

## CANADA

## DEPARTMENT OF ENERGY, MINES AND RESOURCES

## OTTAWA

## MINES BRANCH INVESTIGATION REPORT IR 66-83

# INVESTIGATION OF A GOLD-COBALT-BISMUTH ORE FROM THE MARIAN RIVER AREA FOR PRECAMBRIAN MINING SERVICES LIMITED, YELLOWKNIFE, N. W. T.

by

## G. I. MATHIEU

## MINERAL PROCESSING DIVISION

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#### SUMMARY OF RESULTS

Head analysis of the Marian River ore gave the following results:

<u>AU</u> <u>Co</u> <u>Bi</u> <u>As</u> <u>Fe</u> <u>S</u> 0.14 oz/ton 2.36% 0.63% 40.8% 22.5% 16.0%

The important mineralogical characteristics of the ore are as follows:

1. Gold occurs as minute inclusions in arsenopyrite;

- 2. Cobalt is present as a constituent of arsenopyrite, probably in solid solution;
- 3. Bismuthinite (the only bismuth-bearing mineral identified) appears partly as free grains and partly intergrown with arsenopyrite.

\* Research Scientist, Mineral Processing Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada. Therefore, very little concentration of gold and cobalt was possible, although some concentration of bismuth was achieved at low recovery. Results of typical tests on the flotation concentration of these metals follows:

Product	Bi(%)	Analysis <u>Co(%)</u>	Au(oz/ton)	Distr <u>Bi</u>	ibutio <u>Co</u>	n(%) Au
Bi conc Co conc Au conc	19.30	2.76	0.18	52.1	92.0	92.5

Cyanidation of the gold concentrate and hydrochloric acid leaching of the bismuth concentrate resulted in extraction of 81.9% of the gold, 94.5% of the bismuth.

A test simulating a flowsheet, which included bulk flotation, bismuth separation and gold cyanidation gave the following results:

Product	<u>Bi(%)</u>	Analysis <u>Co(%)</u>	Au(oz/ton)	Distr <u>Bi</u>	ibutio <u>Co</u>	n(%) <u>Au</u>
Bi conc Co conc Au sol'n	19.51 0.34 Tr	1.50 2.67 Tr	0.19 0.035 0.145	49.0 42.6	1.0 92.0	1.9 17.5 72.6

In plant practice, a small increase in bismuth recovery might be expected with recirculation of intermediate products.

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#### INTRODUCTION

#### Location of Property

The ore submitted for investigation originated from Marian River area, about 100 miles northwest of Yellowknife, N.W.T. Two ore zones have been defined at the property. Lach of these outcrops for about 750 feet and consists of bands of massive and disseminated sulphides. No diamond drilling had been done on the property.

#### Shipment and Instructions

On August 18, 1965, two bags of ore, each weighing about 62 pounds, were received from Mr. N. W. Byrne, President, Precambrian Mining Services Limited, Box 190, Yellowknife, N.W.T. Mr. Byrne requested that the results of the investigation be sent to Dr. F. B. Brien, P.Eng., 6558 - 43rd Avenue N.E., Seattle 15, Washington, U.S.A.

#### Purpose of Investigation

Although the initial objective was to determine if a 10-15% cobalt concentrate could be produced from the Marian River ore, this was precluded by the occurence of this metal in the arsenopyrite lattice. However, when analysis of the ore showed that gold and bismuth occured in economic grade, the investigation was directed towards the recovery of these metals.

#### Sampling and Analysis

The ore from the two bags was mixed thoroughly and crushed to minus 10 mesh. A representative sample was riffled out for chemical analysis and mineralogical examination.

#### TABLE 1

#### Chemical Analysis\* of Head Sample

Gold (Au)	- 0.14 oz/ton
Silver (Ag)	- 0.08 " "
Cobalt (Co)	- 2.36 %
Bismuth (Bi)	- 0.63 "
Arsenic (As)	-40.84 "
Iron (Fe)	-22.46 "
Sulphur (S)	-16.04 "
Insoluble	- 4.15 "

\* From Internal Report MS-AC-66-247

A spectrographic analysis on a portion of the head sample indicated the following elements listed in their approximate order of decreasing abundance:

#### TABLE 2

<u>Spectrographic Analysis\* of Head Sample</u>

I - As, Fe (>10%)
II - Si, Co, Ca (10 - 1%)
III - Ti, Ni, Al, Cu, Mg (1.0 - 0.1%)
IV - Cr, Na, Mn, V, Mo ( <0.1%)</pre>

\* From Internal Report MS-AC-65-1102

#### Mineralogical Examination\*

A portion of the head sample was sent to the Mineralogy Section of the Mineral Sciences Division for examination.

