

MERCURY CONTROL IN THE CYANIDATION OF GOLD ORES

J. D. Miller¹, E. Alfaro¹, M. Misra², J. Lorenzo²

¹ Department of Metallurgical Engineering, University of Utah, Salt Lake City, UT 84112

² Department of Metallurgical Engineering, University of Nevada Reno, NV 89557

ABSTRACT

The control of mercury during gold cyanidation is of particular interest to the gold mining industry. Typically, mercury(II) is released as cyano complexes during leaching and follows gold through the solution concentration and purification step (carbon adsorption and electrowinning or zinc cementation). Ultimately, prior to the production of doré metal, elemental mercury is removed by retorting. Process alternatives for mercury control are considered; not only the isolation and removal of mercury from the system but also the stabilization and fixation of mercury in the ore so that it is not released during cyanide leaching. In this regard, a number of different chemical processes for mercury control are reviewed and their usefulness discussed based upon their characteristic features. These processes include precipitation, adsorption, solvent extraction, ion exchange and cementation.

INTRODUCTION

Although the annual production of gold in the United States has been increasing steadily, oxide gold ore reserves are rapidly diminishing. In this regard, more complex ores, including refractory gold ores and low-grade ores, are being processed. In many cases, these resources contain significant amounts of mercury along with the gold values. Generally, the extraction of gold is accomplished by cyanidation in which leaching occurs by the addition of cyanide at alkaline pH. Cyanide, which is a strong lixiviant for gold, is an equally strong lixiviant for mercury. According to Sharpe (1), simple and complex cyanides of mercury are restricted to the oxidation state of 2 and dissolve as the $\text{Hg}(\text{CN})_2^0$ and $\text{Hg}(\text{CN})_4^{2-}$ complexes of mercury. The distribution diagram is shown in Figure 1.

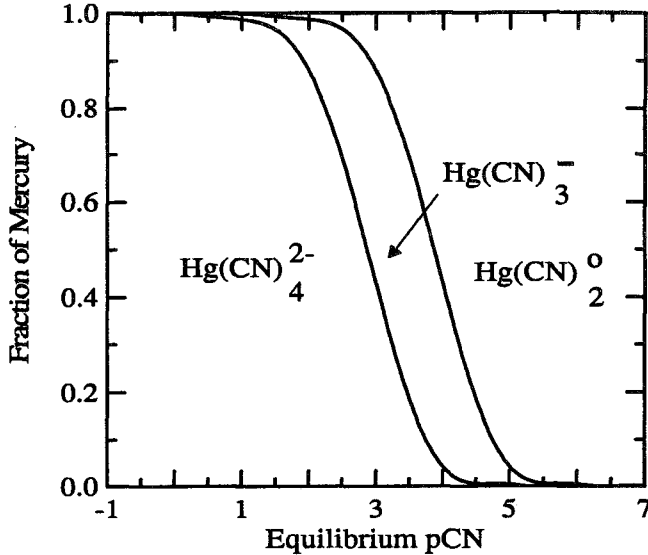


Figure 1: Distribution diagram for mercury cyanide complexes.

The presence of mercury in all steps of cyanidation poses a threat to both the environment and to the health of the plant workers. The presence of high concentrations of mercury (> 50 g/t) has been shown to reduce significantly the efficiency of gold cementation with zinc (2). Mercury vapor can be released during carbon stripping, carbon regeneration, electrowinning, and retorting (3). Further, in the CIP process, mercury builds up in the recycled leach solutions and report to the tailings because only part of the mercury is adsorbed on carbon in the loading circuit (4). Thus tailings impoundment can also represent an environmental concern.

Inorganic mercury can be transformed into methylmercury and dimethylmercury by the action of micro-organisms under aerobic conditions and is favoured by alkaline conditions. These compounds are volatile and can be released to the atmosphere (5). Furthermore, the methylation of mercury by sulfide reducing bacteria, producing a form of mercury that is more toxic and biologically available to biota, has been demonstrated (6).

It is evident that the problem of mercury in the cyanidation of gold and silver ores is a two fold problem:

1. the presence, and possible volatilization of mercury from post-leaching process streams which presents environmental and health problems, and
2. the presence of mercury, in its various forms, within the tailings impoundment.

Methods to control mercury during gold cyanidation are of particular interest to the gold mining industry. In this regard, a number of different chemical processes for mercury control are reviewed and their usefulness discussed based upon their characteristic features. These include precipitation, adsorption, solvent extraction, ion exchange and cementation.

