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**A study of the thiosulfate leaching of gold from carbonaceous
ore and the quantitative determination of thiosulfate in the
leached solution**

Hemmati, Mahin, M.S.

University of Nevada, Reno, 1987

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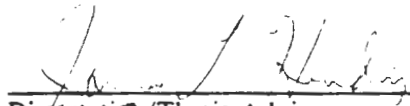
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in Metallurgical Engineering

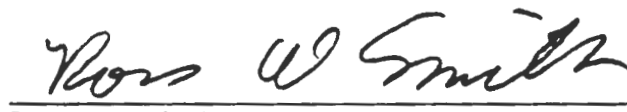
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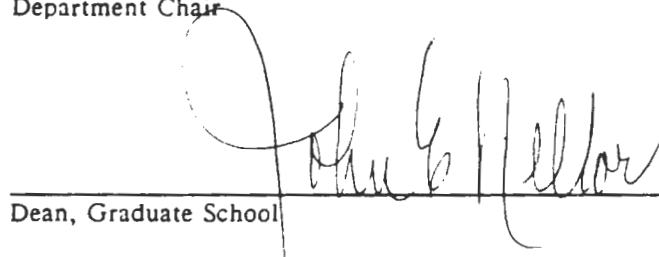
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April 1987

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ABSTRACT

The objective of this study was to investigate leaching of gold from carbonaceous ore using ammoniacal thiosulfate solutions, and to determine the consumption of thiosulfate during leaching. The quantitative determination of thiosulfate in the leached solution in the presence of sulfite and sulfate ions was accomplished by means of flow injection analysis with electrochemical detection. Lower thiosulfate consumption, which is an indication of solution stabilization, was achieved by addition of sulfite and maintenance of the solution at a pH of 10.5. Decomposition of thiosulfate was found to be greater at high temperatures. The optimum conditions for extraction of gold were found to be pH of 10.5, and temperature of 35°C. At these optimum conditions, 73 percent gold extraction was obtained.

It was also shown that thiosulfate will leach gold from oxide ores but not as effectively as cyanide. While a high percent extraction of gold was obtained from the carbonaceous ore, poor recovery of gold was observed from the sulfide ore, which is also a refractory ore as is the carbonaceous ore. A comparison of thiosulfate with cyanide leaching of gold from carbonaceous ore showed that thiosulfate is chemically better and economically more advantageous.

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INTRODUCTION

The extraction of gold from ores that contain carbon and organic compounds presents a problem to a cyanide mill. The presence of carbonaceous matter in gold ores causes poor release of gold from the carbonaceous matrix. Unfavorable gold extraction of these ores has been attributed, for the most part, to the adsorption of the gold cyanide complex on the carbonaceous matter, with subsequent loss to the tails [1]. Moreover, the use of cyanide solutions is difficult with respect to some ores, especially those containing copper and/or manganese, since these materials easily contaminate the cyanide solution and cause a poor recovery of the precious metals [2].

Ammonium thiosulfate has been proposed as an alternative for gold and silver extraction, and it has been investigated by many authors for recovering precious metals. Flett et al. [3] have studied thiosulfate leaching of silver using synthetic silver sulfide and have proposed the chemistry of leaching. Tozawa et al. [4] have studied thiosulfate leaching of gold using metallic gold and have determined the effects of several variables on leaching. The leach solutions for both of these investigations are similar to the system presented in this study. Extraction of gold and

silver from a copper ore was studied by Berezowsky [5] and was found to be feasible.

The present thesis describes laboratory results and work leading to use of a thiosulfate oxidation pressure leach system for the extraction of gold which is present in carbonaceous matter. The leach solution contains added ammonium sulfite and copper sulfate. Leaching proceeded without any pretreatment of the ore. Parameters considered were pH, temperature, time, oxygen partial pressure, and concentrations of thiosulfate, sulfite, and copper. Each one of these parameters was varied, keeping the rest constant, in order to study their individual effect on gold extraction and thiosulfate consumption.

There are several features of this method which are worth noting. Copper is a necessary reagent in solution since it acts as a catalyst in the extraction reaction. Another requirement is the maintenance of pH in the alkaline region of at least 10.0. Ammonium hydroxide is the preferred means for maintaining the desired pH. Available free ammonia not only accelerates the rate of reaction of the precious metals in the leach liquor, but also helps to stabilize the ammonium thiosulfate solution. The presence of oxygen or another suitable oxidant assists the

thiosulfate leaching process, although it is necessary that conditions do not favor the degradation of thiosulfate by oxidation. Sulfite is added to the solution in order to stabilize thiosulfate and prevent its decomposition.

