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MINES BRANCH INVESTIGATION REPORT IR 67-27

LABORATORY AND PILOT PLANT INVESTIGATION ON MOLYBDENITE-BISMUTH ORES FROM MOLY HILL MINING CORPORATION LIMITED, ROUYN, QUEBEC

by

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MINERAL PROCESSING DIVISION

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Mines Branch Investigation Report IR 67-27

by

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SUMMARY OF RESULTS

Three ore samples were received for pilot plant and laboratory investigation. These were identified as Central Zone, West Zone "A" and West Zone "B", and contained 0.13 to 0.46% MoS_2, 0.028 to 0.25% Bi and 0.03 to 0.66 oz Ag/ton.

Bulk flotation tests were made on each of the ores separately, and on two appropriate combinations. The recoveries obtained varied from 89.5 to 97.6% for the molybdenite, 55.4 to 89.0% for the bismuth and 58.1 to 97.2% for the silver, with the best results corresponding to the higher grade ores.

All the bulk concentrates were leached in hydrochloric and nitric acid to extract the bismuth and to remove some of the contaminants. The molybdenite concentrates left after leaching met the specifications of commercial grade, with the exception of the residue from the West Zone "B" ore which assayed 81.6% MoS_2. However, it was possible to obtain a final molybdenite concentrate of over 90% MoS_2 with this ore by either reflotation after leaching or by molybdenite separation from bulk concentrate before leaching.

A molybdenite concentrate meeting some specifications for lubricant-grade was produced by regrinding and cleaner flotation of the leached concentrate from the Central Zone ore.

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CONTENTS

LIST OF TABLES

LIST OF TABLES (continued)

INTRODUCTION

Moly Hill Mining Corporation Limited holds 33 claims situated about 15 miles east of Malartic, Northwestern Quebec. Although only a small part of this property has been probed by trenching and drilling, 300,000 tons of ore averaging 0.20% MoS_2 and 0.06% Bi have been already indicated with the main mineralized zone still opened at north and south. About one half of this tonnage was reported to be recoverable by open pit mining.

Because the company intends to process this ore in the near future, a pilot plant investigation was required to develop the best flowsheet for recovering the molybdenite and bismuth values. In some instances, the pilot plant work was complemented by laboratory tests.

Shipments

A total of three shipments of ore was received from the property. These were said to be from different points of the two principal ore zones. Identification and weight of each shipment are shown below:

Designation	Date Received	Weight
Central Zone	19 - 8 - 66	66.5 tons
West Zone "A"	2 - 8 - 66	25.5 tons
West Zone "B"	5 - 1 - 66	615 lb

As will be shown later in this report, these three samples differed mineralogically.

Purpose of Investigation

Initially, the investigation was aimed at the best technique for a separated treatment of each type of ore. However, when the company indicated an interest for processing the ore from both zones together, tests were made on combined samples, namely, Central Zone plus West Zone "A" and Central Zone plus West Zone "B". The latter was considered to be more representative of the whole open pit orebody.

Although the main objective of the investigation was a high recovery of molybdenum and bismuth with the production of final molybdenite concentrate of commerical grade (90% MoS_2), an attempt was also made to meet the specifications of lubricant-grade (+98% MoS_2, etc...).

Sampling and Analysis

As each lot of ore was crushed for processing, hand specimens were selected for mineralogical examination. At appropriate size, representative head samples were taken for chemical analysis.

TABLE 1

Chemical Analysis* of Head Samples

Elements	Central Zone	West Zone "A"	West Zone "B"
Molybdenite (MoS_2)	0.46 %	0.13 %	0.13%
Bismuth (Bi)	0.028%	0.048%	0.25%
Copper (Cu)	0.003%	0.010%	0.02%
Lead (Pb)	$<$0.01 %	$<$0.01 %	0.02%
Iron (Fe)	0.38 %	0.33 %	0.56%
Insoluble	95.45 %	97.30 %	98.14%
Silver (Ag)	0.03 oz/ton	0.04 oz/ton	0.66 oz/ton

*From Internal Reports MS-AC-66-248, 770 and 811.

Mineralogical Examination*

Hand specimens and portions of the head samples were submitted to the Mineralogy Section of the Mineral Sciences Division for microscopic examination.

Polished sections were prepared from each type of ore and the minerals present were identified by microscopical and X-ray diffraction studies.

Central Zone Ore

Molybdenite is the most abundant valuable mineral, although bismuthinite and native bismuth also show in minor quantity. Other metallic minerals, such as chalcopyrite, argentite, stannite, magnetite, goethite and pyrite were identified in the ore. The non-metallic minerals consist of quartz, feldspar, muscovite and amphibole.

Molybdenite occurs mostly as free masses, but a few minute inclusions were also observed in gangue minerals, particularly in quartz.

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*From Investigation Report IR 66-46 and Internal Report MS-66-71.

Bismuthinite, which accounts for about 75% of the total bismuth, is present mostly as large free grains. The native bismuth occurs largely as inclusions in bismuthinite and chalcopyrite.

Only small quantities of chalcopyrite, pyrite, argentite and stannite were found in the sections examined. Some grains of these minerals were free while the others were intergrown together.

West Zone "A" Ore

This sample contains both molybdenite and bismuth in relatively small amount. The other important constitutents are magnetite, chalcopyrite, pyrite, pyrrhotite, quartz and feldspar.

Molybdenite is present as large flakes. Some of these contain inclusions of magnetite in sufficient quantity to become strongly magnetic.

The bismuth-bearing minerals are bismuthinite, native bismuth, an unidentified bismuth mineral, bismite, bismutite and arsenobismite. Bismuthinite, which accounts for about 70% of the total bismuth, is present mostely as free grains while the other bismuth minerals are intergrown with each other.

West Zone "B" Ore

Contrary to the other ores, bismuth is the most valuable constituent in this sample, while molybdenite is present in lesser amounts.

