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MINES BRANCH INVESTIGATION REPORT IR 62-48

SAMPLING AND TESTING OF A MOLYBDENUM ORE FROM THE KENORA DISTRICT PROPERTY OF EVENLODE MINES LIMITED

by

G. O. HAYSLIP

MINERAL PROCESSING DIVISION

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SUMMARY OF RESULTS

The shipment of ore was crushed, sampled and assayed and was found to contain 0.95% MoS2.

Concentration tests showed an overall recovery of 67.8% of the molybdenite with 40.2% being obtained in a concentrate assaying 87.38% MoS₂.

The low recovery of molybdemum is believed to be due to the presence of an alteration product ferrimolybdite.

A series of pilot plant investigations confirmed the small scale tests.

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INTRODUCTION

Location of Property

The property from which this sample was said to have been taken consists of several claims at the east end of High Lake in Ewart township, about 30 miles east of Kenora, Ontario.

Shipment

The shipment, made up of 11 tons of ore, was received on July 21, 1961. The shipment was submitted by Dr. R. B. Graham of R. Bruce Graham and Associates Ltd., 54 St. Leonards Avenue, Toronto 12, Ontario.

Purpose of Investigation

In his letter of July 14, 1961, Dr. Graham requested that the shipment be crushed and sampled to determine the molybdenum and gold content of the ore. In addition, he requested that concentration tests be done to determine the grade of concentrate that could be produced, as well as the recovery of molybdenum.

Sampling and Assaying

The shipment of 11 tons of ore was crushed to minus 1/4 in. The ore was sampled by means of an automatic sampler which removed 2 per cent of the ore. This sample was crushed to minus 10 mesh and a final head sample was riffled out for chemical and mineralogical analyses. The remainder of the minus 10 mesh material was used for test work.

The chemical analysis of the sample was as follows:

	0.016 oz/ton
	0.38 "
-	0.95 per cent
-	0.16 " "
	2.27 " "
-	1.06 " "
-	trace*

* by spectrographic analysis

MINERALOGICAL EXAMINATION

Introduction

Several selected hand specimens of the ore and a sample of the minus 10 mesh head sample were submitted for microscopic examination. A sample of a flotation tailing was also submitted for examination.

Four polished sections of the hand specimens were prepared, but the molybdenite was completely lost in the polishing process and textural relationships could not be observed. The head sample and the tailing sample were separated into fractions by heavy liquids and the mineralogy of each fraction was determined by microscopical and X-ray diffractometer studies.

Results of Investigation

The metallic minerals present in the ore are pyrite, chalcopyrite, molybdenite, magnetite, bornite, cuprite, ferrimolybdite, and graphite. The chief non-metallic minerals are quartz, muscovite, and feldspar. The molybdenite is present as plates up to 1/8 inch thick. The ferrimolybdite, an oxidized product of molybdenite, occurs as a yellow coating on the rock surfaces and on molybdenite. Graphite and cuprite were observed in a narrow veinlet in the gangue.

The mineral content was determined by separating the head sample by means of heavy liquids and weighing the fractions. The mineralogy of each fraction was determined by means of the X-ray diffractometer and the results are given in Table 1.

TABLE 1

Mineral Content of Head Sample No. 1 as Determined by Heavy Liquid Separations and X-ray Diffractometer Analyses

<u>Mineral</u>	Weight %	
Quartz and Muscovite	96.7	
Pyrite	1.8	
Nolybdenite	0.65	
Chalcopyrite	0.50	
Magnetite	0.1	
Bornite, Cuprite and		
Ferrimolybdite	0.25	
Graphite	trace	
Total.	100.00	

* From Mineral Sciences Division Internal Reports No. MS-61-82, Sept. 12, 1961. and MS-61-866, Dec. 14, 1961, by Dr. W. Petruk.

The tailing sample was separated into fractions by means of heavy liquids (sp gr = 2.96 and 4.1). The minerals present in the 4.1 float and 4.1 sink fractions were identified microscopically and by X-ray diffraction and the mineral contents of these fractions were estimated visually. The results are shown in Table 2.