PRECIPITATION

Selective stabilization and/or removal of mercury can be accomplished by precipitation with inorganic sulfides and other thiol based organic compounds. The gold cyanide complex is stable with respect to sulfide precipitation in alkaline cyanide solutions and on this basis a selective separation is possible.

Inorganic Sulfides

The precipitation of mercury from alkaline gold cyanide solutions has received some attention in the past few years, and studies have been reported using H_2S , Ag_2S , FeS , Na_2S , and CaS (4, 7). Sandberg et. al (4) at the U.S. Bureau of Mines in Salt Lake City, Utah have reported on mercury precipitation from gold cyanide leach solutions with CaS and Na_2S . They found that Na_2S and CaS can serve as precipitants for mercury but report that redissolution of mercury occurs when Na_2S is used (4). Calcium sulfide, on the other hand, is reported to minimize the rate of mercury redissolution but, like all other sulfides, tends to precipitate silver from solution as well. However, it was found that the addition of copper to solution reduced the extent of silver precipitation while having no effect on the mercury precipitation rate (7).

The tendency of mercury to redissolve from HgS , the precipitant formed by the above reagents, can be explained by the Eh-pH diagram for the Hg-S-Water system shown in Figure 2. The stability domain for HgS is situated in a very narrow Eh region at the pH for cyanidation (10.5 to 11). Consequently, even a modest change in the oxidation potential could lead to the destabilization of HgS and may explain the redissolution reported in the literature.

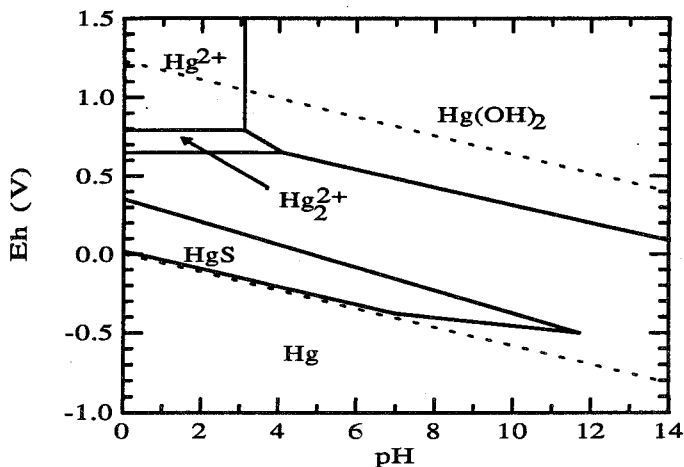


Figure 2. Electrochemical phase diagram for the mercury-sulfur-water system at 25°C. (Total dissolved sulfur = 8.6×10^{-5} M, total dissolved mercury = 4.3×10^{-5} M)

Sulfur Based Organic Compounds

The use of thiol surfactants and sulfur-based organic compounds (SBOCs) as precipitants of mercury from cyanide leach solutions is currently under investigation at the University of Nevada. This research is based on the results from preliminary experiments which show that thiol surfactants and SBOCs have the potential to form insoluble organo-metallic compounds of mercury with a far greater stability than HgS. It is reported that SBOCs also have the ability to precipitate both the dicyano and tetracyano complexes of mercury, $\text{Hg}(\text{CN})_2$ and $\text{Hg}(\text{CN})_4^{2-}$ respectively, while Na_2S and CaS are only able to efficiently precipitate HgS from the tetracyano complex and not from the more stable dicyano mercury complex.

SBOCs form insoluble metallic salts except with alkali and alkaline earth elements (8). Furthermore, mercury salts prepared from SBOCs have the characteristic of being the most insoluble of such thiol salts and have been observed to form very massive and dense particles. Table 1 shows the precipitation from $\text{Hg}(\text{CN})_2$ and $\text{Hg}(\text{CN})_4^{2-}$ solutions with the addition of an appropriate SBOC. As can be seen, more than 95% of both cyanomercury complexes can be precipitated at a molar ratio of SBOC to Hg of 2:1. Preliminary experiments suggest that mercury can be stabilized with the addition of SBOC during cyanidation without any detrimental effect on gold recovery.

Table 1. Precipitation and Removal of Mercury from Cyanide Solutions with SBOC.