The polished sections show two general type of material: a coarse-grained assemblage consisting of feldspar, colourless mica, and mineralized quartz; and a medium-grained rock composed of feldspar, quartz, biotite, peroxene and amphibole. The mineralized quartz contains grains and masses of molybdenite, bismuth-bearing minerals, chalcopyrite and galena.

Molybdenite occurs as bundles of flakes and is relatively free of impurities. The bundles vary from about 0.1 to 5 mm in size.

The bismuth-bearing mineral grains and masses are composed of bismuthinite (Bi_2S_3), emplectite ($BiCuS_2$), two unidentified minerals, native bismuth (Bi), bismutite $(BiO)_2CO_3$), bismite (Bi_2O_3) and arsenobismite ($Bi_2AsO_4(OH)_3$). Bismuthimite is the principal bismuth-bearing mineral. It occurs largely as irregular masses and contains grains of an unidentified mineral, native bismuth and emplectite. The X-ray diffraction pattern of the unidentified mineral is similar to that of a mixture of bismuthinite and arsenobismite, but it could not be correlated to that of any known mineral. The emplectite contains minute inclusions of native bismuth, chalcopyrite and another unidentified mineral. This mineral is present only as very fine grains too small to be dug out for X-ray diffraction study. The bismuth-bearing mineral grains and masses are altered to bismite, bismutite and arsenobismite along the edges and fractures. In some places, this altered zone contains inclusions of native bismuth.

A few grains of chalcopyrite and galena are present in the ore. Some of the galena contains inclusions of native bismuth and some is intergrown with chalcopyrite.

OUTLINE OF INVESTIGATION

The ore samples received from the Central Zone and from the West Zone "A" were both in sufficient amounts for testing in the pilot plant. This testwork consisted of bulk flotation of molybdenite and bismuth from each of the two ores separately, and also from a mixture of the two. Some tests were made after grinding to -35 and -48 mesh. In each case, the bulk concentrates were treated in hydrochloric acid and nitric acid to eliminate undesired impurities and also to extract the bismuth.

Because of the difficult accessibility to the high bismuth ore (West Zone "B"), only 600 lb was supplied for testing. This limited the scope of the investigation to laboratory scale. Bulk flotation tests were done to determine the effect of grinding, use of sodium silicate and duration of flotation on grade and recovery. In view of the company's interest in treating mixed ore from the West Zone "B" and the Central Zone, representative samples (100 lb each) of these two lots were blended and floated using optimum conditions found previously. The bulk concentrates from the mixed ore and from the West Zone "B" ore were treated in hydrochloric and nitric acid to extract bismuth and eliminate contaminants. The molybdenite concentrate left after leaching of the bulk concentrate from the West Zone "B" ore assayed only 81.6% MoS_2. To upgrade this product, two techniques were investigated, namely, reflotation of the leached concentrate and separation of the molybdenite from the bulk concentrate prior to leaching.

Finally, the high molybdenite content (97.6% MoS_2) of the leached concentrate from the Central Zone ore prompted further investigation to achieve some specifications of lubricant-grade from this product.

DETAILS OF INVESTIGATION

Part i - Pilot Plant

(a) Description of Equipment and Layout

The general layout for all the pilot plant work is illustrated by the following flowsheet.

