Bureau of Mines Report of Investigations/1984

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UNITED STATES DEPARTMENT OF THE INTERIOR

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	lb	pound
g	gram	lb/ton	pound per ton
g/L	gram per liter	mL	milliliter
h	hour	min	minute
kg	kilogram	pct	percent
L	liter	ppm	part per million
L/min	liter per minute		

CALCIUM SULFIDE PRECIPITATION OF MERCURY DURING CYANIDE
LEACHING OF GOLD ORES

By R. G. Sandberg,[1] W. W. Simpson,[2] and W. L. Staker[2]

ABSTRACT

Many gold-bearing ores throughout the Western United States contain small quantities of mercury. The presence of mercury not only decreases the gold-loading capacity of the activated carbon, but complicates the fire refining of cathodes and creates a potential health hazard.

The Bureau of Mines investigated methods to limit mercury extraction during gold ore leaching and to remove mercury from cyanide leach solutions. Mercury dissolution was reduced from 40 to 10 pct by decreasing NaCN concentration from 20 lb to 0.34 lb per ton of solution at a pH of <11. When 0.1 lb CaS per ton of ore was added to a leach slurry, mercury extraction was reduced from 27 to 0.4 pct. Less than 0.5 pct of the mercury was solubilized when <0.1 lb CaS per ton of ore, along with lime and NaCN, was added to a simulated grinding circuit.
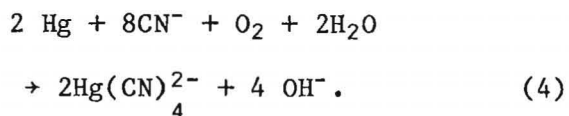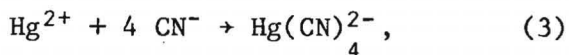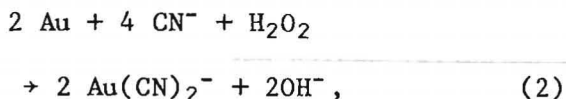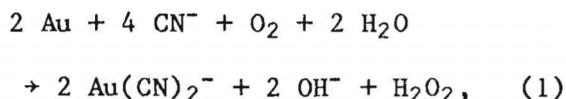
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INTRODUCTION

Numerous low-grade gold-silver ore deposits are being mined and milled throughout the Western United States (1-7).[3] In addition to gold and silver, many deposits contain as much as 15 ppm of mercury. During cyanidation, 10 to 30 pct of the mercury and 85 to 90 pct of the gold are typically solubilized. The reactions for gold and mercury extraction with cyanide are (8-9)--

$$2\ Au + 4\ CN^- + O_2 + 2\ H_2O$$

$$\rightarrow 2\ Au(CN)_2^- + 2\ OH^- + H_2O_2, \quad (1)$$

$$2\ Au + 4\ CN^- + H_2O_2$$

$$\rightarrow 2\ Au(CN)_2^- + 2OH^-, \quad (2)$$

$$Hg^{2+} + 4\ CN^- \rightarrow Hg(CN)_4^{2-}, \quad (3)$$

$$2\ Hg + 8CN^- + O_2 + 2H_2O$$

$$\rightarrow 2Hg(CN)_4^{2-} + 4\ OH^-. \quad (4)$$

Mercury builds up in the recycled leach solutions because only part of the mercury is adsorbed on carbon in the loading circuit. Mercury and gold are stripped from the carbon with caustic cyanide solution and electrowon on steel wool cathodes. Mercury must either be recovered or precipitated because of the health hazard during smelting of cathodes and regenerating of activated carbon. One gold mill recovers mercury by retorting the cathodes prior to smelting (10). Another mill autoclaves the ore to extract minimal mercury (6). Soluble mercury can be precipitated with sulfides, as shown by the chemical equation

$$Hg(CN)_4^{2-} + MS \rightarrow HgS + M^{2+} + 4CN^-. \quad (5)$$
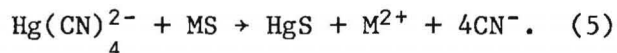
Silver, iron, and zinc sulfides have been used to precipitate mercury from gold cyanide solutions (8) but require slurry filtration before sulfide addition to reclaim excess reagents. Silver sulfide addition would tie up considerable silver, and silver sulfide excess would have to be recovered. Iron sulfide is undesirable because of cyanide loss due to ferrocyanide. Calcium and sodium sulfides, which were used in this investigation, are not harmful to the gold recovery process. The objective of this Bureau of Mines investigation was to study the effects of operating parameters on mercury extraction and examine methods for mercury removal from gold cyanide leach slurries.

MATERIAL, EQUIPMENT, AND OPERATING PROCEDURE

Ore used in this study was obtained from a gold mining operation in northern Nevada. The ore body is a well-oxidized hydrothermal deposit with gold finely disseminated as micrometer-size particles. The limestone-siltstone host rock has been silicified to a jasperoid which has partially weathered to a clay (10). A screen analysis of the ore (table 1) shows gold and mercury disseminated throughout all screen fractions. Chemical analysis is given in table 2.

Ore was dry-ground to minus 10-mesh in a laboratory roll crusher. Charges weighing 1 kg were wet-ground in distilled water at 50 pct solids in a ball mill using a 10-kg ball charge. The total grinding time was between 30 min and 1 h, depending on the mesh size required. The ore was generally ground to 90 pct minus 200 mesh, except as noted. The slurry was filtered, and the filter cake was dried at ambient temperature for several days.