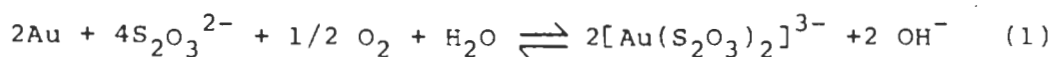
The most influential factors affecting the stability of thiosulfate solutions are the pH, the temperature, the solution concentration, the presence of atmospheric oxygen, and the exposure to light. Decomposition is reported to be catalyzed by copper(II) ions as well as by decomposition products [6]. The presence of sulfite ions in the leach solution is an essential factor. Sulfite ion is necessary to inhibit the decomposition of thiosulfate which, if permitted to occur, would result in precipitation of gold sulfide with resultant diminished recovery.

THEORY

Previous studies [3,4,5] reported significant data on the thiosulfate leaching of gold; however, they were conducted with synthetic and relatively pure systems or with elemental gold. It is clear that the chemistry of leaching of gold by thiosulfate solution from ores is complex and even more complex for carbonaceous ore. Decomposition of thiosulfate, side reactions of thiosulfate with other metals in the ore,

and adsorption of precious metals on carbonaceous matter in the ore are a few phenomena that occur in this system. Therefore, the effect of the variables and the optimum conditions in gold extraction under this system can differ substantially from a pure system. The principal reactions and mechanisms of gold extraction with thiosulfate, however, can be considered to be the same as with pure elemental gold.

Tozawa [7] reported that gold dissolves in thiosulfate solution in the presence of oxygen as follows:

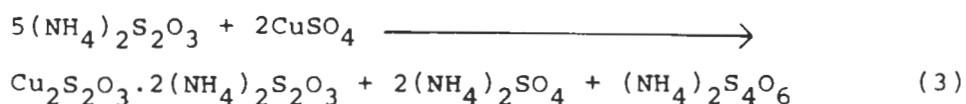


Cu(II) ion has a strong catalytic effect on the rate of the reaction. Studies on the leaching chemistry of thiosulfate by Flett [3] have shown the reactions that are involved in the leaching process. It is generally believed that the addition of copper sulfate to a thiosulfate solution results in the formation of cuprous thiosulfate which is soluble in excess thiosulfate and forms a cuprous thiosulfate.ammonium thiosulfate complex salt [8]. When such a solution is used to leach gold, considering the most soluble copper complex

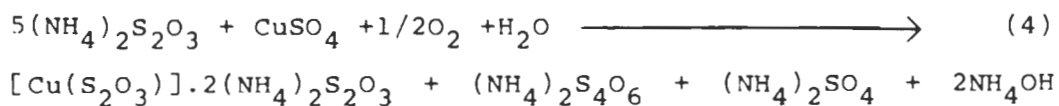
salt which is $\text{Cu}_2\text{S}_2\text{O}_3 \cdot 2(\text{NH}_4)_2\text{S}_2\text{O}_3$, the expected reaction is:



An overall representation of the chemistry of gold dissolution is given as follows [8]:



in which gold replaces copper and forms a gold thiosulfate complex $[\text{Au}_2\text{S}_2\text{O}_3 \cdot 2(\text{NH}_4)_2\text{S}_2\text{O}_3]$. In the presence of oxygen, however, this reaction is eventually superseded by an overall reaction stoichiometry that involves the cupric rather than cuprous state.

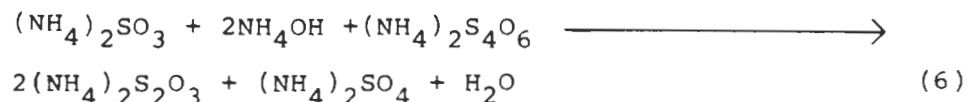


The presence of ammonium ion helps to stabilize the cupric state as either $\text{Cu}(\text{NH}_3)_4^{2+}$ or $\text{Cu}(\text{NH}_3)_5^{2+}$. Ammonium ion is necessary to stabilize the thiosulfate ion for the reaction with gold.

Under oxidizing conditions, oxidative degeneration of thio-sulfate could take place according to reaction (5) which is particularly likely to occur in the presence of copper [9].



Furthermore, the addition of ammonium sulfite to the solution, produces thiosulfate from tetrathionate by the reaction [9]:



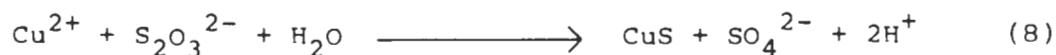
In the presence of sulfite, therefore, relatively little tetrathionate would form in the solution. Moreover, an equilibrium reaction occurs in the thiosulfate leach solution as presented by the following reaction [10].