Molar Ratio SBOC/Hg	Percent Precipitated from $\text{Hg}(\text{CN})_2$ ^o Solution	Percent Precipitated from $\text{Hg}(\text{CN})_4^{2-}$ Solution
1:1	71.5	96.0
2:1	96.8	95.8
4:1	99.5	97.0
10:1	97.8	97.8
20:1	98.0	99.0

ADSORPTION

Activated Carbon

Of course, it is well known that gold is adsorbed from cyanide leach solutions by activated carbon. The graphitic structure of activated carbon appears to be the major factor influencing gold adsorption from alkaline cyanide solutions (9, 10, 11). Although activated carbon is highly selective for gold and silver over most other metal species, under certain conditions mercury is an important exception. It has been reported that the neutral mercury cyanide complex, $\text{Hg}(\text{CN})_2^o$, competes directly with $\text{Au}(\text{CN})_2^-$ for adsorption sites and can even displace some of the adsorbed gold from carbon (12). Examination of adsorption isotherms confirms that the adsorption potential of activated carbon is similar for gold and mercury and that a

selective separation should not be expected (13). Nevertheless, it appears that selective stripping of mercury from activated carbon may be possible under appropriate control of process conditions. For example, at room temperature and high cyanide concentration mercury can be selectively stripped from a mercury-gold loaded carbon at a slow rate resulting in 95% removal of the mercury with essentially no gold release.

Crumb Rubber

Discarded automobile tires upon treatment, may become effective sorbents for the removal of heavy metals in waste effluents, particularly mine drainage (14). Although crumb rubber from waste automotive tires has been shown to adsorb metals from acid solutions (15), the use of this material for the selective removal of mercury from alkaline gold cyanide solutions has not been considered until recently.

Synthetic rubber is composed primarily of polymerized polyisoprene, and polybutadiene-styrene containing sulfur and carbon black. This carbon black (approx.30% w/w), has been shown to have adsorption properties similar to activated charcoal (16). In this regard, it has been suggested that for mercury(II) adsorption from acid solutions by scrap rubber the carbon black may well provide the main sites for adsorption and that sulfur sites or other constituents of vulcanized rubber are of secondary importance. This position is supported by tests with sulfur-free rubber which were shown to be equally efficient for the removal of inorganic mercury from acid or neutral solutions (17).

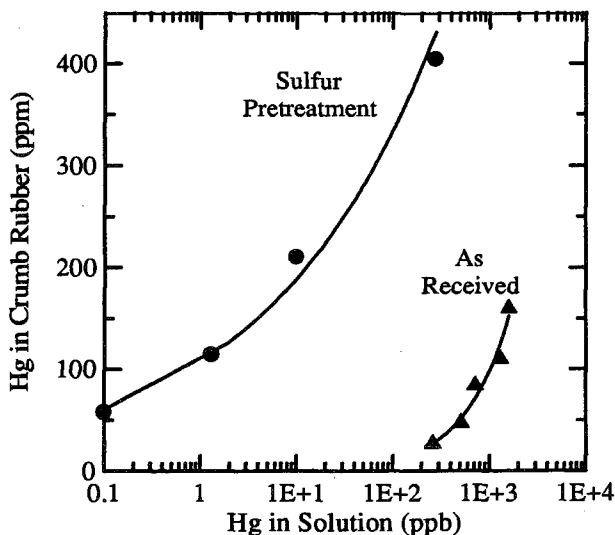


Figure 3. Adsorption of mercury from alkaline cyanide solution (pH 11) using crumb rubber (-140 mesh).

Of course, sulfur is present in vulcanized rubber (approx. 1-2% w/w) in the form of thiol groups and polysulfide links which might reasonably be expected to provide sites for the adsorption of "soft" metals such as mercury (18). Also it has been postulated that adsorption of mercury(II) from aqueous solutions by vulcanized rubber occurs at least partially by an ion-exchange type mechanism involving displacement of zinc(II) from the rubber structure (18).

In view of the foregoing, research is in progress at the University of Utah to selectively remove mercury from alkaline cyanide solutions using crumb rubber from recycled automotive tires. Minus 140 mesh crumb rubber was equilibrated with alkaline cyanide solutions containing gold and mercury. The extent of mercury removal is significant, as presented in Figure 3. This crumb rubber was used as received and pre-treated with elemental sulfur at about 130°C. Importantly, gold was not adsorbed under these conditions.

Further research needs to be done in order to define the optimum conditions in the preparation of sulfur treated crumb rubber for the selective removal of mercury from alkaline cyanide solutions.