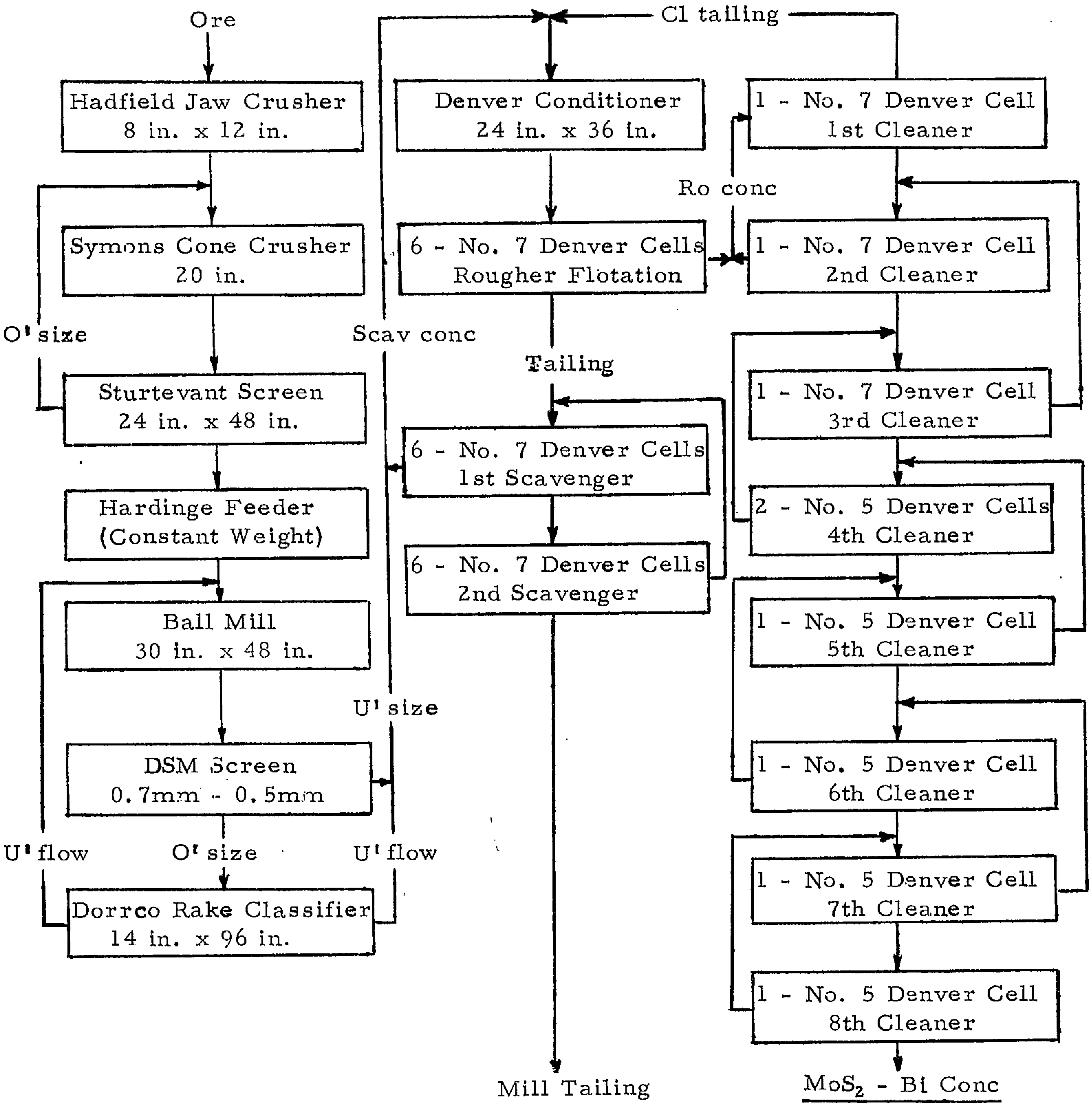

Figure 1. Pilot Plant Flowsheet.

The raw ore, fed successively to the Hadfield and Symons crushers, was reduced to either -3/8 in. or -1/4 in. and stored in a bin. From this bin, the crushed ore was drawn by a Hardinge feeder and fed to a ball mill at the rate of about 950 lb/hr.

To avoid excessive production of fines, the ground material was classified successively by a DSM screen and a Dorrco rake classifier. The undersize product from the screen and the overflow of the classifier constituted the flotation feed. In order to obtain a feed substantially either minus 35 mesh or 48 mesh, the conditions as shown in Table 2 were maintained.

TABLE 2

Pilot Plant Operating Conditions

Conditions	Grinding	
	-35 m	-48 m
Ball mill feed	-3/8 in.	-1/4 in.
Ball charge	1500 lb	1800 lb
DSM screen opening	0.7 mm	0.5 mm
Recirculating load	75 %	160 %
Pulp density:		
Ball mill discharge	69 % solids	65 % solids
Classifier sand	73 % "	70 % "
Flotation feed	34 % "	31 % "

By using these parameters, the desired fineness of flotation pulp was obtained without changing the feed rate to the ball mill. The success of the technique was consistent with the two types of ore treated in the pilot plant. This is illustrated by the results of the screen tests shown in Table 3.

TABLE 3

Size Analyses of Flotation Feed

Size	Central Zone		West Zone "A"		Central + West Zone "A"	
	-35 m	-48 m	-35 m	-48 m	-35 m	-48 m
+ 35 mesh	2.4	-	2.1	-	2.2	-
- 35+ 48 "	5.7	1.4	5.6	1.2	5.6	1.4
- 48+ 65 "	12.2	6.0	9.1	5.4	10.7	5.9
- 65+100 "	14.7	14.2	14.2	14.0	14.5	14.3
-100+150 "	15.7	17.6	16.0	18.4	14.9	17.6
-150+200 "	9.4	11.1	12.0	11.8	11.3	11.5
-200 "	39.9	49.7	41.0	49.2	40.8	49.3
Totals	100.0	100.0	100.0	100.0	100.0	100.0

After a conditioning stage, the flotation pulp was fed successively to three groups of six No. 7 Denver Sub A cells for rougher and scavenger flotation. The rougher concentrate was cleaned three times in No. 7 cells and five times in No. 5 cells. In some cases, small changes were made to this basic circuit. The modifications made, are noted in the details of the test.

(b) Bulk Flotation

Central Zone, Tests 1 - 3

Conditions for Tests 1 and 2 were similar, except that flotation feed was ground to -35 mesh and -48 mesh respectively. In Test 3, the grinding was also -35 mesh, but Aerofloat 33 was used in attempt to increase the bismuth recovery. The amounts of reagents and the points of addition are shown in Table 4.

TABLE 4

Reagents and Conditions (Tests 1 - 3)

Point of Addition	Reagents	lb/ton of ore	pH
Ball mill	Kerosene	0.06	
Conditioner	1:1 Pine oil-Dowfroth 250 Xanthate Z-6 Test 3: Aerofloat 33	0.04 0.04 0.01	7.4-7.7
Rougher flotation	Dowfroth 200*	0.005-0.02	7.3-7.6
1st scavenger flotation	Xanthate Z-6	0.04	7.4-7.6
2nd scavenger flotation	Xanthate Z-6	0.04	7.4-7.7

*Water-soluble frother added in dilute form as required to obtain a stable froth.

TABLE 5

Flotation Results on Central Zone Ore

Test	Product	Weight %	Analysis*			Distribution %		
			%		oz/t			
			MoS_2	Bi	Ag	MoS_2	Bi	Ag
1 (-35m)	MoS_2-Bi conc	0.48	92.3	2.58	4.88	97.6	55.4	70.1
	Mill tailing	99.52	0.011	0.010	0.01	2.4	44.6	29.9
	Feed (calcd)	100.00	0.45	0.022	0.03	100.0	100.0	100.0
2 (-48m)	MoS_2-Bi conc	0.51	94.9	2.62	4.56	97.4	59.6	70.2
	Mill tailing	99.49	0.013	0.009	0.01	2.6	40.4	29.8
	Feed (calcd)	100.00	0.50	0.022	0.03	100.0	100.0	100.0
3 (Aerofloat 33)	MoS_2-Bi conc	0.50	89.9	3.67	6.00	97.4	72.3	75.0
	Mill tailing	99.50	0.012	0.007	0.01	2.6	27.7	25.0
	Feed (calcd)	100.00	0.46	0.025	0.04	100.0	100.0	100.0

*From Internal Report MS-AC-66-751, 755 and 761.