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[3]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

TABLE 1. - Mercury and gold analysis of
several ore screen fractions

Mesh size	Wt pct	Analysis		Distribution, pct	
		Hg, ppm	Au, oz/ton	Hg	Au
Plus 48...	63.4	17	0.084	63.4	70.5
Plus 270..	24.2	17	.061	24.2	19.5
Minus 270.	12.4	17	.061	12.4	10

TABLE 2. - Chemical analyses of ore,
percent[1]

Ag..oz/ton..	0.056		Hg.....ppm..		17
Al..........	3.2		Mg..........		0.64
As..........	0.12		Mn..........		0.11
Au..oz/ton..	0.076		Mo..........		0.02
Ba..........	0.077		Ni..........		<0.01
Ca..........	8.9		Ph..........		0.18
Cd..........	0.005		Pb..........		0.01
Co..........	<0.01		Si..........		=>23
Cr..........	0.01		Th..........		<0.01
Cu..........	0.008		Ti..........		<0.15
Fe..........	4		Zn..........		0.026

[1]Except as otherwise indicated.

The sodium cyanide, lime, and sodium
and calcium sulfides used in this study
were all reagent grade. A 16- by 20-
mesh coconut shell activated carbon
was used in the sulfide precipitation
investigation.

Leaching tests to determine the effect
of NaCN, pH, temperature, and retention
time were conducted in an air-agitated
Pachuca-type vessel, shown in figure 1.
This vessel was a 1-L separatory funnel
with attachments at the bottom for intro-
ducing air to aerate and agitate the
solution. The vessel was heated with
electrical heating tape, covered with in-
sulation and aluminum foil, and regulated
by a powerstat. An NaOH scrubbing column
was placed in the top of the vessel to
prevent HCN losses. Pachuca leaching
tests were initiated by adding 50 g of
dried, ground ore to 200 mL of leaching
solution at a predetermined temperature
in the leaching vessel and leaching
for varying lengths of time. Airflow
to the vessel was 2 L/min. Upon test

completion, the leach slurry was filtered
on a Buchner funnel with No. 2 Whatman
filter paper. The filtrate volume was
measured, and aliquots were collected for
analysis. Residues were washed, dried at
room temperature, weighed, and analyzed
for gold and mercury by a fire assay-
atomic adsorption technique. Following a
24-h cyanide leach, mercury precipitation
was accomplished by adding either sodium
or calcium sulfide to the leach slurry.
The slurry was mixed by rolling in a 250-
to 4,000-mL closed plastic bottle for
varying lengths of time at 20° to 25° C.
The solids content was about 40 pct.

Additional sulfide precipitation tests
were conducted with carbon added to the
leach slurry. Five hundred grams of dry-
ground ore was leached with 870 mL leach
solution containing 0.34 lb NaCN per ton
of solution and 2.5 lb lime per ton of
ore. The slurry was rolled in a plastic
bottle for 24 h at 20° to 25° C. Follow-
ing cyanide leaching, varying amounts of
sulfide were added to the slurry and
mixed by rolling for 1 h. The slurry was
contacted with about 18 g/L activated
carbon for an additional hour. The car-
bon was screened from the slurry, and the
slurry was contacted with fresh carbon a
second and third time to simulate a
three-stage carbon-in-pulp adsorption.
Gold, silver, and mercury analyses were
accomplished using radioisotopes. The
isotopes were added to the leach slurry
after a 24-h cyanide leach and counted on
a Tracor Northern TN-1710 gamma counter.[4]
The carbon was counted after removal from
the slurry. Because all of the gold was
adsorbed on the carbon, the ratio of gold
counts in the slurry before carbon
adsorption to the counts on the carbon
after carbon adsorption is equivalent to
100 pct adsorption. The amount of
mercury or silver adsorbed on the carbon
was determined from the mathematical
equation:

[4]Reference to specific products does
not imply endorsement by the Bureau of
Mines.

$$\frac{Pct\ Hg}{100} = \frac{Hg\ counts\ on\ total\ carbon/Hg\ counts\ on\ 5\ mL\ starting\ slurry}{Au\ counts\ on\ total\ carbon/Au\ counts\ on\ 5\ mL\ starting\ slurry}. \qquad (6)$$

(Pct Ag is determined by substituting for Hg.)

Radioisotope analysis compared well with atomic adsorption analysis; therefore, self-determination correction factors for different sample compositions and gamma ray energies were not used.

In addition to the carbon-in-pulp tests, work was done by adding CaS to a laboratory ball mill, along with NaCN and lime. Calcium sulfide was varied from 0.012 lb to 0.09 lb per ton of ore. One thousand grams of minus 10-mesh ore, 1.25 g CaO (2.5 lb/ton ore), 0.8 g NaCN (0.53 lb/ton solution), 1,000 mL water, and the CaS were added to the ball mill and

ground for 45 min. The resulting slurry was washed into a 9-L bottle with 2,000 mL water and mixed. Gold and silver were removed from the slurry by adsorbing on 6- by 16-mesh activated carbon at ambient temperatures in three stages. One gram of carbon was used in each stage. The first-stage carbon was in contact with the slurry for 4 h, the second stage for 2 h, and the third stage for 18 h. Gold, silver, and mercury on the carbon were analyzed by neutron activation and gamma counting. The residue and solution were determined by atomic adsorption.

RESULTS AND DISCUSSION

EFFECT OF PROCESS VARIABLES ON MERCURY EXTRACTION

Cyanide Concentration

The effect of NaCN concentration on mercury extraction was determined by varying the NaCN concentration between 0.34 and 20 lb NaCN per ton of solution. Minus 325-mesh ore was slurried and air-agitated for 8 h at pH 11.5 and 20° C. The results of these tests are shown in figure 2. Mercury extraction increased from 10 pct at 0.34 lb NaCN per ton of solution to 40 pct at 20 lb NaCN per ton of solution.