It is clear that without the sulfite ion being present, the equilibrium would move to the left and produce sulfide which causes the precipitation of metal sulfides, such as gold sulfide. This reduces the gold in solution and also increases the thiosulfate consumption rate. Thus, the addition of sulfite to the solution would prevent the

thiosulfate disproportionation and in turn inhibit the sulfide precipitation.

At high temperatures, an undesirable amount of copper may be precipitated as a sulfide, due to decomposition of thiosulfate, with subsequent reduction of gold. The two possible reactions, which may affect the extraction, are considered here. One is the formation of cupric sulfide by the reaction between cupric ions and thiosulfate ions and the other is the oxidation of the cuprous thiosulfate complex ion [4]:



It is well known that an increase in temperature decreases the uptake of gold by steam-activated carbon. In fact, this is the basis for the high temperature elution of gold in the carbon-in-pulp process. However, adsorption studies conducted by Zailseva [12] indicate the opposite effect in the case of carbonaceous ores. This result suggests that chemical interaction plays a much more important role in the case of carbonaceous ores, compared with synthetic activated carbon.

QUANTITATIVE DETERMINATION OF THIOSULFATE

A common method for determination of thiosulfate ion is volumetric analysis [6,13]. Thiosulfate concentrations can be measured through their stiochiometric reduction of iodine in a buffered solution. However, if both sulfite and thiosulfate are present together, their iodometric determination becomes very difficult and time consuming. Ion chromatography has been a successful method compared to wet chemical methods and other instrumental techniques for the quantitative determination of aqueous solutions of sulfite, sulfate, and thiosulfate [14]. However, an alternative and a need for a reliable, fast, and less complex method was the reason why a novel Flow Injection Analysis(FIA) method was developed. The method allows for the accurate analysis of thiosulfate in the presence of both sulfite and sulfate ions.

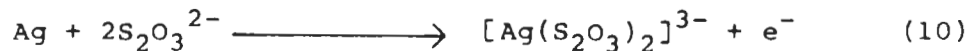
FIA is a relatively new analytical technique. The method can be used for any reaction whose product is detectable by standard analytical methods. FIA differs from traditional analytical methods in that measurements are not made at equilibrium. This method saves time traditionally used for mixing. It replaces beaker, pipets, and volumetric flasks with small open ended tubes through which one pumps the solutions [15]. The main parameters that should be adjusted

are dispersion, residence time, velocity of the carrier stream, and sampling frequency.

FIA is based on injection of a sample into a continuously moving, nonsegmented carrier stream, propelled by a peristaltic pump or by some other means. The injected sample forms a well defined zone which is then transported towards a detector by the carrier stream. The detection in this case is done by an amperometer, which measures the electrochemical current in response to a fixed electrode potential [16].

Performed at constant potential, the electrochemical method considered is based on the passage of current through the cell, which results from the tendency of the reacting species to transfer electrons. The flow-through amperometric cell(Dionex Corp.) consists of a silver working electrode, a platinum counter electrode, and a Ag/AgCl(1M NaCl) reference electrode which is separated from the flowing stream by an ion exchange membrane. The cell was potentiostated and the anodic currents, which are proportional to the concentration of thiosulfate, were measured by an Ion-chrom /Amperometric Detector(Dionex Corp.). The resulting signals were recorded on a strip chart recorder (Honeywell Electronic 195). The potential applied at the working silver electrode was 0.10V versus Ag/AgCl reference

electrode. The analytical procedure is based on the following reaction:



There were no interferences from either sulfite or sulfate ions, since no change in the oxidation current was seen when thiosulfate solutions containing up to 60 times excess of sulfate and sulfite were injected. The reason is that silver does not form stable complexes with either of these ions [17]. The method allows for up to 60 analyses per hour and has a detection limit (twice the baseline noise) of 10 ppb. This method has been found to be preferable to the other methods mentioned previously, since it is accurate, less complex, and fast.

EXPERIMENTAL

ORE MATERIAL

Principal studies were conducted on carbonaceous ore (pink ore) from the Freeport-McMoran Jerrit Canyon gold mine. The mine is located in the Independence mountain range in northeast Nevada, U.S.A.