About 100 grams were screened into -35+65, -65+150 and -150+270 mesh fractions. Each fraction was then separated into sink and float sub-fractions by means of a heavy liquid having a specific gravity of 3.30. One polished section was prepared from a representative portion of each of the sink sub-fractions and examined under the ore microscope, while the float sub-fractions were combined and run on the X-ray diffractometer to determine the principal gangue constituents. The remaining portions of the sink sub-fractions were combined and the arsenopyrite concentrated by means of a Franz Isodynamic Separator. A portion of the arsenopyrite concentrate was submitted to the Analytical Chemistry Subdivision for cobalt analysis, and the remainder was made into a polished section. The minerals in the polished sections were identified by microscopical and X-ray diffraction methods.

The minerals found in the polished sections of the sink sub-fractions and their approximate abundance are as follows:

Major mineral	••••	arsenopyrite.
Minor minerals	••••	hematite, bismuthinite.
Trace minerals	-	native silver, native gold, chalcopyrite, pyrite, pyrrhotite, ilmenite, magnetite, emplectite, anatase.

\* From Internal Reports MS 66-7 and -56.

As shown above the sink sub-fraction consists essentially of arsenopyrite. Some of the arsenopyrite grains contain inclusions of native gold, native silver, hematite, bismuthinite and pyrrhotite, while a few others are intergrown with either hematite or bismuthinite. The inclusions vary from about 3 to 65 microns in size.

A chemical analysis of arsenopyrite concentrated from the sink sub-fractions reported 5.75% cobalt. A careful microscopic examination of the polished section of the concentrate showed that it consisted of arsenopyrite, and contained only trace amounts of the other minerals in the sample. Since no cobalt minerals could be found in the concentrate, it is apparent that the cobalt is present as a constituent of arsenopyrite probably in solid solution.

Bismuthinite and hematite were found in the sub-fractions, partly intergrown with arsenopyrite. The native gold and silver. in the sink fractions occurs as minute inclusions in arsenopyrite. These vary in size from 2 to 20 microns. The chalcopyrite and pyrite occur mainly as free grains. As mentioned previously, a few combined grains of bismuthinite and chalcopyrite were also found. The magnetite, emplectite and anatase are present as free grains, while one grain of pyrrhotite is intergrown with arsenopyrite.

The principal minerals in the float sub-fraction were identified as quartz, mica, scorodite and chlorite.

#### OUTLINE OF INVESTIGATION

Because of the high arsenopyrite content in the ore (about 80%) and the intimate association of gold with that mineral, only two techniques appeared practical for recovering gold: straight cyanidation of the raw ore, and flotation of the gold bearing arsenopyrite followed by cyanidation of the concentrate. Both methods were tested.

Since the cobalt in the ore is present as a constituent of arsenopyrite, the investigation to concentrate this metal was limited to one rougher flotation test. Further concentration by chemical means (such as roasting, leaching and precipitation) should be possible, but such a study was not within the scope of the present work.

Bismuth, because of its greater economic value, was investigated more thoroughly than cobalt. Both selective flotation and bulk flotation followed by separation stages were tried to concentrate the oismuth values. A preliminary extraction test was made to assess the feasibility of recovering the bismuth from a flotation concentrate by acid leaching. In a final test, attempts were made to simulate a flowsheet which included separate concentration of the bismuth and cobalt, and extraction of the gold by cyanidation.

## DETAILS OF INVESTIGATION

## Recovery of Gold

## i) Straight Cyanidation, Tests 1 - 9

Lots of 1000 g of ore were ground for 20, 30, and 40 min and cyanided in a solution maintained at 1.0 lb of NaCN/ton and 0.5 lb of CaO/ton. The contact time varied from 24 to 72 hrs. Results of these tests are shown below.