Currently, cyanide mill operators add 0.02 to 2 lb NaCN per ton of solution. Some gold producers are interested in increasing mercury recovery; to others, mercury is a nuisance. The cost of increased cyanide would have to be weighted against the value of mercury. Gold extraction was unaffected over the range studied.

Leaching Time

The leaching time was varied from 8 to 48 h. Ore ground to minus 200-mesh was

leached at pH 11.5 and 20° C with 10 lb NaCN per ton of solution and air agitation. The effect of leaching time on mercury extraction is shown in figure 3. The data show that 24 pct of the mercury was extracted during the first 8 h; additional leaching for 40 h extracted another 4 pct. A leaching time of 1 h or less was required to recover 82 pct of the gold, and 15 h was required to recover 90 pct. In some mill operations the ore is in contact with cyanide solution during grinding, solid-liquid separation, leaching of thickener underflow, and carbon-in-pulp treatment so that the total retention time is more than 24 h. Controlling mercury extraction by reducing contact time would be difficult.

Solution pH

Sodium cyanide leach solution containing 10 lb NaCN per ton of solution was adjusted to pH values between 10.6 and 11.9 with lime and mixed with minus 325-mesh ore for 8 h. The results of these tests are shown in figure 4. Mercury extraction increased from 21 pct at pH 10.6 to a maximum of 32 pct at pH 11.5. Gold extraction increased from 91 pct at pH 10.6 to a maximum of 97 pct at pH 11.5.
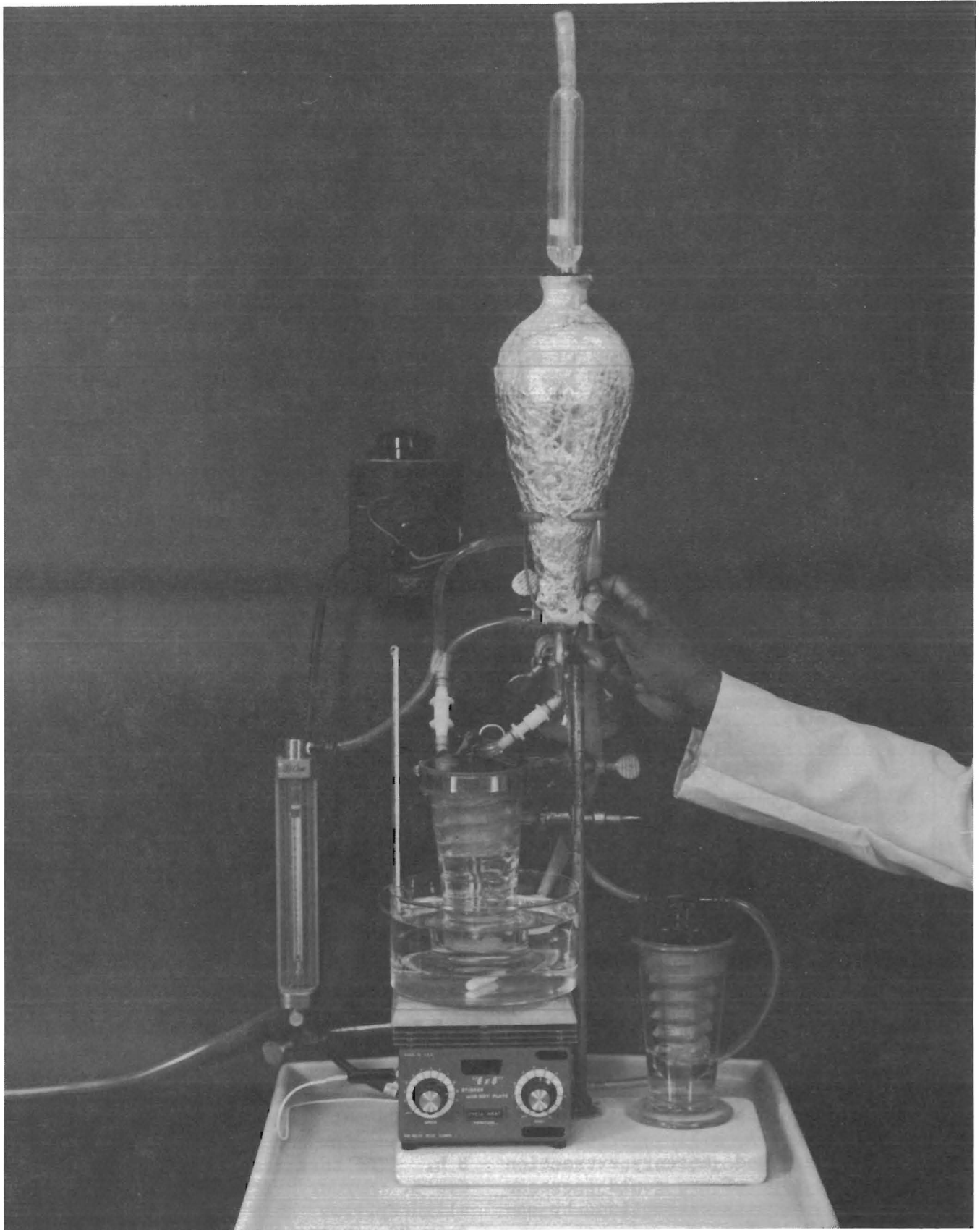
FIGURE 1. - Pachuca leaching vessel for mercury extraction.
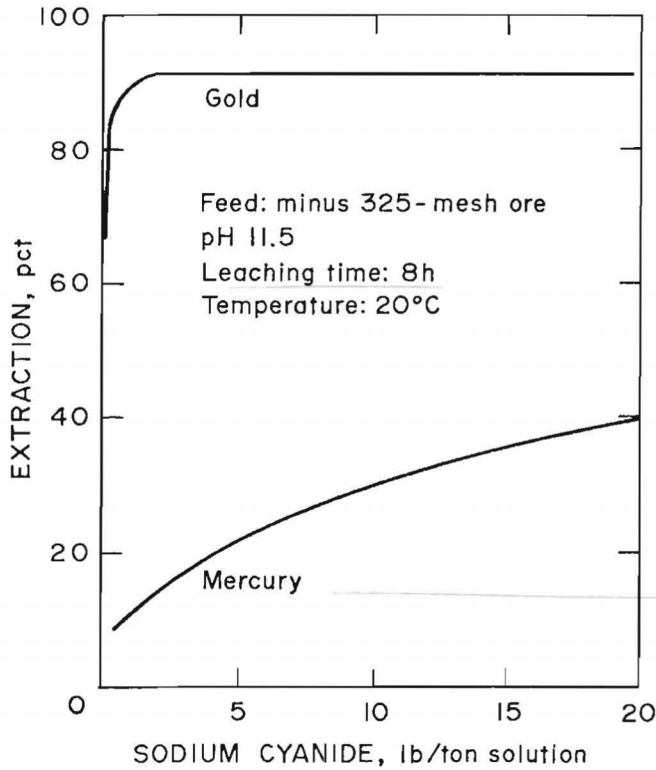
FIGURE 2. - Effect of sodium cyanide concentration on mercury and gold extraction.