TABLE 2

Fraction	Quartz, etc. Wt %	Goethite Nt %	Jarosite Nt %	Molybdenite Nt %	Chalco- pyrite, Pyrite, Wt %
2,96 float	98.8	ang ene		end 440	
4.1 "	0.05	0.60	0.25	0.20	
4.1 sink				0.06	0.04
Tota1	98.85	0.60	0.25	0.26	0.04

Estimated Mineral Content of Tailing Sample

The molybdenite in the 4.1 sink is completely liberated, but the molybdenite in the 4.1 float is attached to grains of quartz.



Figure 1 - Photomicrograph of an oil immersion mount prepared from the heavy fraction in a flotation tailing. This fraction contains tiny molybdenite grains (black) intergrown with quartz (white).



Figure 2 - Photomicrograph of a polished section showing molybdenite (white) in gangue (grey). The black areas are pits in the polished section.

DETAILS OF INVESTIGATION

After sampling the shipment of ore, small scale tests were done on the minus 10 mesh material. Due to the difficulty of obtaining a sufficient quantity of rougher concentrate for cleaning purposes, it was decided to make a pilot plant test of the main reject from the sampling plant. After the pilot plant tests, more small scale tests were made.

Tests 1, 2 and 3

These tests were done to determine the optimum grind for the ore. Three thousand gram lots of ore were ground at 67 per cent solids with reagents and then floated.

Reagents and Conditions

<u>Operation</u>	<u>Reagents - 1b/ton</u>	Time <u>min</u>	рН
Test 1, Grind (54.3% -200m) Test 2, Grind (68.2% -200m)	Reagent 325 - 0.1 Reagent 325 - 0.1	12 25	
Test 3, Grind (73.7% -200m) Flotation	Reagent 325 - 0.1 Dowfroth 250 - 0.05 Dowfroth 250 - 0.02	30 5 5	7.8

Results of Test 1

Product	Weight %	Assay % MoS2	Distn % MoS2
Feed (calcd)	100.0	0.80	100.0
Rougher conc	2.6	19.30	62.5
Rougher tail	97.4	0.31	37.5

Results of Test 2

Product	Weight %	Assay % MoS2	Distn % MoS2
Feed (calcd)	100.0	0.92	100.0
Rougher conc	1.8	26.57	52.2
Rougher tail	98.2	0.45	47.8

Results of Test 3

Product	Weight %	Assay % MoS2	Distn % MoS2
Feed (calcd)	100.0	1.02	100.0
Rougher conc	2.5	20.50	´ 50 . 0
Rougher tail	97.5	0.52	50.0

Test 4

In this test, three lots of ore were ground and the rougher concentrates combined. The concentrates were filtered to remove some of the froth and after conditioning with reagents, the concentrate was cleaned once.

Reagents and Conditions

<u>Operation</u>	<u>Reagents</u> -	lb/ton	Time <u>min</u>	рН
Grind (64.3% -200m)	Z-2 00	- 0.02	20	
Flotation	Dowfroth 250	- 0.02	· 5	8.0
	Dowfroth 250	- 0.02	5	
Conditioning	Sodium cyanide	- 0.2	3	8.4
Cleaner flotation		~ • • =	. 3	
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Results of Test 4

Product	Weight	Assays %		Distn %	
AT OULD T	76	MoS2	Cu	MoS2	Cu
Feed (calcd)	100.0	0.87	0.17	10040	100.0
MoS2 c1 conc	0.6	68.25	3.24	47.1	11.8
MoS ₂ cl tailing	1.3	8.02	7.16	11.5	52.9
Rougher tailing	98.1	0.37	0.058	41.4	_ 35 .3

Test 5

Three lots of ore were ground, and conditioned with kerosene and pine oil and then floated. The rougher concentrate was filtered, ground, conditioned with reagents and cleaned twice.