The ore was crushed to minus 6 inch, using a 4 X 6 inch jaw crusher and 6 inch crushing rolls. The crushed ore was thoroughly blended by the cone and quarter technique. Head samples were taken by using a riffle type splitter box. The crushed ore was then pulverized to minus 100 mesh for the autoclave leaching tests. A seive analysis of this ore is given in Table 1. Since random sampling of the precious metal ore is very important, before each leach, the pulverized ore was mixed on a roller for about one half hour.

This ore is not amenable to cyanidation and is classified as a refractory ore. A sample of the ore was analyzed for its mineralogical form, grade of gold in ore, and carbon content. An Inductive Coupled Plasma(ICP) analysis was done by the United States Bureau of Mines(USBM). The optical minerology was done by Dr. Hsu of the Nevada Burea of Mines. This study showed that the ore consisted of fragments of dark, impure limestone with minor amounts of fine grained pyrite and local white calcite veinlets. The carbon analysis done also by the USBM showed the percentage of carbon to be 4.9, and the organic carbon content was 2.5 percent. The average grade of ore was assayed to be 0.43 oz/ton gold.

Besides the Freeport pink ore, a few other ores were also examined by autoclave leaching; they are described below.

Table 1- Screen analysis of the carbonaceous ore:

<u>mesh size</u>	<u>percent by weight</u>
-100 to +140	14.3
-140 to +200	23.7
-200 to +325	4.5
-325	57.5

Oxide Ore:

This ore is also from the Freeport operation. The oxide ore is readily amenable to cyanidation and it is not classified as a refractory ore. The optical mineralogy study indicated that the ore consisted of fragments of dark silicified rock (mostly fine grained quartz) with local vuggy quartz veinlets (white). No visible sulfide minerals were observed. Also some fragments of dark, impure limestone with mixed local white calcite veinlets were found. The gold content of this ore is 0.25 oz/ton.

Gooseberry Silver Ore:

This ore is from the Gooseberry mine operation which is located in Storey County, Nevada, U.S.A. The optical mineralogy of the ore showed that it consisted of mainly quartz and calcite with minor to trace amounts of feldspar. The ore was assayed to be 0.16 oz/ton gold, and 6.2 oz/ton Ag.

Duval Sulfide Ore:

The Duval gold and silver milling operation is located near Battle Mountain, Nevada, U.S.A. The mineralogical study of the ore showed it consisted of calcite, chlorite, dolomite, feldspar, magnetite, quartz, pyrite, and galena. The gold and silver contents of the ore were respectively 0.21 oz/ton and 1.3 oz/ton.

Pinson Oxide Ore:

Pinson mining company is located near Battle Mountain, Nevada, U.S.A. The mineralogical study of this ore indicated that it consisted of kaglinite, quartz, and felspar. The gold and silver content of this ore were 0.06 oz/ton gold and 0.10 oz/ton silver.

AUTOCLAVE LEACHING

Experiments were carried out in a 500cm³ stainless steel autoclave (Autoclave Engineers, Inc.). It consisted of a stirring shaft with maximum diameter impeller of 3.18 cm, cooling coil, and thermocouple all connected through the bolted lid. The autoclave was heated with an electric heating jacket. Heating was controlled by an electric controller, but the flow of cooling water was controlled

manually to balance the heating from the reaction and maintain a constant temperature. The autoclave was preheated to nearly the desired temperature, then it was charged with the prepared leach solution and a measured quantity of ore. The lid was tightened, and agitation was started. The autoclave was purged with nitrogen and then the desired oxygen pressure was applied and maintained. Operating temperatures ranged from 25 to 85°C, while the oxygen partial pressure was generally 103 kpa. The pH of the solution was measured before and after leaching.

When the leaching was completed, the leach and slurry proceeded to a liquid-solid separation step. Samples of the filtrate were stored in amber nalgene bottles at about 4°C for periods that ranged from one to five weeks. Because of this, some sample decomposition may have occurred and thiosulfate concentrations are probably accurate to within five percent. These samples were analyzed for the concentration of thiosulfate by FIA with electrochemical detection. The resultant residue from the filtration was dried and analyzed for gold content. The technique used in this study to analyze gold was a combination of fire assay and atomic absorption. A proper flux was prepared to fire assay the leached ore and head sample. The dore from this was then dissolved in HNO₃ and HCl solution to detect the gold content by atomic absorption (Perkin-Elmer model

2380). Reagent grade ammonium thiosulfate, ammonium hydroxide, copper sulfate, ammonium sulfite, and distilled water were used to prepare the solutions. The purity of the thiosulfate samples used were determined by FIA. In tables 2 and 3 errors corresponding to duplicate experiments under the same conditions for percent gold extraction and thio-sulfate consumption are tabulated.