FIGURE 3. - Effect of leaching time on mercury and gold extraction.

FIGURE 4. - Effect of pH on mercury and gold extraction.
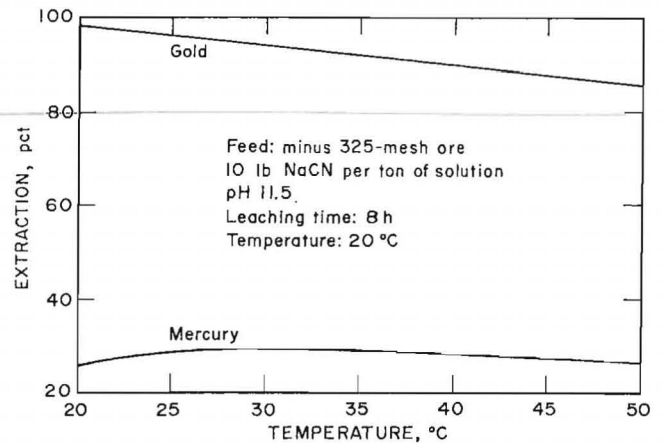
FIGURE 5. - Effect of temperature on mercury and gold extraction.

Temperature

Gold operations generally leach between pH 10.5 to 11. A mill operation interested in increased mercury extraction from a similar type of ore could increase both gold and mercury extraction by operating near pH 11.5.

A test series to determine temperature effects on gold and mercury extraction was conducted using minus 325-mesh ore. Leach solution of pH 11.5 containing 10 lb NaCN per ton of solution was air-agitated with the ore at 20° to 50° C for 8 h. The test results are shown in figure 5. Mercury extraction increased only slightly, but gold extraction decreased from 98 pct at 20° C to 85 pct at 50° C.

The decreased gold extraction may be caused by the partial destruction of sodium cyanide or reduction of dissolved oxygen at elevated temperature.

Particle Size

Different particle size fractions were leached at pH 11.5 and 20° C for 8 h with a cyanide leach solution containing 1 to 10 lb NaCN per ton of solution. Test results for mercury are shown in figure 6. Generally, mercury extraction increased as particle size decreased and sodium cyanide concentration increased. Mercury extraction from minus 10- plus 48-mesh and minus 48- plus 270-mesh fractions increased by 5 and 10 pct, respectively, as the NaCN increased. Mercury extraction from minus 270-mesh and minus 325-mesh particle size fractions increased from about 9 pct to 30 pct as the NaCN concentration was increased from 1 to 10 lb NaCN per ton of solution. Grinding to less than minus 270-mesh does not improve mercury extraction.

Additional tests showed that gold extraction increased from 73 pct to 91 pct as the particle size decreased from minus 10 plus 48 mesh to minus 270 mesh.
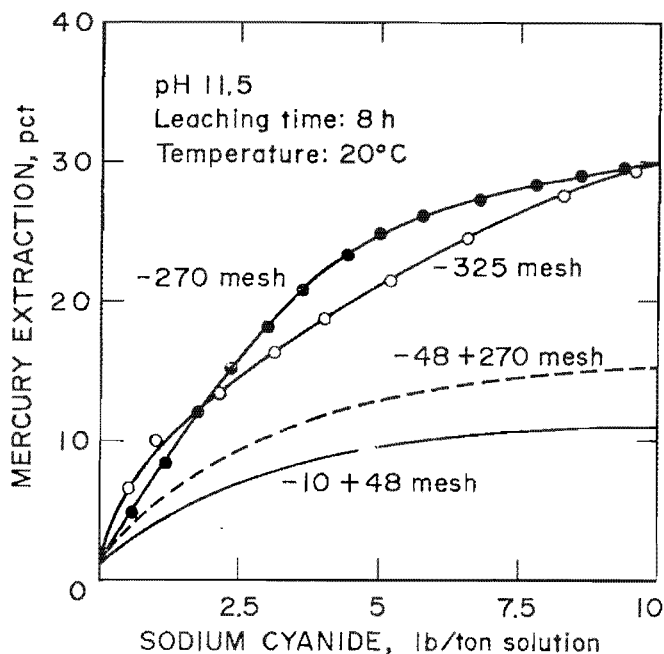
MERCURY REMOVAL BY SULFIDE PRECIPITATION

Figures 2-6 show that >10 pct of the mercury was extracted from the ore under all conditions. Mercury must therefore be recovered as a byproduct or precipitated as an insoluble product and disposed of to the tailings pond. Sulfide precipitation offers an alternative method for gold producers who are faced with a mercury problem and are not interested in mercury recovery.