West Zone "A" Ore, Tests 4 - 6

Because of the rather low grade of this ore, the flotation flowsheet was modified as follows:

1. Both the first and second scavenger circuits were reduced to four cells;
2. The three No. 7 cleaner cells were replaced by two No. 5 twin-cell units;
3. In Test 6, two extra cleaning stages were added at the end of the circuit (each of these consisting of one No. 5 Denver cell).

The ore was ground to -35 mesh for Test 4 and to -48 mesh for Tests 5 and 6. The details of the flotation conditions are shown in Table 6.

TABLE 6

Reagents and Conditions (Tests 4 - 6)

Point of Addition	Reagents	lb/ton of ore	pH
Ball mill	Kerosene	0.10	
Conditioner	1:1 Pine oil-Dowfroth 250	0.04	7.5-7.7
	Aerofloat 33	0.01	
	Xanthate Z-6	0.08	
	Sodium silicate	0.10	
Rougher flotation	Dowfroth 200*	0.01-0.03	7.4-7.7
	Xanthate Z-6	0.04	
1st scavenger flotation	Dowfroth 200*	0.01-0.03	7.5-7.8
	Xanthate Z-6	0.02	
2nd scavenger flotation	Xanthate Z-6	0.02	7.7-7.9
Cleaner flotation	Xanthate Z-6	0.02	7.6-7.8
	Sodium silicate	0.05	

*Added in dilute form and as required to obtain a stable froth.

TABLE 7

Flotation Results on West Zone "A" Ore

Test	Product	Weight %	Analysis* % MoS$_2$	Analysis* % Bi	Analysis* oz/t Ag	Distribution % MoS$_2$	Distribution % Bi	Distribution % Ag
4 (-35m)	MoS$_2$-Bi conc	0.18	62.2	14.4	17.1	89.5	74.2	60.7
	Mill tailing	99.82	0.014	0.009	0.02	10.5	25.8	39.3
	Feed (calcd)	100.00	0.13	0.035	0.05	100.0	100.0	100.0
5 (-48m)	MoS$_2$-Bi conc	0.21	63.5	14.2	16.8	90.1	75.3	64.0
	Mill tailing	99.79	0.014	0.010	0.02	9.9	24.7	36.0
	Feed (calcd)	100.00	0.15	0.040	0.06	100.0	100.0	100.0
6 (Extra cleaning)	MoS$_2$-Bi conc	0.17	65.1	14.8	16.9	88.1	66.0	59.1
	Mill tailing	99.83	0.015	0.013	0.02	11.9	34.0	40.9
	Feed (calcd)	100.00	0.13	0.038	0.05	100.0	100.0	100.0

*From Internal Reports MS-AC-66-716 and 726.

Microscopic examination of the above concentrates showed high contamination by magnetite, pyrrhotite and pyrite. The magnetite was present mainly as inclusions in the molybdenite grains and could not be removed by magnetic separation without considerable loss of molybdenite.

Central Zone and West Zone "A", Tests 7 - 8

These two pilot plant tests were made on a mixed ore (5 tons from each zone) ground to -35 and -48 mesh respectively. The flotation flowsheet shown in Figure 1 was used with the reagents and conditions as shown in Table 8.

TABLE 8

Reagents and Conditions (Tests 7 - 8)

Point of Addition	Reagents	lb/ton of ore	pH
Ball mill	Kerosene	0.07	
Conditioner	1:1 Pine oil-Dowfroth 250	0.04	7.4-7.8
	Xanthate Z-6	0.06	
	Aerofloat 33	0.01	
	Sodium silicate	0.05	
Rougher flotation	Dowfroth 200*	0.01-0.02	7.3-7.5
1st scavenger flotation	Xanthate Z-6	0.04	7.4-7.7
2nd scavenger flotation	Xanthate Z-6	0.04	7.5-7.8

*Added in dilute form and as required to maintain a stable froth.

TABLE 9

Flotation Results on Mixed Ore from Central Zone and West Zone "A"

Test	Product	Weight %	Analysis*			Distribution %		
			%		oz/t			
			MoS_2	Bi	Ag	MoS_2	Bi	Ag
7 (-35m)	MoS_2-Bi conc	0.34	88.8	5.76	8.36	95.0	66.3	74.0
	Mill tailing	99.66	0.016	0.010	0.01	5.0	33.7	26.0
	Feed (calcd)	100.00	0.32	0.030	0.04	100.0	100.0	100.0
8 (-48m)	MoS_2-Bi conc	0.35	89.5	5.98	8.01	94.0	65.6	73.7
	Mill tailing	99.65	0.020	0.011	0.01	6.0	34.4	26.3
	Feed (calcd)	100.00	0.33	0.032	0.04	100.0	100.0	100.0

*From Internal Report MS-AC-66-770.

To determine the cause of the low bismuth recovery, representative samples from the mill tailings were sent to Dr. Petruk of the Mineral Sciences Division for microscopic examination. He reported that the only bismuth minerals present were those with optical properties similar to those of bismite, bismutite and arsenobismite.

The resistance of these minerals to flotation indicates that an increase in bismuth recovery is unlikely.

(c) Acid Leaching of Pilot Plant Bulk Concentrates, Test 9

Samples representative of the concentrates from the eight previous tests were used in a series of leaching tests. In these tests, a 100 g sample was agitated for three hours at 70°C in 200 mls of 37% hydrochloric acid, while another was treated similarly with the addition of 35 mls of concentrated nitric acid for the final 30 minutes.

The results of tests on concentrates from the same ore zone were practically identical and average figures only from each zone are shown in Table 10.