DETERMINATION OF THIOSULFATE

FIA applied together with an electrochemical detector was used for determination of thiosulfate in the presence of sulfite and sulfate ions. The system manifold is illustrated in Figure 1. It is constructed from a FIAstar 5020 analyzer (Tecator Inc.), equipped with two peristaltic pumps and a variable volume injection. For most of the analyses a 100 μ l sample loop was used and the injection time was 10s. A FIAstar chemifold-I was used and all connections were made with 0.5mm i.d. microline tubing (Thermoplastics Scientifics). This system was applied together with the electrochemical detector to detect the concentration of thiosulfate in a solution. The electrochemical detection method that was used in these experiments is explained on page 8 of this thesis.

Table 2- Accuracy of thiosulfate consumption based on
duplicate experiments:

	$(S_2O_3^{2-})_I$ (M)	$(S_2O_3^{2-})_F$ (M)	$(S_2O_3^{2-})$ Consumed (M)	$(S_2O_3^{2-})$ Consumed (%)	mean St.Dv. of (M) consumed	mean St.Dv. of (%) consumed
1-	1.19	1.006	0.184	15.5		
	1.19	1.022	0.168	14.1		
2-	0.712	0.635	0.077	10.8		
	0.712	0.663	0.049	6.9		
3-	0.712	0.589	0.123	17.3		
	0.712	0.574	0.138	19.4	9.8E-3	1.2

Table 3- Accuracy of gold extraction based on duplicate
experiments:

	$(Au)_I$ oz/ton	$(Au)_F$ oz/ton	percent Au extraction	St.Dv.	mean St.Dev.
1-	0.439	0.208	52.6		
	0.439	0.224	49.0	1.8	
2-	0.423	0.139	67.1		
	0.411	0.121	70.6	1.8	
3-	0.450	0.121	73.1		
	0.426	0.123	71.1	1.0	1.5

The carrier stream consisted of 0.01M KNO_3 and 0.01M Na_2CO_3 solutions with a flow rate of 1.0 ml/min. In order to obtain a linear calibration of the system, standard samples of 2, 3, 4, and 5ppm thiosulfate were injected. Samples of the leach solution were diluted to the proper concentration to be in the detection range. They were also buffered with Na_2CO_3 to adjust pH. The samples were injected in duplicate and concentration results were obtained based on the comparison of the peak heights of samples and standards. Some initial samples were also examined to obtain the purity of the thiosulfate initial concentrations.

The standard samples were injected between the samples to obtain reproducibility of the data. The results are shown in table 4, and the calibration curve is shown in Figure 2. These data show that error involved in FIA measurements is very small, and data are reproducible. Therefore, errors involved in sample measurements are due to sample storage and not to the analytical technique.

Table 4- Accuracy of FIA in thiosulfate measurements based on standard solutions:

$\text{S}_2\text{O}_3^{2-}$ (ppm)	peak height	mean	St.Dev.
2	169,169,171	169.7	1.15
3	219,219,219	219	0
4	249,249,250	249.3	0.58
5	267,267,268	267.3	0.58