Mercury Precipitation From Leach Slurry

Gold ore having 17 ppm mercury was leached with solutions containing 0.34 and 6 lb NaCN per ton of solution and lime, and then contacted with Na_2S. The results of these tests are shown in figure 7. Increasing Na_2S lowered mercury extraction when low concentrations of cyanide (0.34 lb NaCN per ton of solution) were used. However, increasing the sulfide in solution increased mercury extraction when high concentrations of cyanide were present in the leach solution. This is probably due to the formation of a soluble HgS_2^{2-} complex [11]. High concentrations of Na_2S have been used to extract mercury from concentrates [12]; therefore, care must be taken to control the amount of sulfide added.

To determine the effect of Na_2S concentration and time on mercury sulfide precipitation, additional tests were conducted. Cyanide solutions were contacted with ore for 24 h to leach 12 pct of the mercury; then Na_2S was added. The results are listed in table 3. Sodium sulfide precipitated >99.9 pct of the solubilized mercury with 0.02 lb Na_2S per ton ore, but within 0.5 h, the precipitated mercury started to redissolve. After 4 h, about 30 pct of the precipitated mercury sulfide had redissolved.

FIGURE 6. - Effect of sodium cyanide concentration and particle size on mercury extraction.

TABLE 3. - Mercury sulfide precipitation and redissolution from cyanide leach slurries with sodium sulfide

Test	Na$_2$S, lb/ton ore	Time, h	Hg redissolved, pct
1	0.02	0.25	<0.01
		.5	.9
		1	1.6
		2	2.3
		4	3.6
2	.05	.5	.1

[1]Leaching conditions prior to Na$_2$S addition: 0.34 lb NaCN per ton of solution, 2.5 lb lime per ton of ore, 40 pct solids, ambient temperature, 24 h.

Because of redissolution of mercury sulfide with Na$_2$S, tests were conducted with CaS. Cyanide solutions at 0.34 and 1.0 lb NaCN per ton of solution were used to extract 12 and 27 pct, respectively, of the mercury in 24 h. After leaching,
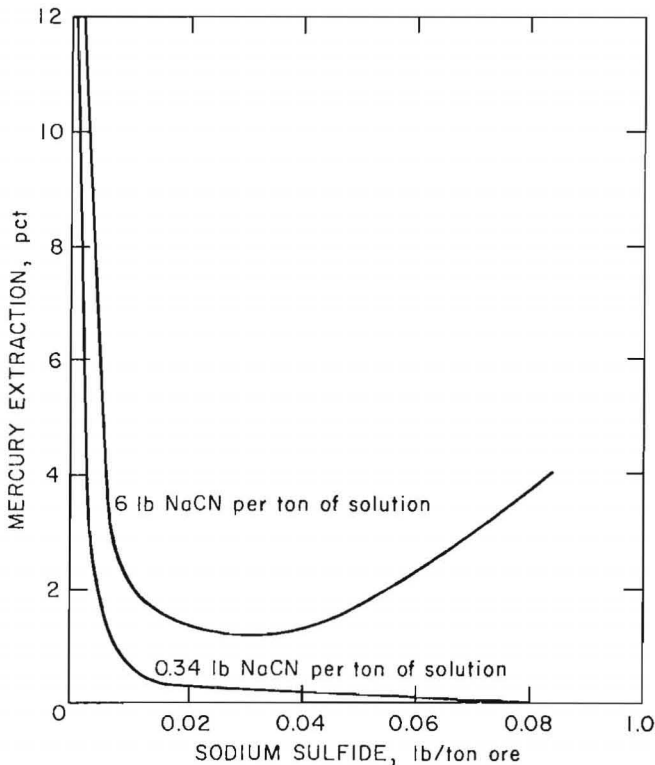
CaS was added and samples were taken periodically. The results of these tests are listed in table 4. Calcium sulfide precipitated about 96 pct of the solubilized mercury in 0.25 h, and 99.8 pct in 0.5 h, using 0.02 lb CaS per ton of ore. After 7 h, about 7 pct of the precipitated mercury sulfide had redissolved. Increasing the CaS concentration to 0.09 lb per ton of ore resulted in nearly 100 pct of the soluble mercury precipitating, and after 24 h the mercury sulfide did not redissolve.

TABLE 4. - Mercury sulfide precipitation and redissolution from cyanide leach slurries with calcium sulfide

Test	CaS, lb/ton ore	Time, h	Hg redissolved, pct
1[1]	0.007	0.25	[2]6
		1	2
		2	2
		4	3
		7	4
		24	12
2[1]	.02	.25	[2]3.6
		.5	.2
		3	.3
		7	.8
3[1]	.09	24	<.01
4[3]	.02	.25	.2
5[3]	.08	1	.4
		4	.4
		24	.4

[1]Leaching conditions prior to CaS addition: 0.34 lb NaCN per ton of solution, 2.5 lb lime per ton of ore, 40 pct solids, ambient temperature, 24 h.
[2]Mercury precipitation still occurring.
[3]Leaching conditions prior to CaS addition: 0.34 lb NaCN per ton of solution, 1.5 lb lime per ton of ore, 25 pct solids, ambient temperature, 24 h.

A comparison between mercury sulfide redissolution with Na$_2$S and CaS is shown in figure 8 (test 1, table 3; and test 2, table 4). About 10 times more mercury sulfide redissolved with Na$_2$S than with CaS after 4 h.

