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Pilot Mill Flotation of Anorthositic Platinum-Palladium Ore From the Stillwater Complex

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

diam	diameter	mi	mile
ft	foot	min	minute
g	gram	μm	micrometer
gal	gallon	oz	troy ounce
hr	hour	pct	percent
kwhr	kilowatt-hour	pH	negative logarithm of the effective hydrogen ion concentration
L	liter		
lb	pound	ppm	part per million

PILOT MILL FLOTATION OF ANORTHOSITIC PLATINUM-PALLADIUM ORE FROM THE STILLWATER COMPLEX

By E. Morrice[1]

ABSTRACT

The Bureau of Mines investigated methods for beneficiating platinum-group metal ores from the Stillwater Complex, Montana, as part of its program to increase the supply of critical and strategic minerals and metals from domestic resources. This report presents results of a 5-day continuous campaign employing a pilot flotation mill to recover sulfide concentrate containing platinum-palladium values from anorthositic ore. A mercaptobenzothiazole-sulfuric acid reagent suite was used. The pilot flotation mill was operated at an average feed rate of 106 lb/hr of ore. A rougher concentrate containing 8.5 pct of the feed was prepared; it assayed 1.1 oz/ton Pt and 3.5 oz/ton Pd. Recovery of platinum and palladium were 91 and 87 pct, respectively.

[1]Metallurgist, Reno Research Center, Bureau of Mines, Reno, Nev.

INTRODUCTION

The Bureau of Mines has investigated recovery methods for platinum-group metals (PGM) from domestic resources since 1918.[2] A significant PGM deposit occurs within the lower part of the Banded Zone of the Stillwater Complex, Montana. In 1967 an exploration program initiated by Johns-Manville Corp. resulted in delineation of a favorable horizon in the complex that was essentially continuous along a strike length of 24 mi.[3] The horizon exposed in the Johns-Manville West Fork Adit has an estimated grade of 0.47 oz/ton Pt-Pd and a Pd-Pt ratio of 3.5. The mineralization occurs in sulfide-bearing anorthositic rocks. Bureau of Mines[4] bench-scale studies on the flotation of West Fork Adit ore have been reported. Since 1977, the Anaconda Minerals Co. has been exploring a PGM deposit that occurs east of the West Fork Adit in a sheared and serpentinized portion of the Banded Zone.[5]

This report presents the results of a pilot mill study employing a mercaptobenzothiazole (AERO 404)[6]-sulfuric acid reagent suite in the flotation of ore from an anorthositic rock unit in Anaconda's Minneapolis Adit.

ACKNOWLEDGMENT

The author acknowledges assistance from Anaconda Minerals Co. (Atlantic Richfield Corp.) for providing the ore sample and geological information on the deposit.

SAMPLE DESCRIPTION

A 20-ton bulk sample of minus 2-in mine-run ore from the Minneapolis Adit was obtained for pilot mill testing. Analysis of the sample showed Pt, 0.10 oz/ton; Pd, 0.34 oz/ton; Au, 0.007 oz/ton; Cu, 0.03 pct; and Ni, 0.06 pct.

The material was fresh anorthositic gabbro and contained serpentinized and sericitized rocks. The unaltered material exhibited a typical gabbroic texture of anhedral calcic plagioclase and varying amounts of anhedral to subhedral pyroxene. The altered material ranged from fresh-appearing rock, which showed minor replacement by epidote-clinozoisite, to rock showing extensive replacement by epidote-clinozoisite, sericite, kaolinite, calcite, chlorite, and serpentine. In comparison, the West Fork Adit ore was relatively unaltered anorthositic gabbro that contained calcic plagioclase, clinopyroxene, orthopyroxene, olivine, and minor amounts of serpentine. In both the Minneapolis Adit and West Fork Adit ores, accessory minerals included pyrrhotite, pentlandite, pyrite, chalcopyrite, and magnetite. The PGM mineralization was associated with the sulfides, which made up about 1 pct of the rock.