TABLE 10

<u>Results of Leaching Pilot Plant Bulk Concentrates</u>

Element	Central Zone			West Zone "A"			Central+West Zone "A"		
	Bulk Conc	HCl Residue	HCl+HNO$_3$ Residue	Bulk Conc	HCl Residue	HCl+HNO$_3$ Residue	Bulk Conc	HCl Residue	HCl+HNO$_3$ Residue
MoS$_2$ (%)	92.4	95.5	97.6	63.6	86.3	90.7	89.1	89.7	94.8
Bi "	2.96	0.07	0.05	14.5	0.30	0.06	5.87	0.16	0.04
Cu "	0.30	0.18	0.01	0.98	0.90	0.08	0.50	0.48	0.01
Pb "	<0.05	<0.01	<0.01	0.75	<0.05	<0.05	0.30	<0.05	<0.01
Fe "	1.17	1.08	0.35	4.07	1.91	0.48	1.66	1.29	0.36
Insol "	1.64	1.80	1.94	5.93	7.41	8.01	2.96	3.54	4.25
Ag(oz/ton	5.15	0.17	0.07	16.90	7.92	0.41	8.17	3.96	0.36

Note: Analyses from Internal Reports MS-AC-66-770, 780, 811 and 838.

Part 2 - Laboratory Tests

(a) Bulk Flotation

Reagents and operating conditions for bulk flotation on laboratory scale were basically the same in all tests. These are shown in Table 11.

TABLE 11

Procedure for Laboratory Bulk Flotation Tests

Operation	Reagents	lb/ton of ore	pH
Conditioning	Kerosene	0.08	7.9
	Xanthate Z-6	0.06	
	Aerofloat 33	0.02	
Rougher flotation	1:1 Pine oil-Dowfroth 250	0.02	7.9
Scavenger flotation	Kerosene	0.04	7.7
	Xanthate Z-6	0.04	
	1:1 Pine oil-Dowfroth 250	0.02	
Cleaner flotation (twice)	Kerosene	0.004*	7.8
	Xanthate Z-6	0.004*	
	1:1 Pine oil-Dowfroth 250	0.002*	
Recleaner flotation	Xanthate Z-6	0.001	7.6

*Amounts added to each of the two cleaning stages.

West Zone "B", Tests 10 - 15

These bulk flotation tests were done on 4,000 g samples with the exception of Test 15 which used 96,000 g of ore. The reason for this large sample was to provide enough concentrate for molybdenite separation from the other constituents.

Tests 10 to 12 followed exactly the procedure of Table 11 and differed only in the fineness of the grinding (-35, -48 and -65 mesh respectively). On the other hand, Tests 13 to 15 were all made on ore ground to -48 mesh, but with small additions of sodium silicate to increase the grade of the concentrates (0.20, 0.10 and 0.15 lb/ton of ore respectively). In order to obtain a better recovery of molybdenite and bismuth, rougher and scavenger flotation were both extended to 7 minutes in Test 15. The results of this series of tests are summarized in Table 12.

TABLE 12

Flotation Results on West Zone "B" Ore

Test	Product	Weight %	Analysis*			Distribution %		
			%		oz/t			
			MoS_2	Bi	Ag	MoS_2	Bi	Ag
10 (-35m)	MoS_2-Bi conc	0.46	21.8	40.3	-	86.8	85.5	-
	Middling	0.80	0.80	2.33	-	5.5	8.6	-
	Flot tailing	98.74	0.009	0.013	-	7.7	5.9	-
	Feed (calcd)	100.00	0.12	0.22	-	100.0	100.0	-
11 (-48m)	MoS_2-Bi conc	0.52	20.6	40.5	-	89.2	89.0	-
	Middling	0.88	0.35	1.41	-	2.6	5.2	-
	Flot tailing	98.60	0.010	0.014	-	8.2	5.8	-
	Feed (calcd)	100.00	0.12	0.24	-	100.0	100.0	-
12 (-65m)	MoS_2-Bi conc	0.50	20.1	40.6	-	88.2	86.9	-
	Middling	1.62	0.22	1.05	-	3.2	7.3	-
	Flot tailing	97.88	0.010	0.014	-	8.6	5.8	-
	Feed (calcd)	100.00	0.11	0.23	-	100.0	100.0	-
13 (Na_2SiO_3)	MoS_2-Bi conc	0.51	23.7	42.8	124.6	86.4	86.5	94.9
	Middling	0.95	0.86	1.94	3.61	5.9	7.3	5.1
	Flot tailing	98.54	0.011	0.016	ND	7.7	6.2	0.0
	Feed (calcd)	100.00	0.14	0.25	0.67	100.0	100.0	100.0
14 (Na_2SiO_3)	MoS_2-Bi conc	0.46	23.6	46.6	128.2	85.1	84.8	96.2
	Middling	0.90	1.01	2.61	2.53	7.1	9.3	3.8
	Flot tailing	98.64	0.010	0.015	ND	7.8	5.9	0.0
	Feed (calcd)	100.00	0.13	0.25	0.60	100.0	100.0	100.0
15 (Na_2SiO_3 and longer flotation)	MoS_2-Bi conc	0.56	23.6	46.8	127.0	89.6	88.8	97.2
	Middling	1.55	0.36	1.12	1.95	3.8	5.9	2.8
	Flot tailing	97.89	0.010	0.016	ND	6.6	5.3	0.0
	Feed (calcd)	100.00	0.15	0.29	0.73	100.0	100.0	100.0

*From Internal Report MS-AC-66-140, 217, 237, 316, 319 and 325.
ND - none detected.

Central Zone and West Zone "B", Test 16

Representative samples weighing 32,000 g were cut from each of the two ore zones. These were combined, ground to -48 mesh and floated according to the procedure shown in Table 11. However, as in Test 15, an addition of sodium silicate was made and the duration of flotation was extended for better results.

TABLE 13

Flotation Results on Mixed Ore from Central Zone and West Zone "B"

Product	Weight %	Analysis* %		oz/t	Distribution %		
		MoS_2	Bi	Ag	MoS_2	Bi	Ag
MoS_2-Bi conc	0.48	55.5	24.1	50.8	89.8	83.4	90.8
Middling	2.16	0.28	0.48	0.70	2.0	7.5	5.6
Flot tailing	97.36	0.025	0.013	0.01	8.2	9.1	3.6
Feed (calcd)	100.00	0.30	0.14	0.27	100.0	100.0	100.0

*From Internal Reports MS-AC-66-603 and 744.