SOLVENT EXTRACTION

Even though solvent extraction is a well-known hydrometallurgical process, it has not found application in the gold mining industry, primarily because solvent extraction appears to be uneconomical in comparison with cementation and carbon adsorption. Nevertheless, some research at the University of Utah has shown that solvent extraction can be successful in the selective extraction of gold using modified amines, alkyl phosphorous esters, and guanidines (13, 19). Typical cyanoanions such as copper, zinc, and iron are not extracted from alkaline cyanide solutions by these extractants. However, recent experiments have shown that mercury cyano complexes have a different behavior and are co-extracted with gold. One exception is the alkyl phosphorous esters for which good gold selectivity with respect to mercury can be achieved (20).

Solvation Extraction by Alkyl Phosphorous Esters

The principal advantages of gold solvent extraction by alkyl phosphorous esters are the fast reaction kinetics and the excellent selectivity for gold extraction with respect to typical cyanoanions (13). The principal disadvantage found, particularly when using dibutyl butyl phosphonate (DBBP), was the difficulty in stripping gold from the organic phase. This problem might be solved by direct electrolysis of the organic solution for gold recovery (21).

Undiluted DBBP has been shown to be an excellent extractant for gold over a wide range of pH values, requires minimal ionic strength adjustments (21, 22) and the losses of organic to the aqueous phase are reported to be less than 20 ppm (22). Also, the selectivity factors for gold over other transition metal cyanoanions [$\text{Cu}(\text{CN})_4^{2-}$, $\text{Zn}(\text{CN})_4^{2-}$, $\text{Ni}(\text{CN})_4^{2-}$, $\text{Fe}(\text{CN})_6^{3-}$] have been reported to be high as 1000. Only silver is extracted at a moderate level (21, 23).

Gold extraction chemistry by DBBP and other alkyl phosphorous esters was found to be due to the solvation extraction of the aurocyanide ion pair which in the case of a sodium counter ion can be represented as:



In the case of mercury, at high cyanide concentrations, greater than $\sim 1 \times 10^{-2}$ M, the tetracyanoanion, $\text{Hg}(\text{CN})_2^{2-}$, is not extracted from alkaline cyanide solution as an ion pair due to its charge and size (19, 21, 23). At lower cyanide concentrations, less than $\sim 1 \times 10^{-2}$ M, the tetracyano complex is unstable and the neutral aqueous species, $\text{Hg}(\text{CN})_2^0$, is stabilized which is extracted by DBBP, again via a solvation mechanism:



It is also conceivable that this neutral aqueous species is stabilized at low pH values and such a reaction may account for DBBP extraction of mercury at low pH values even if the cyanide concentration is high.

After a detailed laboratory study on pure systems, the treatment of an eluate solution from the carbon strip circuit of a Nevada gold plant was examined. Table 2 shows the data for the carbon eluate solution and it is clear that excellent selectivity is achieved and that extraction of mercury by DBBP is so low that detection was not possible.

Table 2. The Effect of the Aqueous/Organic Phase Ratio in the Extraction of Mercury and Gold from Carbon Eluates with DBBP at 60°C .

(Initial conditions: 1.13 ppm Au, 20 ppm Hg; high concentration of NaOH and NaCN)

Phase Ratio A/O	Percent Extracted		Organic Phase, ppm	
	Au	Hg	Au	Hg
5:1	94.7	< 0.1	5.35	ND
3:1	97.4	< 0.1	3.30	ND
2:1	98.2	< 0.1	2.22	ND
1:1	99.0	0.4	1.11	0.08

When these results at 60°C are compared with the results at 21°C, they clearly show that mercury extraction by DBBP has a significant temperature dependence . This unexpected result confirms the suggestion by Miller et.al. (23), that temperature is one of the most complex and least understood variables in the solvation extraction process. The results obtained at 60°C show a good extraction of gold, slightly lower than at 21°C. However, most importantly, it was not possible to extract mercury by

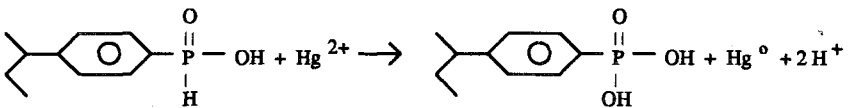
DBBP at 60°C even though significant extraction (40%) was achieved at 21°C. Clearly mercury extraction by DBBP has a significant temperature dependence.