[2]Jolly, James H. Platinum-Group Metals. BuMines Mineral Commodity Profiles, 1978, 23 pp.

[3]Conn, H. K. The Johns-Manville Platinum-Palladium Prospect, Stillwater Complex, Montana, U.S.A. Can. Miner., v. 17, 1979, pp. 463-468.

[4]Bennetts, J., E. Morrice, and M. M. Wong. Preparation of Platinum-Palladium Flotation Concentrate From Stillwater Complex Ore. BuMines RI 8500, 1981, 18 pp.

[5]Bow, C., D. Wolfgram, A. Turner, S. Barnes, J. Evans, M. Zdopski, and A. Boudreau. Investigations of the Howland Reef of the Stillwater Complex, Minneapolis Adit Area. Stratigraphy, Structure, and Mineralization. Econ. Geol., v. 77, 1982, pp. 1481-1492.

[6]Reference to specific brands is made for identification only and does not imply endorsement by the Bureau of Mines.

In both the Minneapolis Adit and the West Fork Adit, the sulfide minerals were disseminated in the rock, but a few occurrences of coarse-grained sulfides were observed. Microscopic examination of screened fractions of crushed ore showed that most of the sulfides could be liberated at 200 mesh. The Bond Index[7] for grinding the ore to minus 200 mesh was 15 kwhr/ton. Specific gravity of the ore was 2.7.

BENCH-SCALE FLOTATION TESTS

Flotation tests were made on the Minneapolis Adit ore to determine its amenability to the AERO 404-sulfuric acid flotation scheme previously developed on a bench-scale for West Fork Adit ore.[8] Samples for flotation were prepared by stage crushing and dry grinding the ore, as described in the previous bench-scale tests. Flotation tests were conducted in Denver No. 12 laboratory flotation cells having stainless steel impellers, stators, and tanks.

Tests were made using 1,600-g charges of ore to determine the amounts of sulfuric acid, AERO 404, and polypropylene glycol methyl ether (Dowfroth 250) required for best rougher flotation. The ore was dry ground in stages to 100 pct minus 200 mesh (55 pct minus 325 mesh). The test conditions were solids concentration in slurry, 36 pct; conditioning time, 10 min; and flotation time, 10 min. These conditions were the best for flotation of West Fork Adit anorthositic ore.

The best reagent suite for floating Minneapolis Adit ore was, per ton of ore, 28 lb sulfuric acid, 0.3 lb AERO 404, and 0.01 lb Dowfroth 250. The amount of sulfuric acid added was necessary because of the acid-consuming minerals--calcite and serpentine. In previous bench-scale flotation of West Fork Adit ore sulfuric acid consumption was 22 lb/ton of ore.[9] Flotation was performed at a slurry pH of 4.0. Metallurgical results are given in table 1.

[7]Bond, Fred C. The Third Theory of Comminution. Trans. AIME, Mining Branch, v. 193, May 1952, pp. 484-494.

[8]Work cited in footnote 4.

[9]Work cited in footnote 4.

TABLE 1. - Bench-scale flotation

	Concentrate	Tailing	Composite
Weight....pct..	9	91	100
Analysis:			
Platinum....oz/ton..	1.0	0.01	0.10
Palladium....oz/ton..	2.7	0.065	0.30
Gold....oz/ton..	0.05	0.002	0.01
Copper....pct..	0.1	0.01	0.02
Nickel....pct..	0.2	0.04	0.05
Distribution, pct:			
Platinum....	91	9	100
Palladium....	80	20	100
Gold....	71	29	100
Copper....	50	50	100
Nickel....	33	67	100

PILOT MILL OPERATION FOR A 5-DAY CAMPAIGN

Figure 1 is a flowsheet of the unit operations used in the pilot mill. A description of the equipment used is given in table 2.