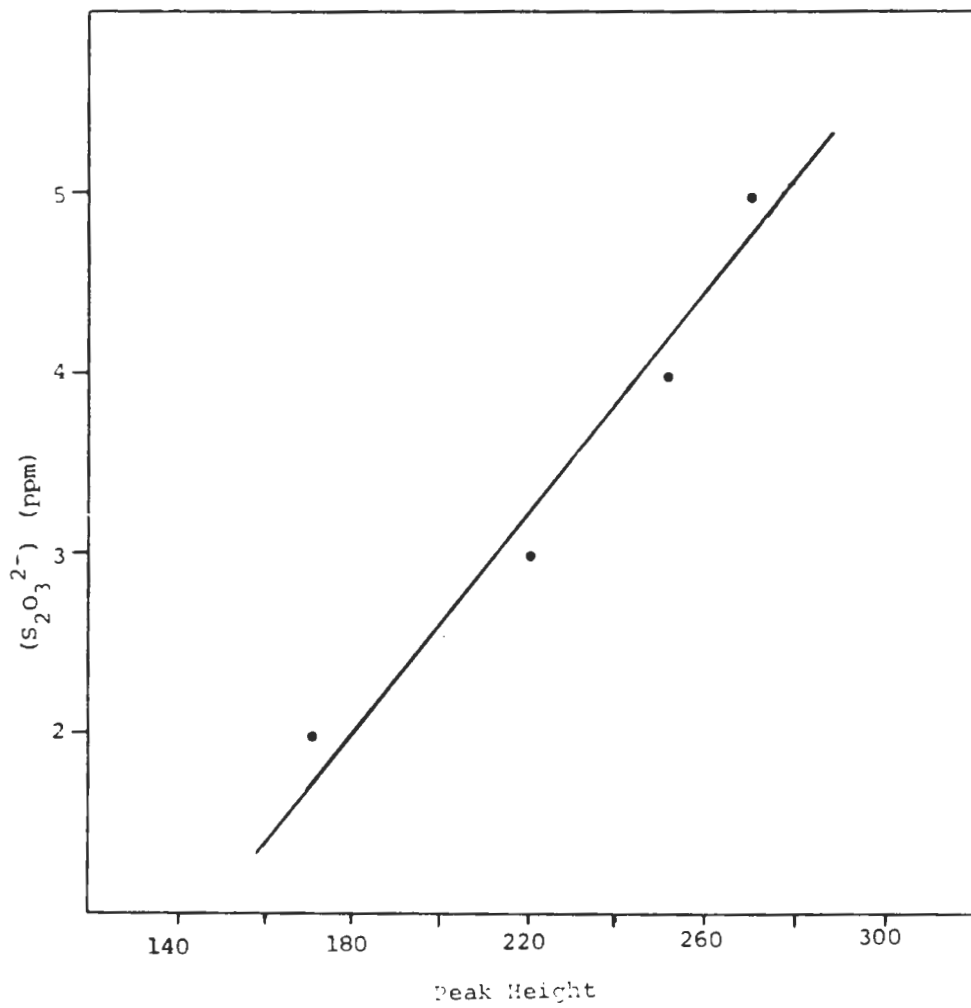


Figure 1- Calibration Curve for Thiosulfate Measurements

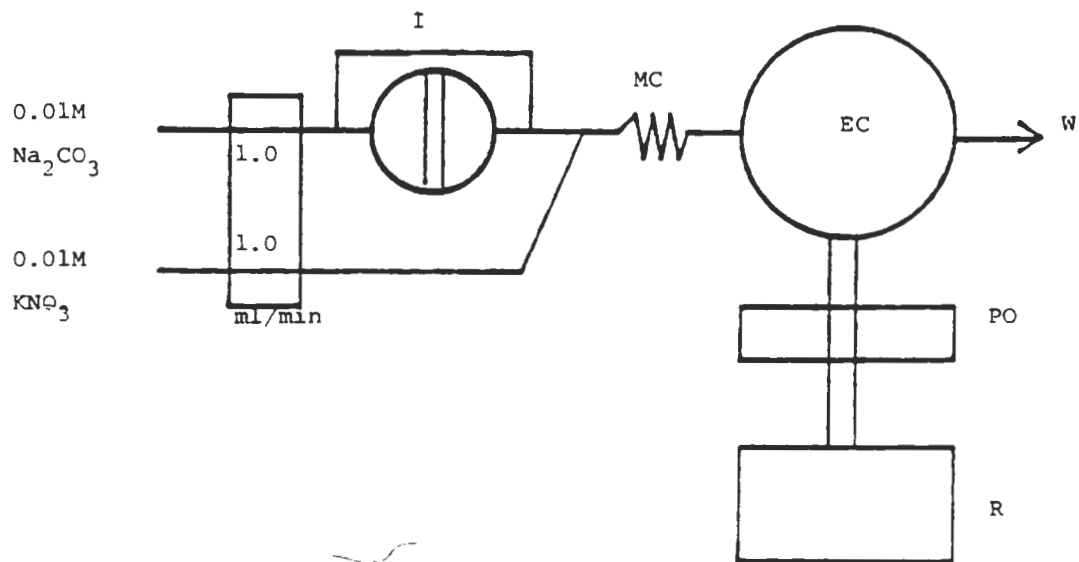


Figure 2 - A Schematic Diagram of the Flow Injection Analyzer
and the Electrochemical Detector