Reagents and Conditions

Operation	<u>Reagents -</u>	1b/ton	Time <u>min</u>	PH
Grind (54.3% -200m)			12	
Conditioning	Kerosene Pine oil	- 0.06 - 0.06	5	7.9
Rougher flotation	Kerosene Pine oil	- 0.02 - 0.02	10	
Conditioning	Sodium silicate	- 0.1	5	
Cleaner flotation	M.I.B.C.	- 0.01	3	
Conditioning	Sodium silicate	- 0.1	5	
	Sodium cyanide	- 0.02		
Cleaner flotation	M.I.B.C.	- 0.01	2	

Resu1	ts	of	Test	5
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Weight	eight Assays %		Distn %	
16	MoS2	Cu	MoS2	Cu
100.0	0.87	0.15	100.0	100.0
0.4	87.38	0.92	40.2	2.7
0.2	55.84	10.69	12.6	14.4
1.4	9.22	4.93	15.0	47.3
98.0	0.29	0,053	32.2	35.6
	0.4 0.2 1.4	MoS2 100.0 0.87 0.4 87.38 0.2 55.64 1.4 9.22	MoS2 Cu 100.0 0.87 0.15 0.4 87.38 0.92 0.2 55.84 10.69 1.4 9.22 4.93	MoS2CuMoS2 100.0 0.870.15 100.0 0.487.380.9240.20.255.64 10.69 12.6 1.49.224.93 15.0

PILOT PLANT TESTS

Several pilot plant tests were made on the shipment of ore. Due to the small quantity of ore it was not possible to make the tests at a high feed rate, nor was it possible to try many variations in different tests.

A feed rate of 500 1b of ore per hour was used. The ore was ground in a 30 in. x 48 in. ball mill in closed circuit with a classifier. The classifier overflow went to a small conditioner and then to the flotation cells.

Rougher flotation was done in four No. 7 Denver cells followed by scavenger flotation in two No. 7 Denver cells. The scavenger concentrate was returned to the conditioner.

The rougher concentrate was fed to three No. 5 Denver cells for cleaning. The cleaner concentrate then was cleaned 3 times in No. 5 Denver cells with the cleaner tailing being returned to the previous cell. The cleaner tailing from the first three cells was sent to three other No. 5 Denver cells for copper flotation with the tailing from these cells being returned to the conditioner at the head of the rougher flotation.

After the first two mill runs it was observed that some of the molybdenite in the concentrate was locked in gangue and it was decided to place a ball mill in the circuit to grind the rougher concentrate. Since the amount of concentrate produced was very small, it was not possible to include a classifier in the grinding circuit and obtain controlled grinding.

A cyclone was also installed to treat the cleaner tailing from the first stage of cleaning before it went to copper flotation. This was to wash out the reagents that had been added in the molybdenite cleaning circuit.

Reagents used were potassium ethyl xanthate and Dowfroth 250, which were added to the ball mill. Sodium cyanide was added to the regrind mill and to the molybdenite cleaner cells. In one test kerosene and pine oil were added in place of the potassium ethyl xanthate and Dowfroth 250.

Filot Plant Test 1

Reagents and Conditions

Operation	Reagents - 1b/ton	Time <u>min</u>	pll
Grind (81.4% -200m)	Pot.ethy1 xanthate - 0.1		,
	Dowfroth 250- 0.04		•
Rougher flotation		8	8.0
Scavenger flotation	,	4	

Pilot Plant Test 1 (contd)

Reagents and Conditions

Operation	Reagents		1b/ton	Time min	рН
Cleaner flotation	Sodium cyanide		0,05	6	8.1
	Sodium cyanide	-	0.05	3	

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Results of Pilot Plant Test 1

Product	Weight %	Assays % MoS2	Distn % MoS2
Feed (calcd)	100.0	1.07	100.0
MoS2 conc	0.7	76,90	50.5
Rougher tailing	99.3	0.53	49.5

Pilot Plant Test 2

Reagents and Conditions

<u>Operation</u>	<u>Reagents – 1b/ton</u>	Time min pH	_
Grind (57.6% -200m)	Pot. ethyl xanthate - 0.1		
	Dowfroth 250- 0.04		
Rougher flotation		8 7.9	
Scavenger flotation		4	
Cleaner flotation	Sodium cyanide - 0.05	6 8.1	
	Sodium cyanide - 0.05	3	