The 20-ton "as received" sample was minus 2 in and was crushed dry to minus 10 mesh with a jaw crusher, cone crusher, and rolls. The rolls were operated in closed circuit with a vibrating screen. The minus 10-mesh ore was stored in 55-gal steel drums, and each drum was sampled with a pipe sampler before being transferred to the ore bin. During the 5-day campaign, an average of 106 lb/hr of ore was fed by a short-belt conveyor from the ore bin into the tube mill. The tube mill contained a 730-lb charge of steel balls ranging from 3/4 to 1-1/8 in and was operated at 75 pct of critical speed. Table 3 shows the screen analyses of the feed, mill discharge, and classifier underflow and overflow.

TABLE 2. - Equipment used in the pilot mill

Item	Quantity	Dimensions	Description
Ore bin.................	1	21-ft^3 capacity.........	Mild steel.
Tube mill...............	1	16-in diam × 48 in long.	Denver.
Classifier..............	1	6-in diam × 60 in long, spiral.	Do.
Thickeners..............	3	3-ft diam × 3 ft high...	Mild steel.
Conditioning tank.......	1	14-in diam × 25 in high.	Polyvinyl chloride.
Flotation cell..........	[1]6	0.4 ft^3 capacity per cell.	Denver, "Sub A," No. 5.
Settling tanks..........	2	3-ft diam × 3 ft high...	Mild steel.
Water distribution tank.	1	.....do.................	Do.
Sulfuric acid feeder....	1	.....do.................	Clarkson Model E, polyvinyl chloride.
AERO 404 and Dowfroth feeders.	2	.....do.................	Clarkson Model E, stainless steel.

[1]In 1 bank.

TABLE 3. - Average screen analyses of grinding circuit products

Size fractions, mesh[1]	Distribution, pct			
	Tube mill		Classifier	
	Feed	Discharge	Underflow	Overflow
10 by 28...............	47.4	0.0	0.0	0.0
28 by 35...............	9.2	.4	1.6	.0
35 by 65...............	16.4	1.1	21.0	.0
65 by 100..............	4.9	3.2	47.1	.6
100 by 150..............	5.3	5.8	19.4	4.9
150 by 200..............	4.2	17.2	4.4	16.6
200 by 325..............	3.7	18.4	1.5	23.1
Minus 325...............	8.9	53.9	5.0	54.8
Composite..........	100.0	100.0	100.0	100.0

[1]Tyler standard screen sieves.