I = Injector EC = Electrochemical Detector

R = Recorder PO = Potentiostat

MC = Mixing Coil , Id = 0.5mm , L = 30cm

EXPERIMENTAL RESULTS

EFFECT OF pH

The effect of pH on the gold extraction and thiosulfate consumption has been examined by varying the ammonia concentration of the leach solution. All the leaching experiments were done on carbonaceous ore (pink ore) unless otherwise stated. The pH was found to be an important factor in gold extraction and thiosulfate consumption. The pH varied from 8.5 to 10.5 which corresponded to ammonia concentrations of 0.03M to 4.5M respectively. Due to formation of a buffer solution, a higher ammonia concentration did not change the pH appreciably. As is shown in Figure 4, ammonia concentrations of 3.0M and 4.5M which both corresponded to pH of 10.5 resulted in the same level of gold extraction and thiosulfate consumption. The experimental results are shown in figures 3 and 4. It should be noted that although the experimental conditions for the results in these two figures were different, they both have the same general behavior. As observed from these graphs, there is a great improvement in gold extraction with increasing pH. Figure 4 shows that recovery has been increased up to 58 percent at pH 10.2 from 6 percent at pH 9.0. The optimum pH for extraction was found to be 10.5. According to these graphs, thiosulfate consumption also

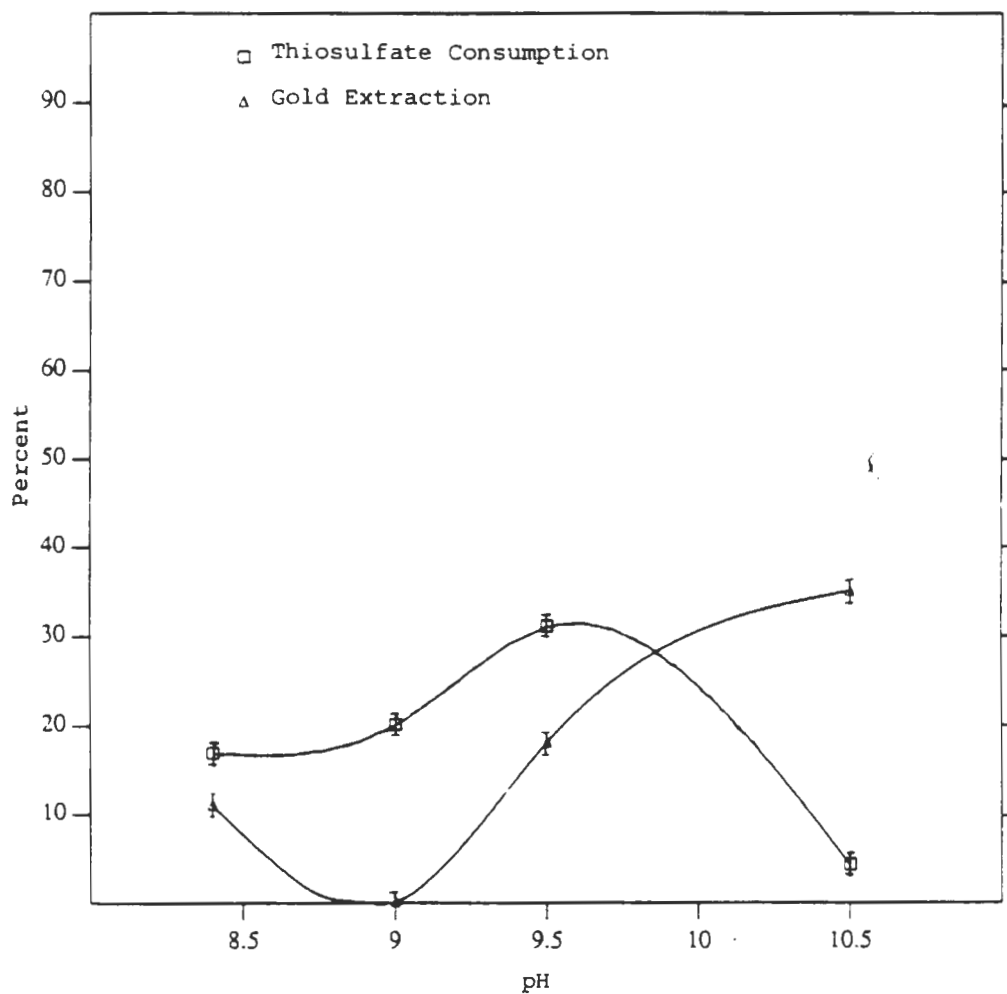


Figure 3- Effect of pH on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 65°C;
Stirring Velocity = 33.3cm/s; $[S_2O_3^{2-}] = 0.48M$;
 $[SO_3^{2-}] = 0.10M$; $[Cu(II)] = 0.04M$