FIGURE 7. - Effect of sodium sulfide and sodium cyanide on mercury extraction.
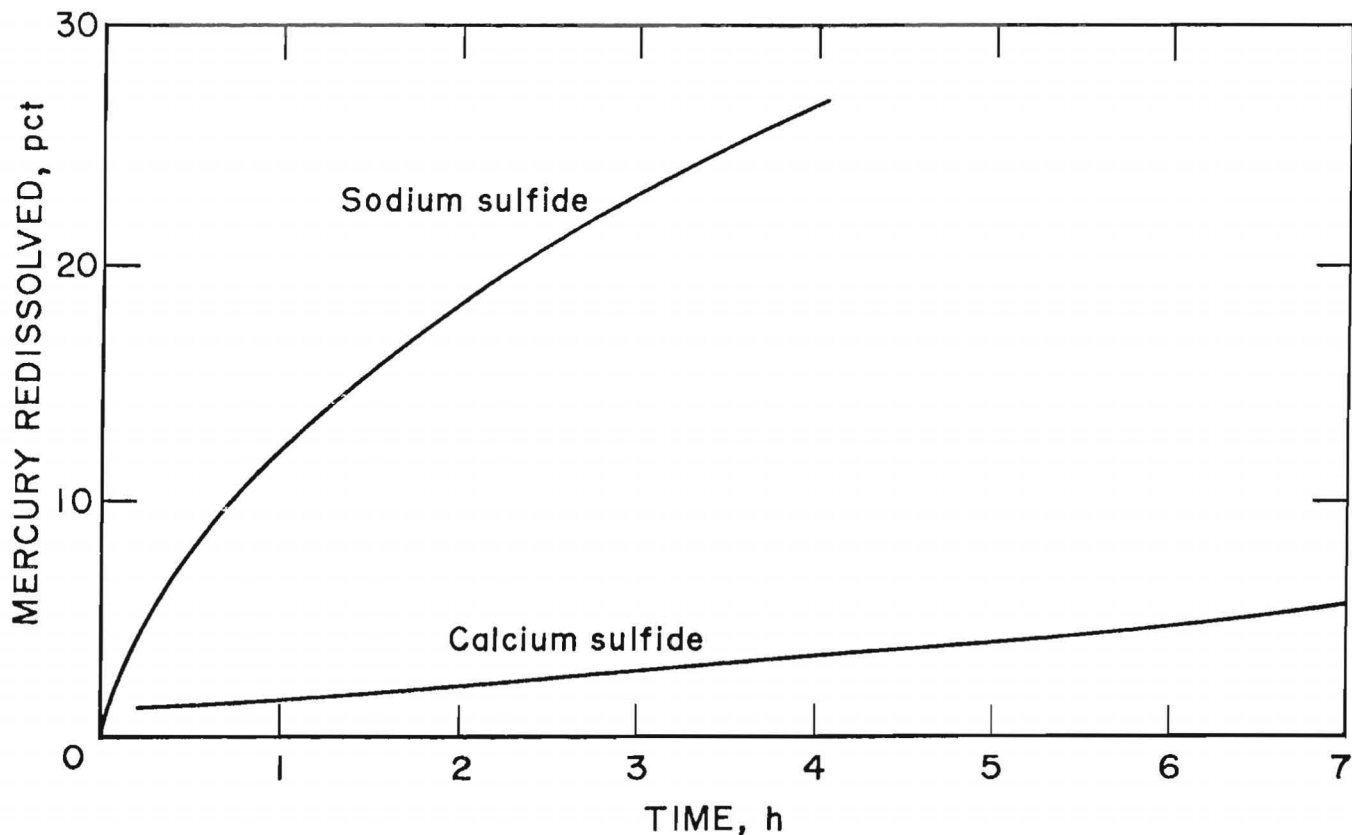
FIGURE 8. - Effect of time on sulfide-precipitated mercury.

Mercury Precipitation Prior to the Carbon-In-Pulp Circuit

Tests were conducted to determine the effect of sulfide addition on carbon adsorption of gold and mercury in a laboratory carbon-in-pulp (CIP) circuit. The results of these tests are given in table 5.

Gold and silver adsorption on carbon without prior sulfide addition was nearly 100 pct after the first stage, while only 61 pct of the soluble mercury adsorbed. Additional adsorption stages recovered all the gold and silver and 9 pct more of the mercury. Addition of 0.023 lb CaS per ton of ore did not decrease gold and silver recovery, but reduced mercury adsorption to about 5 pct. Increasing the CaS concentration to 0.047 lb CaS per ton of ore caused a slight decrease in silver adsorption in the first stage and slightly less overall recovery, but mercury adsorption dropped to 0.9 pct in the three stages.

TABLE 5. - Carbon-in-pulp adsorption with sulfide precipitation, percent[1]

Carbon stage	Au	Ag	Hg
0 lb CaS:[2]			
Stage 1...............	99	97	61
Stage 2...............	1	3	7
Stage 3...............	0	0	2
0.023 lb CaS:[2]			
Stage 1...............	99	96	4.7
Stage 2...............	1	4	.4
Stage 3...............	0	0	.3
0.047 lb CaS:[2]			
Stage 1...............	99	95	.37
Stage 2...............	.6	4.1	.31
Stage 3...............	.1	.5	.20

[1]Percent of soluble metal adsorbed on carbon.
[2]Per ton of ore.