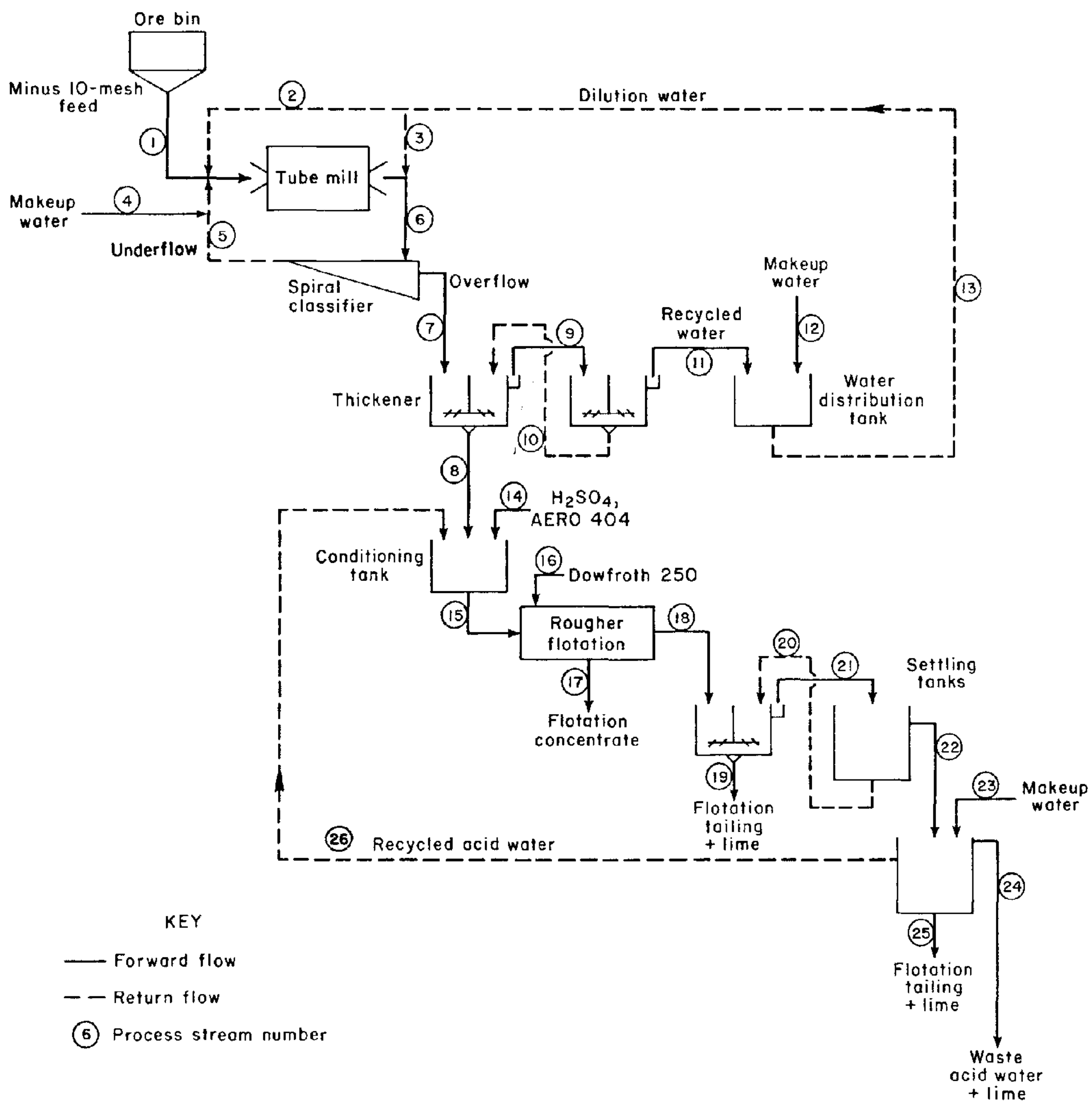

FIGURE 1. - Pilot mill flowsheet.

Discharge point samples were taken at hourly invervals during the 5-day campaign so that the solids concentrations could be adjusted to the desired levels. The feed from the ore bin contained 11 pct moisture. The solids content of the grinding circuit products are given in table 4. A solids content of 54 pct was maintained in the tube mill. The tube mill discharge (72.3-pct minus 200 mesh) was diluted with recycled water from the thickeners and classified. Classifier overflow (77.9-pct minus 200 mesh) was pumped into the primary thickener at 21 pct solids and was discharged at 58 pct solids. A high solids concentration in the thickener discharge facilitated the addition of recycled acid water to the conditioning tank in order to obtain the desired solids concentration of 36 pct for flotation. Bench-scale studies on West Fork Adit ore indicated that a solids concentration of 30 to 40 pct was best for platinum-palladium recovery.

TABLE 4. - Average solids content of grinding circuit products

Sample	Solids concentration, pct
Tube mill discharge...	54
Classifier:	
Feed................	23
Overflow............	21
Underflow...........	62
Primary thickener:	
Overflow............	3
Underflow...........	58
Conditioning tank discharge to flotation cells................	36

A mass balance showing solids and water contents of process streams during a representative shift of the 5-day campaign is given in table 5. Streams are identified by numbers in figure 1. The flow rates of the pilot mill streams were measured twice during every 8-hr shift.