Product	Weight %	Assays % MoS2	Distn % MoS2
Feed (calcd)	100.0	0.92	100.0
MoS2 conc	0.8	72.23	63.0
Rougher tailing	99.2	0.34	37.0

Pilot Plant Test 3

Reagents and Conditions

Operation	Reagents		lb/ton	Time <u>min</u>	<u>pH</u>
Grind (55.6% -200m)	Kerosene	-	0.1		
	Pine oil	-	0.05	•	
Rougher flotation				8	7.8
Scavenger flotation	Pine oil	-	0.01	4	
Rougher conc regrind	Sodium cyanide		0.05		
Cleaner flotation	Sodium cyanide	-	0.05	6	
	Sodium cyanide		0.05	3	
Copper flotation	Pot.ethyl xant	hate	∎-0 . 02	4	
	Dowfroth 250		0.01		
Copper cl flotation			-	4	

Results of Pilot Plant Test 3

	Weight	Assays	3 %	Distn %	
Product	70	NoS2	Cu	MoS2	Cu
Feed (calcd)	100.0	1.09	0.19	1.00.0	100.0
MoS ₂ conc	0.8	76.75	2.08 ·	56.0	9.1
Cu conc	0.7	12.30	15.20	8.2	56.7
Rougher tailing	98.5	0.40	0.065	35.8	34.2

Pilot Plant Test 4

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Reagents and Conditions

<u>Operation</u>	Reagents –	1b/ton	Time <u>min</u>	рH
Grind (58.4% -200m)	Pot.ethyl xanthate Dowfroth 250	- 0.1 - 0.04		
Rougher flotation			8	8.1
Scavenger flotation			4	
Rougher conc regrind	Sodium cyanide	- 0.05		
Cleaner flotation	Sodium cyanide	- 0.05	6	
	Sodium cyanide	- 0.05	3	

Results of Pilot Plant Test 4

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Product	Weight %	Assays % MoS2	Distn % MoS2
Feed (calcd)	100.0	1.10	100.0
MoS2 conc	0.9	79.45	65.5
Rougher tailing	99.1	0.38	34.5

CONCLUSIONS

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The shipment of ore was crushed, sampled and assayed, and was found to contain an amount of molybdenite which was calculated as 0.95 per cent molybdenum disulphide. A slight error results as some of the molybdenum is present as the mineral ferrimolybdite, which is reported as molybdenum disulphide.

The best results in concentrating the ore were obtained in Test 5 where a concentrate assaying 87.38 per cent molybdenum disulphide was obtained with a recovery of 40.2 per cent. Overall rougher recovery was 67.8 per cent.

The low recovery is believed to be due to the presence of the mineral ferrimolybdite. This is an oxidation product of molybdenite and was estimated to be present in an amount up to 0.25 per cent. If this estimate is correct, the recovery of the molybdenum, present as the sulphide, is over ninety per cent.

In Test 1, 2 and 3 it was shown that over-grinding of the ore has a harmful effect on recovery. However, microscopic investigation of the flotation concentrate showed the presence of a large amount of molybdenite associated with gangue. It seems to be necessary, therefore, to make an initial coarse grind before rougher flotation, regrind the concentrate to make good liberation, and then make a high grade concentrate by cleaning.

A pilot plant test was made on the main part of the shipment. Due to the limited amount of ore, the feed rate was kept low and results were no better than in the small scale tests.

Due to the high ratio of concentration in this type of ore, it is difficult to obtain a sufficient quantity of concentrate for proper testing. When the regrind mill was used it was greatly underloaded and no control could be made of the grinding. Similarly, in the cleaning stages there was an insufficient amount of concentrate present to load the cells to their proper capacity.

Any additional work should be done on larger quantities of fresh, unoxidized ore.

ACKNOW LEDG EMENT

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