(b) Acid Leaching of Laboratory Bulk Concentrates, Test 17

Samples of 75 g were cut from the concentrates produced in Tests 15 and 16. Each of these was leached for three hours in 300 mls of hydrochloric acid with an addition of 50 mls of nitric acid for the last 30 minutes. The temperature of the pulp was maintained a 70°C. Analysis of the concentrates before and after leaching are shown in Table 14.

TABLE 14

Results of Leaching Laboratory Bulk Concentrates

Element	West Zone "B"		Central Zone+West Zone "B"	
	Bulk conc	HCl+HNO$_3$ residue	Bulk conc	HCl+HNO$_3$ residue
MoS$_2$ (%)	23.6	81.6	55.5	94.0
Bi "	46.8	0.13	24.0	0.02
Cu "	3.32	0.19	1.34	0.02
Pb "	1.76	0.02	0.43	0.01
Fe "	2.67	1.91	2.68	0.21
Insol "	4.30	12.34	2.90	3.71
Ag (oz/ton)	127.0	56.1	50.8	5.18

Note: Analyses are from Internal Reports MS-AC-66-744, 1066, 1075 and 67-41.

(c) Upgrading Molybdenite Concentrate from West Zone "B" Ore

Two main techniques were investigated to obtain a final molybdenite concentrate assaying 90% MoS$_2$ from the West Zone "B" ore. The first method consisted of refloating the molybdenite concentrate after leaching in order to eliminate enough gangue to attain the desired grade. The second technique involved selective flotation of molybdenite of near-final purity from the bulk concentrate prior to acid leaching. This flotation stage removed appreciable gangue which would otherwise reported in the leached concentrate and lowered its grade accordingly.

(i) Reflotation of Leached Concentrate, Test 18

A 30 g sample, cut from the 81.6% MoS$_2$ residue shown in Table 14, was floated using 0.02 g of kerosene and 0.01 g of pine oil with the results shown in Table 15.

TABLE 15

Results of Reflotation of Molybdenite from Leached Concentrate

Product	Weight %	Analysis* %		Distribution %	
		MoS_2	Insol	MoS_2	Insol
MoS_2 Cl conc	83.0	93.0	2.3	94.6	15.4
MoS_2 Cl tailing	17.0	26.0	61.5	5.4	84.5
Feed (calcd)	100.0	81.6	12.3	100.0	100.0

*From Internal Report MS-AC-67-41.

(ii) Selective Flotation of Molybdenite Before Acid Leaching, Tests 19-24

Although several reagents were tried to isolate molybdenite from the bulk concentrate, only three of these gave satisfactory results. For confirmation, two tests were made with each of the successful reagents. All the tests were made on 50 g samples cut from the bulk concentrate of Test 15. The procedures used and the results obtained are shown in Table 16 and 17.

TABLE 16

Procedures for Selective Separation of Molybdenite from Bulk Concentrate

Test	Reagents	Separation Stages				
		1st	2nd	3rd	4th	5th
19	Sodium cyanide (g)	0.15	0.06	0.06	-	-
20	" " "	0.06	0.06	0.04	0.04	0.04
21	Sodium sulphide (g)	0.05	0.02	0.02	-	-
22	" " "	0.02	0.02	0.01	0.01	0.01
23	Potassium permanganate* (g)	0.05	0.02	0.02	-	-
24	" " "	0.02	0.02	0.01	0.01	0.01

*After an addition of sodium dioxide to reduce the pH to about 4.5.

TABLE 17

Results of Molybdenite Separation from Bulk Concentrate

Test	Product	Weight %	Analysis*							Distribution						
			%						oz/t	%						
			MoS_2	Bi	Cu	Pb	Fe	Insol	Ag	MoS_2	Bi	Cu	Pb	Fe	Insol	Ag
19 (NaCN)	MoS_2 conc	44.4	50.7	30.1	1.17	0.77	1.22	2.4	258.8	95.3	29.9	15.6	19.4	20.3	24.8	90.5
	Bi conc	55.6	2.0	56.2	5.04	2.55	3.83	5.8	21.8	4.7	70.1	84.4	80.6	79.7	75.2	9.5
	Feed	100.0	23.6	44.6	3.32	1.76	2.67	4.3	127.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
20 (NaCN)	MoS_2 conc	42.3	52.0	29.5	1.36	0.43	0.50	1.1	269.8	94.6	28.0	17.3	10.4	7.9	10.8	90.0
	Bi conc	57.7	2.2	55.9	4.76	2.73	4.26	6.7	22.2	5.4	72.0	82.7	89.6	92.1	89.2	10.0
	Feed	100.0	23.3	44.7	3.32	1.76	2.67	4.3	127.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
21 ($Na_2S.9H_2O$)	MoS_2 conc	33.9	69.8	15.0	2.44	0.15	1.57	1.0	38.6	96.8	10.4	24.9	2.9	19.9	7.9	10.3
	Bi conc	66.1	1.2	66.0	3.77	2.58	3.23	6.0	172.3	3.2	89.6	75.1	97.1	80.1	92.1	89.7
	Feed	100.0	24.4	48.7	3.32	1.76	2.67	4.3	127.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
22 ($Na_2S.9H_2O$)	MoS_2 conc	29.7	75.7	10.4	1.78	0.31	2.18	1.0	38.2	93.3	6.6	15.9	5.2	24.4	7.0	8.9
	Bi conc	70.3	2.3	62.0	3.97	2.37	2.87	5.7	164.5	6.7	93.4	84.1	94.8	75.6	93.0	91.1
	Feed	100.0	24.1	46.7	3.32	1.76	2.67	4.3	127.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
23 (SO_2+$KMnO_4$)	MoS_2 conc	29.6	73.4	13.9	1.83	0.08	1.26	0.8	58.0	94.5	8.6	16.3	1.4	13.9	5.5	13.5
	Bi conc	70.4	1.8	61.9	3.94	2.47	3.27	5.8	156.0	5.5	91.4	83.7	98.6	86.1	94.5	86.5
	Feed	100.0	23.0	47.7	3.32	1.76	2.67	4.3	127.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
24 (SO_2+$KMnO_4$)	MoS_2 conc	27.7	84.8	5.2	1.28	0.13	2.13	0.9	77.9	92.1	3.2	10.7	2.0	22.1	5.8	17.0
	Bi conc	72.3	2.8	60.7	4.10	2.38	2.88	5.6	145.5	7.9	96.8	89.3	98.0	77.9	94.2	83.0
	Feed	100.0	25.5	45.3	3.32	1.76	2.67	4.3	127.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