TABLE 5. - Process stream flow rates for a representative shift

Stream	Flow, lb/hr		Stream	Flow, lb/hr	
	Solids	Water		Solids	Water
1....	106	13	14....	0	20
2....	<.1	63	15....	106	186
3....	<.1	354	16....	0	4
4....	0	10	17....	9	37
5....	1.5	1	18....	97	153
6....	107.5	441	19....	92	47
7....	106	440	20....	17	30
8....	106	71	21....	22	136
9....	11	374	22....	5	106
10....	11	5	23....	0	3
11....	<.1	369	24....	<.1	1
12....	0	48	25....	5	3
13....	<.1	417	26....	<.1	105

METALLURGICAL RESULTS

Each drum of ore was sampled and analyzed for Pt, Pd, and Au before being dumped into the ore bin. The froth from the flotation cells and the flotation tailing stream were sampled and analyzed for Pt, Pd, and Au twice during an 8-hr shift. Average metallurgical results obtained over 15 consecutive 8-hr shifts are shown in table 6. For the campaign, bulk concentrates averaging 8.5 pct of the feed weight, 1.1 oz Pt, 3.5 oz Pd, and 0.06 oz Au per ton with accompanying Pt, Pd, and Au recoveries of 91, 87, and 74 pct, respectively, were obtained. Reagent additions were 30 lb H_2SO_4, 0.4 lb AERO 404, and 0.01 lb Dowfroth 250 per ton of ore.

The pH of the flotation pulp was monitored with an Orion Research Model digital pH meter (fig. 2). Decreasing the pH in the flotation circuit from 4.0, which was used in bench-scale tests, to 3.7 improved Pt and Pd recovery. If the pH was permitted to increase above 4, very little material floated. Increasing the addition of AERO 404 from 0.3 to 0.4 lb/ton of ore resulted in higher Pt-Pd recovery. The dosage (0.01 lb/ton of ore) of Dowfroth 250 was the same as determined in the bench-scale tests.

Concentrates were periodically washed from the froth launder into a bucket and filtered on a Denver pressure filter before drying. During the 15th shift, 10-min samples of flotation froth were taken simultaneously from each of the six cells. Analytical results of the samples are shown in table 7. The higher Pt and Pd contents of the froths from cells 1 and 2 indicated that these froths are suitable for subsequent Pt-Pd extraction and should be removed from the circuit. The froth products from the last four cells should be upgraded in a cleaner flotation step.

FIGURE 2. - Measurement of flotation slurry pH.

TABLE 6. - Pilot mill flotation[1]

	Concentrate	Tailing	Composite
Solids................lb/hr..	9	97	106
Analysis:			
Platinum...........oz/ton..	1.1	0.01	0.11
Palladium..........oz/ton..	3.5	0.05	0.35
Gold...............oz/ton..	0.06	0.002	0.007
Copper................pct..	0.24	0.01	0.03
Nickel................pct..	0.35	0.03	0.06
Distribution, pct:			
Platinum....................	91	9	100
Palladium...................	87	13	100
Gold........................	74	26	100
Copper......................	69	31	100
Nickel......................	52	48	100

[1]Average values for 15 consecutive 8-hr shifts.

TABLE 7. - Froth products from the flotation cells

Cell	Percentage of total froth	Analysis, oz/ton	
		Pt	Pd
1.................	54	1.7	5.2
2.................	22	1.5	4.9
3.................	4	1.0	3.6
4.................	7	.6	2.9
5.................	4	.6	2.9
6.................	9	.3	1.0
Composite.....	100	1.4	4.4

EXAMINATION OF PRODUCTS

The bulk concentrate and tailing from the 5-day campaign were sampled, and detailed analyses are given in table 8. The concentrate was 93-pct minus 325 mesh.

