.6
Pyrrhotite conc	35.2	0.36	1.30	21.1	29.0
Flotn tailing	45.7	0.17	0.50	12.9	14.5
Feed (calcd)	100.0	0.60	1.57	100.0	100.0

\*From Internal Report MS-AC-63-851

Test 13

This test was similar to Test 12 except that the lime was added to the grinding stage. In spite of this, a 25 min conditioning stage was necessary to initiate the copper flotation. In this test, the copper cleaner flotation was made with 0.2 lb lime/ton (pH-11.4) and the nickel cleaner flotation with 0.5 lb lime/ton (pH-10.6)

TABLE 14

Flotation Results With Cleaning at High Alkalinity

Product	Weight %	Analysis* %		Distribution %	
		Cu	Ni	Cu	Ni
Cu cl conc	1.0	19.88	1.00	29.5	0.7
Cu cl tailing	0.7	3.06	1.85	3.1	0.9
Ni cl conc	6.0	3.10	8.85	27.6	35.1
Ni cl tailing	7.2	0.73	2.94	7.9	14.0
Fe conc	33.5	0.35	1.34	17.4	29.6
Flotn tailing	51.6	0.19	0.58	14.5	19.7
Feed (calcd)	100.0	0.67	1.51	100.0	100.0

\*From Internal Report MS-AC-63-1112

A few other tests were carried out in which attempts were made to increase the nickel grade by cleaning procedure with Guartec or after heating the pulp to 90°F. These methods failed to improve the results.

Roasting and Leaching

The pyrrhotite concentrates produced in Tests 12 and 13 were combined for this part of investigation. The mixed product assayed 0.35% Cu and 1.32% Ni.

Test 14

A 50 g sample of pyrrhotite concentrate was pulped in 75 cc water containing 30 g Na<sub>2</sub>SO<sub>4</sub>. The pulp was filtered and the filter cake dried. Approximately 3 g of salt remained with the sample. This procedure was used to obtain an homogenous distribution of salt through the pyrrhotite concentrate for a sulphatizing roast.

The material was then placed in a furnace and the temperature was gradually raised from 300°C to 650°C over a period of three hours. The temperature of the roasted product was lowered to 225°C and held there for 10 min. The calcine was then leached in water at about 60°C for one hour. The residue was filtered, washed and assayed for copper and nickel.

TABLE 15

Results of Roasting and Leaching

Weight Lost %	Residue Analysis*		Extraction**	
	%		%	
	Cu	Ni	Cu	Ni
22	0.11***	0.54***	68.6	59.1

\*From Internal Report MS-AC-63-922

\*\*Calculated by difference

\*\*\*Expressed in % of original feed

Test 15

This test was similar to Test 14 except that the pyrrhotite concentrate was reground for 15 min prior to roasting and leaching.

TABLE 16

Results of Roasting and Leaching After Regrinding

Weight Lost %	Residue Analysis*		Extraction**	
	%		%	
	Cu	Ni	Cu	Ni
27	0.11***	0.45***	68.6	65.9

\*From Internal Report MS-AC-63

\*\*Calculated by difference

\*\*\*Expressed as % of original feed

A mineralogical examination of the pyrrhotite concentrate showed numerous fine particles of pentlandite disseminated through the pyrrhotite. The difficulty of dissolving nickel from pentlandite by a sulphatizing roast probably accounted for the low extraction.

### CONCLUSIONS

The sample of ore received assayed 0.62% Cu and 1.53% Ni. The copper and nickel minerals were finely intergrown with pyrrhotite and gangue, and complete liberation of the valuable minerals could not be obtained.

Several factors were studied in an attempt to obtain a differential flotation of chalcopyrite, pentlandite and pyrrhotite with the following results:

- a) Low alkalinity either with or without aeration was ineffective;
- b) High alkalinity with lime made the chalcopyrite flotation slightly selective;
- c) Reagent Z-200 appeared sharply selective for the copper flotation;
- d) High alkalinity in combination with Z-200 gave good results.

The best test results (Table 14) gave a copper concentrate assaying 19.88% Cu and 1.0% Ni, a nickel concentrate assaying 3.1% Cu and 8.85% Ni, and a pyrrhotite concentrate with 0.35% Cu and 1.34% Ni. The total copper and nickel recovered in these three concentrates was 75% and 65%, respectively.

Aeration of the pulp during conditioning, and addition of Quartec failed to improve the selectivity of the collectors used.

About two-thirds of the copper and nickel contained in the pyrrhotite concentrate was extracted by a sulphatizing roast followed by water leaching. More testing under a controlled atmosphere might improve this extraction.

The low grades and recoveries obtained for both copper and nickel concentrates were attributed to the intimate association of these minerals with the pyrrhotite. Consequently, it is doubtful if further improvement can be achieved with this type of ore using mineral dressing techniques.

The investigation showed that flotation of a bulk concentrate with a ratio of concentration of 2:1, recovered 80 to 85% of the copper and nickel. Although beyond the scope of the present test-work, a research investigation on the recovery of the metal values from this type of concentrate using "roast-leach" or other hydrometallurgical techniques might be worthwhile.

### ACKNOWLEDGEMENT

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