*From Internal Reports MS-AC-352, 416, 420 and 744.

Note: The following assays were calculated by difference from the feed analysis:

a) Silver in the molybdenite concentrate;

b) Copper, lead, iron and insoluble in the bismuth concentrate.

The remaining fractions of molybdenite concentrates from Tests 21 to 24 were combined to make up a 50 g sample. This product was leached for three hours in 200 mls of hydrochloric acid with the addition of 30 mls of nitric acid for the final 30 minutes.

In Table 18, a comparison is given between the final molybdenite concentrates obtained by the two techniques, namely, reflotation after acid leaching and separation of molybdenite from bulk concentrate followed by acid leaching.

TABLE 18

Analyses* of Upgraded Molybdenite Concentrates from West Zone "B" Ore

Element	Technique	
	Reflotation after Leaching	Separation Followed by Leaching
MoS_2 (%)	93.0	96.5
Bi "	0.12	0.02
Cu "	0.20	0.02
Pb "	0.02	0.02
Fe "	0.19	0.14
Insol "	2.34	1.31
Ag (oz/ton)	66.1	0.15

*From Internal Reports MS-AC-66-1075 and 67-41.

Part 3 - Production of Lubricant-Grade Molybdenite

References to specifications for lubricant-grade molybdenite vary slightly as shown in Table 19.

TABLE 19

Specifications for Lubricant-Grade Molybdenite*

Chemical:	U.S. Specification MIL-L-7866(Aer.)	ROCOL Powder	British Ministry Supply C.S. 2819
MoS_2	98.0% (min.)	99.1% (min.)	
Mo			59.0 % (min.)
S			38.5 % (min.)
Insol	1.5% (max.)	0.9% (max.)	
SiO_2			0.02% (max.)
Fe			0.10% (max.)
Cu			0.05% (max.)
Moisture	0.7% (max.)	0.02% (max.)	
Oil	0.5% (max.)	0.09% (max.)	
Volatils			1.10% (max.)
Ether extract			0.02% (max.)
Corrosion on:			
Copper	Nil	Nil	
Steel	Nil	Nil	
Fineness (% pass):			
100 mesh	100%	100%	100%
200 "	85%	98%	99%
240 "			95%
325 "	60%	88%	

*From Mineral Report 6 (1963), "Molybdenum" V.B. Schneider, Mineral Resources Division, Department of Mines & Technical Surveys.

Comparison of these specifications with the analyses of the leached concentrate from the Central Zone ore (97.6% MoS_2, 0.01% Cu, 0.35% Fe and 1.94 Insol) shows that the main objective would be the reduction of iron and insoluble matter (mostly silica) below specification levels. This was attempted by a series of cleaning flotation steps. Control analyses of iron and insoluble were made at regular intervals on the cleaned concentrate. These analyses and the details of the procedure are shown in Table 20. The test was made on a 150 g sample cut from the leached concentrates from the Central Zone ore.

TABLE 20

Procedure and Control Analyses for Production of Lubricant-Grade Molybdenite

Operation	Time min	Reagents	Grams	pH
Conditioning	3	Lime	0.20	7.1
1st cleaning	4	Sodium cyanide	0.10	10.1
		Sodium silicate	0.05	
		Kerosene	0.03	
		Pine oil-Dowfroth 250	0.02	
2nd cleaning	4	Sodium cyanide	0.05	10.2
		Sodium silicate	0.02	
Analyses of concentrate: 0.12% Fe and 0.78% Insol				
Regrinding	10			
3rd cleaning	3	Sodium cyanide	0.05	10.0
		Sodium silicate	0.02	
		Kerosene	0.02	
		Pine oil-Dowfroth 250	0.01	
4th cleaning	3	Sodium cyanide	0.03	10.3
		Sodium silicate	0.01	
5th cleaning	3	Kerosene	0.01	10.1
		Pine oil-Dowfroth 250	0.01	
Analyses of concentrate: 0.02% Fe and 0.31% Insol				
Regrinding	10			
6th cleaning	3	Sodium cyanide	0.03	10.2
		Sodium silicate	0.01	
		Kerosene	0.01	
		Pine oil-Dowfroth 250	0.01	
7th cleaning	3	Sodium cyanide	0.02	10.4
		Sodium silicate	0.01	
8th cleaning	3	Kerosene	0.005	10.1
		Pine oil-Dowfroth 250	0.005	
Analyses of concentrate: 0.02% Fe and 0.15% Insol				

*From Internal Reports MS-AC-987, 1031.

The final cleaned concentrate met lubricant-grade specifications for all the mineral analyses made with the exception of silica (British Ministry Supply). This might be reduced by hydrofluoric acid treatment. The combined cleaner tailings (31% by weight of the original sample) constituted satisfactory commercial grade molybdenite as shown below.

TABLE 21

Analyses* of Flotation Concentrate and Tailing

Element	Cleaned Concentrate	Combined Tailing
MoS_2 (%)	99.5	91.9
Mo	59.7	-
S	40.1	-
Insol	0.15	5.9
SiO_2	0.12	-
Fe	0.018	1.9
Cu	0.001	003

*From Internal Report MS-AC-67-81.