Thiol Extractants

Of course, the possibility of using thiol extractants is of particular interest. Although mercury extraction from acid solutions has been studied, no work has been reported on mercury extraction from cyanide solutions with thiol extractants, such as dithiocarbamates and dithiozones. It is expected that excellent separations should be possible and that stripping could be achieved with concentrated solutions of cyanide.

ION EXCHANGE

The use of selective resins to remove mercury was accomplished some 25 years ago by Law (24) who used a resin with a isothiuronium group to remove mercury from aqueous solution. He reported improved removal at pH values greater than 6, but the resin was found to degrade at high pH (25). At the same time the chloride complexes of mercury were found to be a convenient way to analyze mercury using anion exchange resins (26). An important use of a thiol resin was reported by Bibler et. al (27) in a work done around a serious radioactive waste problem in which mercury was used as a catalyst for the dissolution of uranium alloys. Finally a polystyrene-supported phosphinic acid has been found to reduce mercury(II) to the metallic state:



The P-H bond is stable in water for long periods and reacts only when the metal ion reduction potential is 0.3 V. Mercury escapes from macroporous support as a liquid and is removed as such (28, 29).

In the case of alkaline cyanide solutions, the Bureau of Mines investigated the use of anion-exchange resins for the recovery of precious metals from cyanide leach solutions as a process alternative (30). Strong-base resins are generally less selective for precious metals, and are much less affected by pH than weak-base resins. Generally they are difficult to strip.

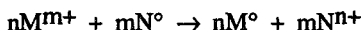
Weak-base resins are generally more selective for precious metals, but have lower loading capacities because protonation is required to extract anions, and a pH of less than 10 is usually required (31).

Both resin types can be eluted at ambient temperature and pressure. Weak-base resins generally can be eluted using a dilute caustic solution. Strong-base resins, however, are more difficult to elute and require stronger treatment (32, 33). It seems that neither the commercially available strong-base resins or weak-base resins will provide the desired characteristics for the selective separation of mercury from gold in alkaline cyanide solutions.

In view of the foregoing it is appropriate to undertake further research on the use of resins with thiol functionality in order to achieve the desired selectivity.

CEMENTATION

Cementation or contact reduction is the process by which one metal is displaced from solution by another more reactive metal according to reactions of the type:



The reaction is a heterogeneous process in which the ion of a more noble metal is displaced from solution by a less noble metal placed in contact with it. The metal displaced from the solution is deposited onto the surface of the less noble metal which is simultaneously dissolved.

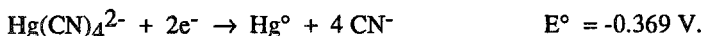
Cementation is used as a process strategy for the primary recovery of noble metals from leach solutions. The most common example of the cementation process is the use of scrap iron to reduce cupric ions from acid solutions to produce cement copper. By way of another more relevant example, metallic zinc dust is frequently used to recover gold from gold-cyanide solutions.

Cementation reactions are also used for the purification of solutions. An example of process stream purification is the addition of zinc dust to zinc electrolytes in order to eliminate cations such as copper and cadmium which would otherwise reduce the current efficiency during zinc electrodeposition. More recently, cementation reactions have been used to purify effluent streams containing heavy metals such as mercury and copper (34).

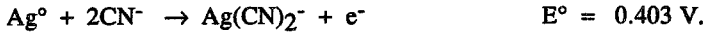
In most cases, cementation reactions have been found to follow first-order kinetics which, in the simplest case, are limited by diffusion of the noble metal through the mass transfer boundary layer. The apparent activation energy for these reactions varies from 2 to 6 kcal/mol and the reaction velocity constants are on the order of 10^{-2} cm/sec for well stirred systems (35).

Information on cementation as a practical method for the recovery of mercury from aqueous solutions is quite limited. Studies have been conducted on the use of zinc dust to recover mercury from wastewater containing mercuric chloride (36). Also, silver and mercury cementation with iron from acidic solutions has been reported (37). Finally the results from the study of mercury cementation from acidic solutions by means of rotating discs of copper and silver have been reported (38, 39). Studies of mercury cementation from alkaline cyanide solutions have not been reported and such research is currently in progress at the University of Utah.

In alkaline cyanide solutions mercury is more noble than gold and in order to achieve selective cementation of mercury from gold only a few possibilities exist. One possibility is the use of silver as the reactive or less noble metal. See Table 3. The cementation of mercury by silver from cyanide solutions involves the cathodic reduction of the tetracyano-mercury(II) complex ion:



which is coupled to the anodic dissolution of the silver as the dicyano-silver(I) ion:



A simplified schematic diagram of the electrochemical cementation reaction is given in Figure 4.