Sulfide precipitation could be used in the present mill operation with only minor modification. Sulfide addition should be made prior to adsorption and

Sodium cyanide

Ore ——

Lime

Grinding

Water recycle

Calcium sulfide

Thickening → Underflow → Leaching

Overflow

Calcium sulfide

Mercury precipitation

Carbon-in-pulp → Tailings pond (HgS)

Filtration → Mercury sulfide

Carbon columns

Carbon regeneration

Carbon stripping

Carbon-in-pulp section

Electrowinning
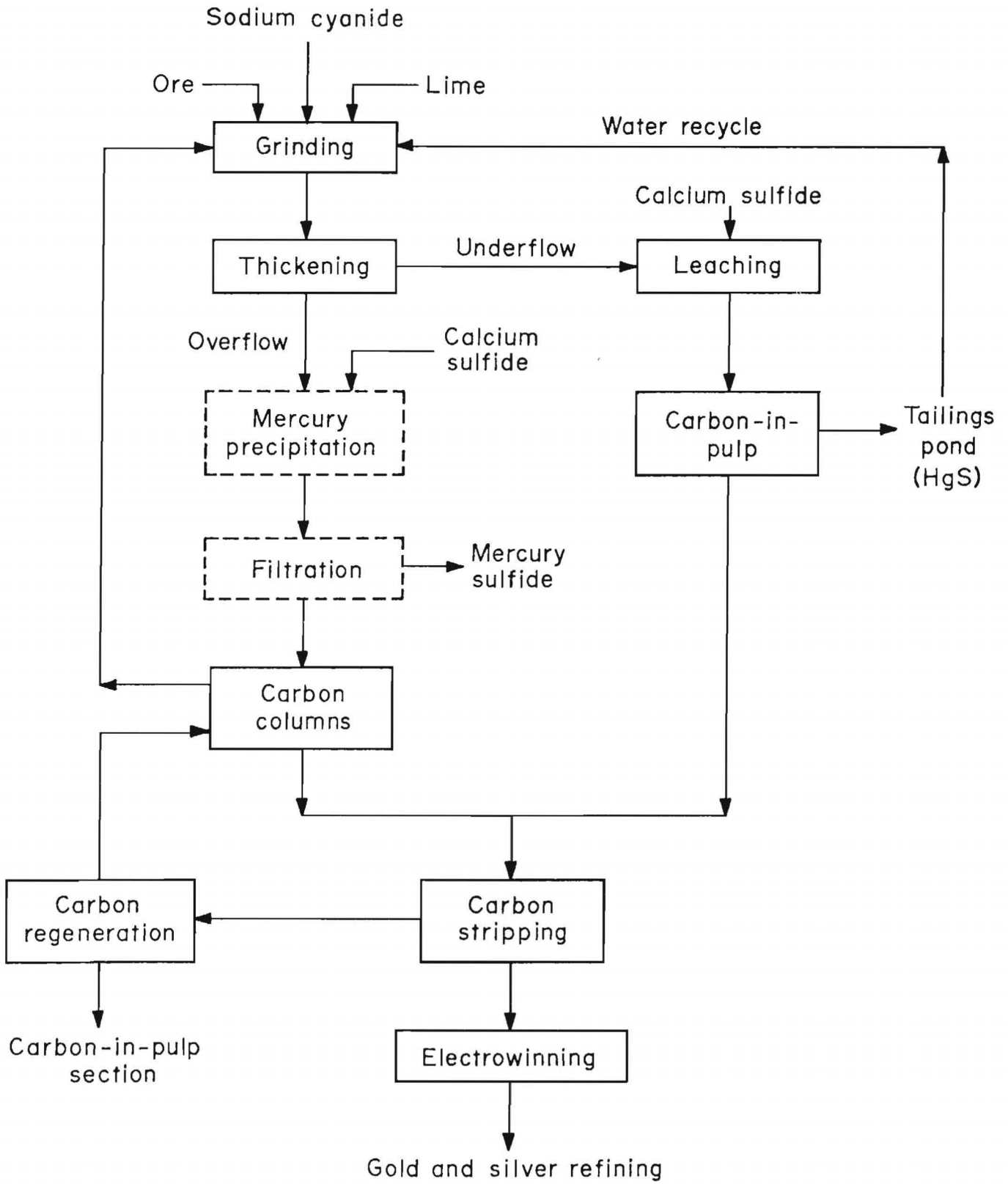
Gold and silver refining

FIGURE 9. - Mercury precipitation from gold cyanide mill solution.