#### TABLE 3

Test	Fineness %-200m	Time hrs	Reagents C lb/ton c NaCN		Residue Assay* oz Au/ton	Extraction** % Au
1	69.5	24	2.6	27.5	0.040	73.3
2		48	3.5	33.1	0.040	73.3
3		72	3.8	39.3	0.040	73.3
4	87.1	24	2.8	25.5	0.040	73.3
5		48	3.6	33.0	0.035	76.7
6		72	3.9	40.2	0.035	76.7
7	98.0	24	3.6	25.4	0.040	73.3
8		48	4.9	34.1	0.035	76.7
9		72	5.8	40.3	0.035	76.7

## Straight Cyanidation of the Ore

\* From Internal Report MS-AC-66-509. \*\* Calculated by difference.

## ii) Flotation and Cyanidation, Test 10

 $_{\rm A}$  2000 g sample of ore was ground for 30 min and floated using the procedure described in Table 4. The concentrate produced was cyanided for 48 hrs at solution strengths of 1.0 lb of NaCN/ton and 0.5 lb of CaO/ton. Flotation and cyanidation results are shown in Tables 5 and 6 respectively.

#### TABLE 4

#### Reagents and Conditions for Gold Flotation

Operation	Time min	Reagents	lb/ton	рН
Conditioning	5	Soda ash Reagent 301 Reagent 208 Dowfroth 250	1.25 0.10 0.05 0.02	7.4
Rougher flotation	20	Reagent 301 Reagent 208 Dowfroth 250	0.05 0.03 0.02	7.2

#### TABLE 5.

## Results of Gold Flotation

Product	Weight %	Analysis*, oz/ton Au	Distribution, %
Flot concentrate Flot tailing	82.8 17.2	0.18 0.07	92.5 7.5
Feed (calcd)	100.0	0.16	100.0

\* From Internal Report 66-456.

#### TABLE 6

#### Cyanidation of Gold Flotation Concentrate

Reagents Consumed lb/ton of ore		Residue Assay* oz/ton	Extrac	ction**, %
NaCN	CaO	лu	In feed	Overall
2.1	15.7	0.035	81.6	75.5

\* From Internal Report MS-AC-66-717 \*\* Calculated by difference.

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## Concentration of Cobalt

## Test 11

A 2000 g sample of ore was ground for 20 min and floated using the procedure shown below.

## TABLE 7

## Reagents and Conditions for Cobalt Flotation

Operation	Time min	Reagents	lb/ton	pH.
Conditioning	5	Soda ash Xanthate Z - 6 Aerofloat 33 Dowfroth 250	1.25 0.06 0.03 0.01	7.2
Rougher flotation No. 1	12	Xanthate Z - 6 Dowfroth 250	0.04 0.01	7.1
Rougher flotation No. 2	8	Xanthate Z - 6 Dowfroth 250	0.04 0.01	7.0

## TABLE 8

## Results of Cobalt Flotation

Product	Weight	Analysis*, %	Distribution, %
	%	Co	Co
Flot conc No. 1	55.4	2.79	63.0
Flot conc No. 2	26.5	2.69	29.0
Flot tailing	18.1	1.08	8.0
Feed (calcd)	100.0	2.45	100.0

\*From Internal Reports MS-AC-66-348 and 796.

The low ratio of concentration necessary for a nearcomplete cobalt recovery confirmed the association of this metal with arsenopyrite, the major constituent of the ore. Recovery of sigmuth

## i) Concentration by Flotation, Tests 12-13

Initially, attempts were made to concentrate the bismuth by selective flotation using sodium cyanide to depress arsenopyrite (Test 12). Next, a bulk flotation concentrate was produced from which the bismuth was refloated while depressing arsenopyrite with the cyanide (Test 13).

Both tests were done on 6000 g samples of ore ground in stages to minus 48 mesh. Flotation procedures and results obtained are shown in Tables 9 and 10 respectively.