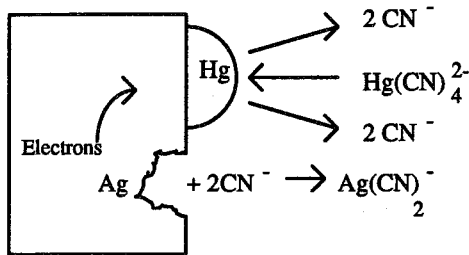


Figure 4. Schematic of the electrochemical cementation of mercury from alkaline cyanide solution with metallic silver.

The gold half cell is more reactive than the silver half cell, as shown in Table 3, and thus selective cementation should be possible. In fact, gold itself could be used for mercury removal from alkaline cyanide solutions. In this regard, cementation experiments were carried out to determine if selective cementation of mercury from gold could be achieved. Silver powder (6 to 26 microns) was used in a solution containing gold and mercury. The amount of silver used was approximately 100 times the stoichiometric requirement and some of the experimental results are presented in Figure 5. Mercury cementation from cyanide solution was about 98 %, whereas little, if any, gold was removed.

Table 3. Half Cell Reactions for Selected Metals in Cyanide Solutions.

Metal	Half Cell Reaction	$E^{\circ}(\text{V})$	$E(\text{V})^*$
Gold	$\text{Au}(\text{CN})_2^{-} + \text{e}^{-} = \text{Au} + 2\text{CN}^{-}$	-0.616	-0.633
Silver	$\text{Ag}(\text{CN})_2^{-} + \text{e}^{-} = \text{Ag} + 2\text{CN}^{-}$	-0.403	-0.405
Mercury	$\text{Hg}(\text{CN})_4^{2-} + 2\text{e}^{-} = \text{Hg} + 4\text{CN}^{-}$	-0.369	-0.260

*[CN] = 0.01 M, [Me] = 10 ppm. T = 25°C

As might be expected, the results were found to conform to first-order reaction kinetics with a reaction velocity constant ranging from 0.005 to 0.01 cm/sec. See Figure 6. The mass transfer coefficient expected under these conditions is about 0.01 cm/sec. The effects of various factors including particle size, cyanide concentration, pH, and agitation need to be investigated to further evaluate cementation for the selective separation of mercury from gold in alkaline cyanide solutions.

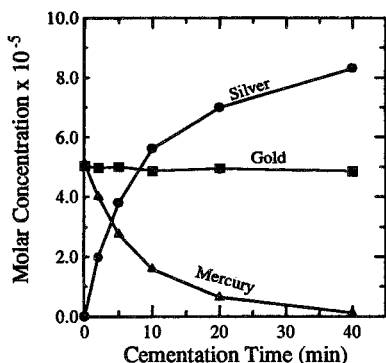


Figure 5. Mercury cementation with silver powder (particle size 6 to 26 microns, 1 liter solution, 100 times stoichiometric requirement).

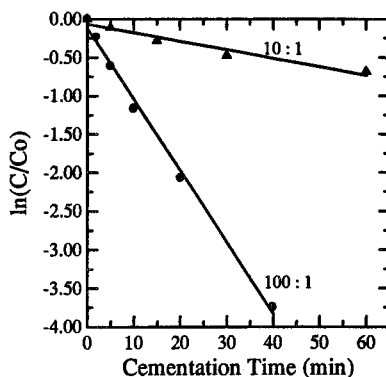


Figure 6. First order kinetic plots for mercury removal by suspended silver particles for two different stoichiometric levels.

SUMMARY

Various process alternatives have been considered to chemically control mercury during the cyanidation of gold ores. These alternatives include precipitation, adsorption, solvent extraction, ion exchange, and cementation.

In many cases, control can be accomplished by the selective reaction of mercury cyanide complexes with reduced sulfur compounds and/or functional groups. For example, precipitation with sulfides or thiol surfactants, adsorption by sulfur pretreated crumb rubber, and solvent extraction by thiol extractants. Gold does not react in these systems and remains stabilized in solution as the $\text{Au}(\text{CN})_2^-$ complex.

Also, results indicate that the selective extraction of gold from mercury in alkaline cyanide solution is possible by solvation extraction with alkyl phosphorus esters.

Finally, it has been shown that selective cementation of mercury from gold in alkaline cyanide solution can be achieved with metallic silver and certain other metals depending on the relative potentials of the half cell reactions.

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