enough time provided to precipitate the mercury. A schematic of a northern Nevada mill operation with required changes for sulfide precipitation is proposed in figure 9. Sulfide addition could be made to the thickener underflow and the clear thickener overflow. In this flowsheet, the underflow goes through a series of leach tanks. Sulfide would be added to the last tank prior to entering the CIP circuit. Mercury in the thickener overflow would be recovered by sulfide precipitation and filtration. The slurry from the CIP circuit would contain precipitated mercury and go to the tailing pond. The remaining part of the flowsheet would be stripping, electrowinning, and refining. Additional tests are required to determine if the strip solution is low enough in mercury so that the gold cathode is not contaminated during the electrolysis.
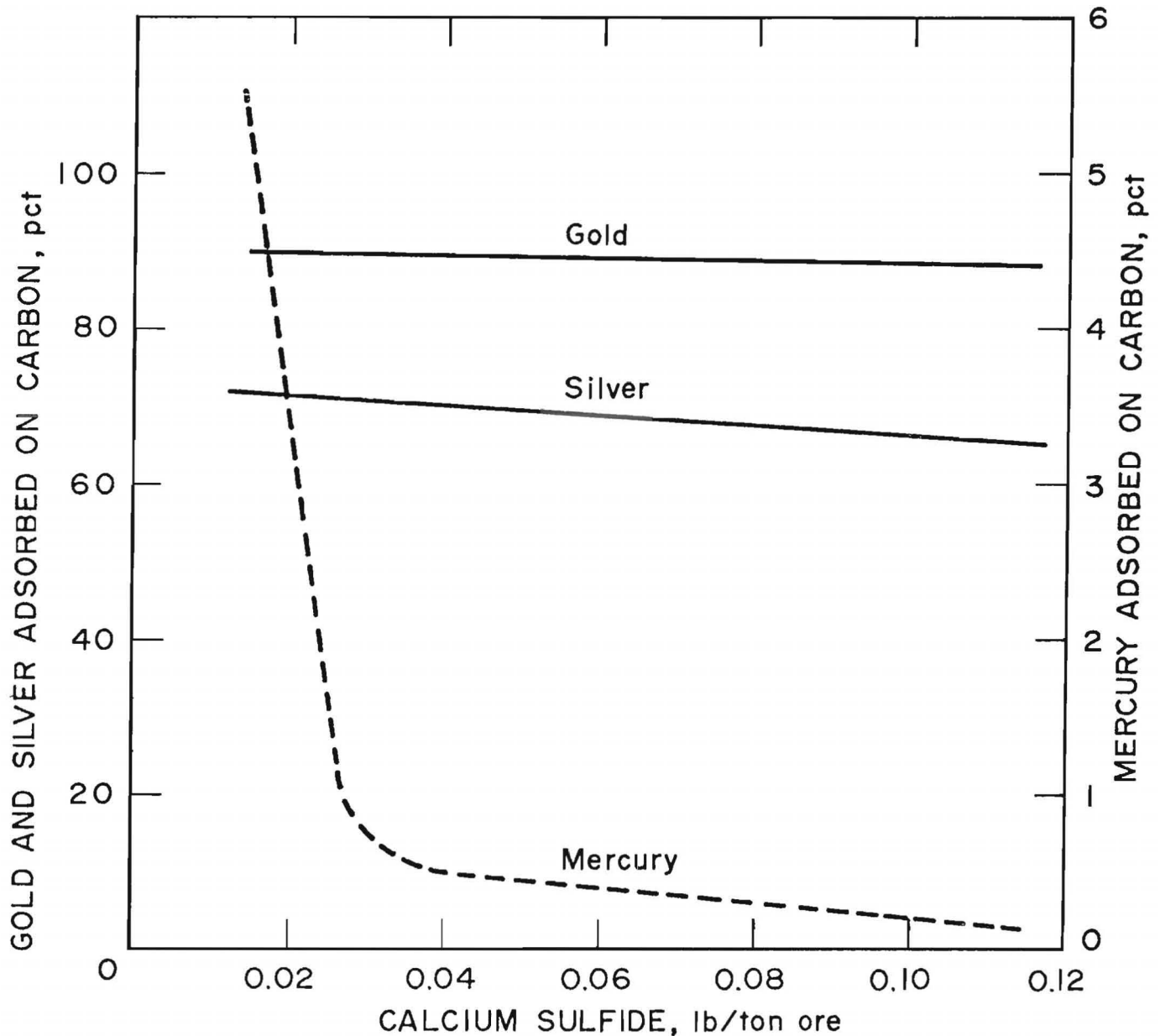

FIGURE 10. - Effect of calcium sulfide additions during grinding on gold, silver, and mercury loading on carbon.

Mercury Precipitation in a Laboratory Grinding Circuit

Several gold operations add NaCN and lime to the grinding circuit to extract gold and silver during grinding. Some of the mercury is also extracted at this time. Tests were conducted to determine if CaS added with NaCN and lime to the ball mill would prevent mercury extraction and affect gold, silver, and mercury loading on carbon. The results of these tests are given in table 6 and figure 10.

TABLE 6. - Addition of calcium sulfide during grinding and its effect on mercury, gold, and silver extraction and adsorption

CaS, lb/ton ore	Extraction, pct[1]			Adsorption on carbon, pct[2]		
	Au	Ag	Hg	Au	Ag	Hg
0.012	99	86	15	91	71	5.5
.024	94	98	1.5	87	74	.55
.048	94	98	2	88	67	.40
.096	95	98	.4	89	64	.17
.096	94	NAp	1.4	NAp	NAp	NAp

NAp Not applicable.
[1]Total extraction including grinding (45 min) and carbon addition (24 h).
[2]Total over 24 h contact with carbon.

Mercury extraction and adsorption on carbon were decreased from 15 to 0.4 pct and 5.5 to 0.17 pct, respectively, by increasing the amount of CaS added to the simulated ball mill circuit from 0.012 to 0.096 lb per ton of ore. Gold extraction may decrease slightly, but additional research is required to verify this. Gold loading on carbon was unaffected. Silver extraction was unaffected, but carbon loading may have decreased slightly. Both gold and silver recovery on carbon was lower than in previous sulfide precipitation tests. This was due to using much less carbon (0.33 g/L rather than the normal 20 g/L). Decreasing the carbon concentration made it possible to analyze for the small amount of mercury adsorbed on the carbon.

Adding CaS to the ball mill would simplify the flowsheet in figure 9. The extra equipment would be unnecessary since the mercury would remain with the residue and be disposed of in the tailings pond. Essentially no change in the mill operation would be required.

CONCLUSIONS

Mercury extraction was decreased from 40 to 10 pct by (1) decreasing NaCN concentration from 20 to 0.34 lb NaCN per ton solution, (2) decreasing the pH from 11.5 to 11, and (3) increasing particle size from minus 270 mesh to minus 10 plus 48 mesh.

Calcium sulfide addition to cyanide leach slurry or to a laboratory grinding circuit containing NaCN and lime reduced mercury dissolution to <0.5 pct. Mercury loading on activated carbon was reduced to <0.2 pct. Gold loading on activated carbon was affected very little by the sulfide addition, but silver may be slightly affected. Additional tests are required to verify this.

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