#### TABLE 9

## Reagents and Conditions for Bismuth Flotation

Technique	Operation	Time	Reagents	lb/ton	pН
Selective	Conditioning	5	Soda ash Sodium cyanide Xanthate Z - 6 Dowfroth 250	1.25 0.10 0.10 0.02	ຮີ.5
Flotation (Test 12)	Rougher flotation	15	Xanthate Z - 6 Dowfroth 250	0.05 0.02	8.4
(1620 16)	lst + 2nd cl "	5	Sodium cyanide Xanthate Z - 6	0.03 0.01	8.7
	3rd + 4th cl "	4	Sodium cyanide	0.01	8.8
Bulk	Conditioning	5	Soda ash Xanthate Z - 6 Dowfroth 250	1.25 0.10 0.02	7.3
	Bulk flotation	15	Xanthate Z - 6 Dowfroth 250	0.05 0.02	7.2
Flotation- Separation	Scavenger "	· 5	Xanthate Z - 6 Dowfroth 250	0.05 0.02	7.0
(Test 13)	Separation "	5	Sodium cyanide Xanthate Z - ó	0.10 0.02	8.2
	lst + 2nd cl "	5	Sodium cyanide Xanthate Z - 6	0.03 0.01	8.5
	3rd + 4th cl "	4	Sodium cyanide	0.01	8.7

# TABLE 10

Results of Bismuth Flotation

Test	Product	Weight	À	nalys	sis	D	istribu	ition
		%	9/0 -		oz/ton		%	
			Bi	Co	Au .	Bi	Co	Au
12 (Selective Flotation)	Bi concentrate 3rd + 4th cl tailing 1st + 2nd cl tailing Flotation tailing	0.8 1.4 4.0 93.8	31.20 2.05 1.65 0.37	2.22 2.15	0.30	36.1 4.2 9.5 50.2		1.1 3.5 8.0 87.4
	Feed (calcd)	100.0	0.69	2.31	0.15	100.0	100.0	100.0
13 (Bulk Flotation- Separation)	Bi concentrate 3rd + 4th cl tailing 1st + 2nd cl tailing Separation tailing Scav concentrate Flotation tailing	1.8 1.7 6.4 41.6 28.5 20.0	19.30 1.60 0.48 0.32 0.28 0.24	2.51 2.53 2.67 2.60	0.28 0.22 0.16	52.1 4.1 4.6 20.0 12.0 7.2	6.9 47.7 31.8	2.2 3.3 9.6 45.5 31.2 8.2
	Feed (calcd)	100.0	0.67	2.33	0.15	100.0	100.0	100.0
	Cl tail + sep tail + scav conc	78.2	0.35	2.63	0.17	40.7	88.2	89.6

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\* From Internal Reports MS-AC-421, 567, 710 and 805.

few additional tests were made using the bulk flotation-separation technique. These were made on ore samples ground to -35 and -65 mesh. The separation was tried successively with sodium cyanide, potassium permanganate and sodium hypochlorite. The results of these tests were inferior to those obtained in Test 12, particularly when using permanganate or hypochlorite.

## ii) Extraction by Acid Leaching, Test 14

The oismuth concentrate used in this test contained 18.2% Bi and was made up from a few assay rejects from previous tests. The procedure for extraction consisted of three stages which are described below:

(1) Mix 39 g of bismuth concentrate with 200 cc of hydrochloric acid (37% HCl), agitate for 1 hr at about 70°C, filter and analyse the pregnant solution for bismuth and iron after diluting to 500 cc;

(2) Repulp the residue with 100 cc of hydrochloric acid and treat as in (1);

(3) Repulp again with 50 cc of hydrochloric acid and 10 cc of nitric acid, agitate for 20 min at about 70°C, and analyse the residue for bismuth and the solution for bismuth and iron after diluting to 500 cc.

#### TABLE 11

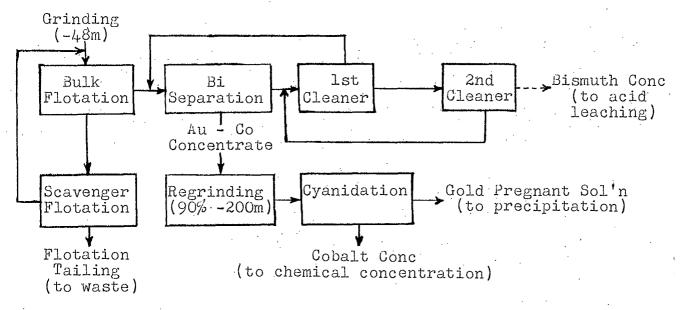
Sampling	Residue	Sol'n	Cumulative		
Point	Weight		Extraction		
After stage (1) After stage (2) After stage (3)	82.0 67.2 57.7	6.75 6.65 0.67	Fe 4.59 3.00 3.24	11.6 1.5 0.04	47.7 94.5 99.9

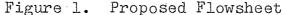
## Acid Leaching of Bismuth Concentrate

\* From Internal Report MS-AC-66-613.