SUMMARY and CONCLUSIONS

The three lots of ore received for investigation contained mineral values as follows:

Sample	Analysis		
	MoS_2 (%)	Bi (%)	Ag (oz/ton)
Central Zone (25 tons)	0.46	0.028	0.03
West Zone "A" (66 tons)	0.13	0.048	0.04
West Zone "B" (614 lb)	0.13	0.25	0.66

Molybdenite occurs in these samples as masses and bundles of flakes. It is relatively free of impurities, with the exception of some molybdenite flakes from the West Zone "A" ore, which contain inclusions of magnetite.

Bismuthinite, the principal bismuth-bearing mineral, is present mostly as free grains. Native bismuth is the second most abundant bismuth mineral. It occurs partly as inclusions in bismuthinite and partly intergrown with bismite, bismutite and arsenobismite.

Ores from the Central Zone and from the West Zone "A" were treated on a pilot plant scale, while the ore from West Zone "B" was tested on a laboratory scale. Bulk flotation tests, made on the three ores separately and in combination were successful in recovering the value constituents in these ores and showed that:

1. No significant improvement of either grade or recovery was achieved when increasing the fineness of grinding from -35 mesh to -48 mesh;
2. Long flotation time was necessary for maximum recovery of molybdenite and bismuth;
3. Supplementing Z-6 promoter with some Aerofloat 33 increased bismuth recovery with little reduction in grade;
4. Sodium silicate was beneficial for reducing gangue in the concentrate.

Analyses of typical concentrates and recoveries achieved by bulk flotation are shown below:

Sample	Grade			Recovery (%)		
	MoS_2 (%)	Bi (%)	Ag (oz/ton)	MoS_2	Bi	Ag
Central Zone	89.9	3.7	6.0	97.4	72.3	75.0
West Zone "A"	63.5	14.2	16.8	90.1	75.3	64.0
West Zone "B"	23.6	46.8	127.0	89.6	88.8	97.2
Central+West "A"	88.8	5.8	8.4	95.0	66.3	74.0
Central+West "B"	55.5	24.1	50.4	89.8	83.4	90.8

Mineralogical studies on representative samples of flotation tailing indicated that most of their bismuth content was in the form of bismite, bismutite and arsenobismite. As these secondary minerals are particularly resistant to flotation, it was inferred that near-maximum recovery of native bismuth and bismuthinite was achieved in the investigation.

To obtain molybdenite of commercial grade, representative samples were cut from the bulk concentrates and leached for 3 hrs in hydrochloric acid. For the final 30 minutes, nitric acid was added to the pulp in order to eliminate more impurities and to obtain a higher extraction of bismuth and silver. In plant practice, these two metals would be precipitate and recovered in subsequent refining process.

The molybdenite residual concentrates after leaching were analysed with the following results:

Sample	MoS_2 (%)	Bi (%)	Cu (%)	Fe (%)	Insol (%)	Ag (oz/ton)
Central Zone	97.6	0.05	0.01	0.35	1.94	0.07
West Zone "A"	90.7	0.06	0.08	0.48	8.01	0.41
West Zone "B"	81.6	0.13	0.19	1.91	12.34	56.10
Central+West "A"	94.8	0.04	0.01	0.36	4.25	0.36
Central+West "B"	94.0	0.02	0.02	0.21	3.71	5.18

The failure of the leached concentrate from the West Zone "B" to meet commercial grade specification (90% MoS_2) was due to the high bismuth content (46.8%) of the bulk concentrate. Therefore, on removal of the leached material, the original 4.3% "insoluble" was increased to 12.3% in the resulting molybdenite concentrate. Since further depression of gangue in the bulk flotation would risk loss of molybdenite and bismuth, other techniques were tried to improve the grade of this final molybdenite concentrate. These consisted of (1) reflotation of the molybdenite after acid leaching and (2) selective flotation of molybdenite from the bulk concentrate prior to acid leaching. Both sodium sulphide and potassium dichromate were found effective in maintaining the bismuth minerals depressed when floating the molybdenite. For instance, a test using potassium dichromate gave a molybdenite concentrate concentrate assaying 84.8% MoS_2 and 5.2% Bi, and a bismuth concentrate (flotation tailing) containing 60.7% Bi and only 2.8% MoS_2. The bismuth removal from this molybdenite concentrate was complete by acid leaching.

A comparison of the final molybdenite concentrates produced by the two techniques is shown below:

Technique	MoS_2 (%)	Bi (%)	Cu (%)	Fe (%)	Insol (%)	Ag (oz/ton)
MoS_2 reflotation (after leaching)	93.0	0.12	0.20	0.19	2.34	66.1
MoS_2 separation (followed by leaching)	96.5	0.02	0.02	0.14	1.31	0.15

One disadvantage of the first technique is the high silver content in the molybdenite concentrate. Although it might be possible to extract this silver by cyanidation, no testing could be done to confirm this because of the small amount of material available.

Because the leached concentrate from the Central Zone was of near-lubricant grade, further testing was done in an attempt to meet these specifications. A sample was ground and cleaned several times by flotation using sodium cyanide and silicate for depression of iron and gangue minerals.

The floated concentrate analysed 99.5% MoS_2, 0.02% Fe, 0.001% Cu, 0.15% Insol and 0.12% SiO_2 (i.e. met the U.S. Specification MIL-L-7866 (Aer.) and the ROCOL Powder Specification for all the mineral analyses). The molybdenite residue from this test (31% by weight) was still of commercial grade. Although this work suggested that molybdenite of lubricant-grade can be produced from a leached concentrate by physical means, the low-impurity requirements of the specifications make it advisable to investigate the technique in plant practice if consideration is given to marketing such a product. Some difficulties may well arise when trying to attain 99% MoS_2 in large scale operation, but the premium price for such a product should be worth the effort.

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