Examination of a polished section of bulk concentrate showed that the sulfide minerals were pyrite, chalcopyrite, pentlandite, and bornite, and that the gangue minerals were pyroxene, plagioclase, epidote, and chlorite. The PGM-bearing grains included braggite, merenskyite, vysotskite, and nickel-bearing vysotskite. All of the PGM-bearing grains were liberated and were <10 µm in diameter. The presence of chlorite $[(Mg,Al,Fe)_{12}[Si,Al)_8O_{20}](OH)_{16}]$ probably accounts for the high MgO content

TABLE 8. - Analysis of concentrate and tailing

	Concentrate	Tailing
Analysis, oz/ton:		
Platinum........	1.1	0.01
Palladium.......	3.5	.05
Rhodium.........	.03	<.0001
Iridium.........	.01	<.0005
Gold............	.06	.002
Analysis, pct:		
Copper..........	.24	.01
Nickel..........	.35	.03
Iron............	5.7	3.5
Sulfide sulfur..	1.2	.2
Sulfate sulfur..	1.5	1.5
SiO_2............	44.6	43.9
MgO............	15.1	7.1
Al_2O_3...........	15.8	24.2
CaO............	8.4	14.1

< Indicates values below detection limit.

(15.1 pct) of the concentrate. The reaction of sulfuric acid and gangue minerals to produce insoluble sulfates would account for the presence of sulfate sulfur in the tailing and concentrate. The sulfate sulfur content of the ore was 0.1 pct.

Since the flotation concentrate would be subsequently treated to recover the PGM values and the tailing would be impounded in a tailing pond, impurity contents of both products are important. In table 9, the impurity contents of the bulk concentrate are compared with those of the bulk tailing. The data show that the impurity levels in both products are low.

The bulk tailing was screened and gave the following size distribution:

Plus 150 mesh............	10 pct
150 by 200 mesh..........	22 pct
200 by 325 mesh..........	22 pct
Minus 325 mesh...........	46 pct

No concentration of platinum or palladium was noted in any of the fractions.

Slurry discharged from the tailing thickener was treated with lime to increase the pH to 7.0. Bench-scale tests indicated that 5 lb CaO per/ton of thickened tailing (66 pct solids) was required to obtain the desired pH. Baker analytical reagent calcium oxide was used. Samples of water from the tailing thickener before and after adding lime were evaporated to dryness, and the residues were analyzed for impurities by an optical emission spectrographic procedure. Chromium, cobalt, copper, lead, zinc, and boron impurities were below detection limits in the untreated tailing water. The addition of lime resulted in a decrease in the levels of aluminum, manganese, and nickel impurities to below detection limits. By adding lime, iron impurity was decreased from 1 g/L to <0.2 g/L.

TABLE 9. - Impurity content of concentrate and tailing, ppm

Element	Analysis	
	Concentrate	Tailing
Antimony...........	99	<6
Arsenic............	110	<8
Beryllium..........	70	5.4
Bismuth, tellurium.	<20	<20
Boron, cadmium.....	<.4	<.4
Chromium...........	550	350
Cobalt.............	<.9	19
Columbium..........	<30	41
Lead, mercury, and tungsten..........	<6	<6
Manganese..........	600	400
Molybdenum.........	4	<1
Phosphorus.........	40	<20
Selenium...........	<120	<120
Strontium..........	70	120
Tin................	<2	7.3
Titanium...........	760	450
Uranium............	<50	<50
Vanadium...........	<5	<5
Zinc...............	290	29
Zirconium..........	<2	<2

< Indicates values below detection limit.

SUMMARY AND CONCLUSIONS

Pilot mill flotation research on a Stillwater Complex, Montana, anorthositic ore demonstrated that a mercaptobenzothiazole-sulfuric acid suite will give a sulfide concentrate containing 88 pct of the PGM values and analyzing 4.6 oz/tonPGM. Since the ore contained serpentinized and sericitized rocks with acid consuming minerals, sulfuric acid consumption was more than previously determined for anorthositic ore obtained from a different location. A more economic flotation technique for treating altered zone ores that contain excessive amounts of acid-consuming minerals needs to be developed. The acid circuit flotation system can be used with sulfide-bearing anorthositic ores.