#### Proposed Flowsheet

By incorporating the features found previously, a flowsheet was developed for the recovery of the gold, cobalt and bismuth from the Marian River ore. This flowsheet is illustrated by Figure 1.





Because the intermediate products are not recirculated in batch testing, it was not possible to duplicate exactly the above flowsheet in the laboratory. However, a test was made which simulated the procedure with the exception that the scavenger concentrate and the cleaner tailings were combined with the goldcobalt concentrate prior to regrinding and cyanidation.

Details of flotation and cyanidation were as in Tests 12 and 10 respectively. The results obtained are summarized below:

#### TABLE 12

Product	Weight	Analysis*			Distribution			
· ·	- %	0/0		oz/ton	90			
		Bi	Ço	Au	Bi	Co	Au	
Bi concentrate Au pregnant sol'n Co concentrate (cyanidation residue)	1.6 80.1	19.51 Tr 0.34	1.50 Tr 2.67	0.19 0.145** 0.035	49.0 42.6	<b>-</b> .	72.6	
Flot tailing	18.3	0.29	0.89	0.070	8.4	7.0	8.0	
Feed (calcd)	100.0	0.63	2.33	0.16	100.0	100.0	100.0	

Test Results with Proposed Flowsheet

From Internal Reports MS-AC-649, 796 and 943.
 \*\* Assay calculated on the basis of cyanidation feed.

Note: additional analyses on the bismuth concentrate gave the following results:

Arsenic		32.0%
Iron	-	21.1%
Sulphur	· _	22.2%
Insoluble		1.2%

To determine the cause of the rather low bismuth recovery, mineralogical studies were done on the cobalt concentrate and the flotation tailing by the Mineral Sciences Division. These consisted of microscopic examination of two polished sections from each product. The following points were established:

- 1. Most of the bismuthinite is present as inclusions in arsenopyrite.
- 2. A few grains of bismuthinite are combined with chalcopyrite.
- 3. Less than 25% of the bismuthinite occurs as free grains ranging from 6 to 130 microns.

\* From Internal Report MS-66-56.

#### CONCLUSIONS

To obtain more information about the valuable constituents of the ore, namely, gold (0.14 oz/ton), cobalt (2.36%) and bismuth (0.63%), each of these was first studied independently of the others. This part of the investigation established the following points:

- 1. Because gold is intergrown with arsenopyrite, very little concentration of this metal was possible. However, cyanidation tests showed that about 75% of the gold could be extracted either from the raw ore or from a bulk concentrate.
- 2. The occurence of cobalt in the arsenopyrite lattice limited its concentration from 2.3% to about 2.8% Co. Considerable research might be necessary to develop a chemical process for economically recovering the cobalt from such a low grade concentrate.
- 3. Partial association of bismuthinite with arsenopyrite held the bismuth recovery to about 50%. With the best procedure found to recover the bismuthinite (i.e. bulk flotation followed by separation), it was not possible to obtain a concentrate grade higher than 19.5% Bi, probably because some of the arsenopyrite remained activated despite the addition of depressant.

Although further hydrometallurgical research on cobalt and bismuth extraction may influence the final process, a test was made on a preliminary flowsheet incorporating the best practices indicated by this investigation. The procedure consisted of floating a bulk concentrate from which the bismuth was separated by flotation and the gold extracted by cyanidation. The residue from cyanidation formed a low grade cobalt concentrate. The test produced a 19.5% grade bismuth concentrate with a 49% recovery, a 2.7% grade cobalt concentrate with a 92% recovery and a pregnant solution containing 73% of the gold in the ore.

Economic or other considerations might require modifications of the proposed flowsheet. In this event, it is suggested that the following points should be taken into account:

- 1. Roasting prior to cobalt extraction should be made only after bismuthinite removal because of the tendency of this mineral to volatilize.
- 2. Cyanidation of the gold should be conducted after bismuthinite flotation because a long contact of this mineral with high concentration of cyanide and lime might affect its subsequent flotation.

Finally, it should be noted that the bismuth-arsenopyrite separation using low concentration of sodium cyanide to depress the arsenopyrite is feasible only when the bismuth occurs as bismuthinite. Native bismuth would also have been depressed by the above reagent.

#### ACKNOWLEDGEMENTS

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