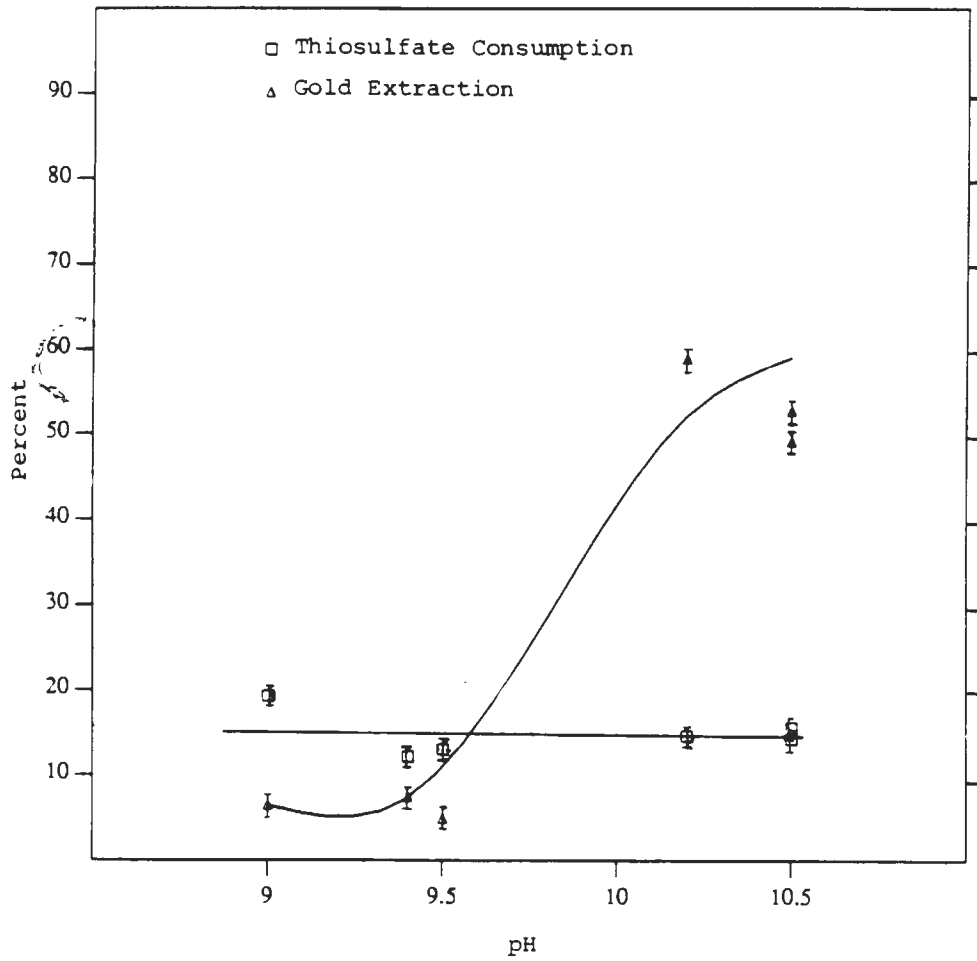


Figure 4- Effect of pH on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 55°C;
 Stirring Velocity = 33.3cm/s; $[S_2O_3^{2-}] = 1.19M$;
 $[SO_3^{2-}] = 0.22M$; $[Cu(II)] = 0.06M$

depended on the pH. At higher pH values slightly less thiosulfate consumption was observed.

EFFECT OF TEMPERATURE

Temperature was found to be an important parameter in gold extraction. Experiments were conducted at the optimum pH of 10.5 and under the following conditions: oxygen pressure = 103 kpa, time = 2hr, stirring velocity = 33.3cm/s, $[\text{NH}_3] = 3.0\text{M}$, $[\text{Cu(II)}] = 0.15\text{M}$, $[\text{SO}_3^{2-}] = 0.224\text{M}$, and $[\text{S}_2\text{O}_3^{2-}] = 0.712\text{M}$. The temperature ranged from 25 to 85°C. The experimental results are shown in Figure 5. The extraction curve has two maxima and one minimum value, the two maxima being at 35 and 75°C and the minimum at 65°C. The optimum temperature for gold extraction for this ore was at 35°C.

Extraction increased from 25 to 35°C, then it gradually decreased reaching a minimum at 65°C; from this point it increased slightly at 75°C. Extraction was 71 percent at 35°C, 33 percent at 65°C, and 45 percent at 75°C. Thio-sulfate consumption, in the range of 25 to 55°C and 65 to 85°C, increased constantly even though the recovery was declining. With a drop in extraction at 65°C, thiosulfate consumption also dropped. Based on these results, thio-sulfate consumption was indicated to be dependent on the

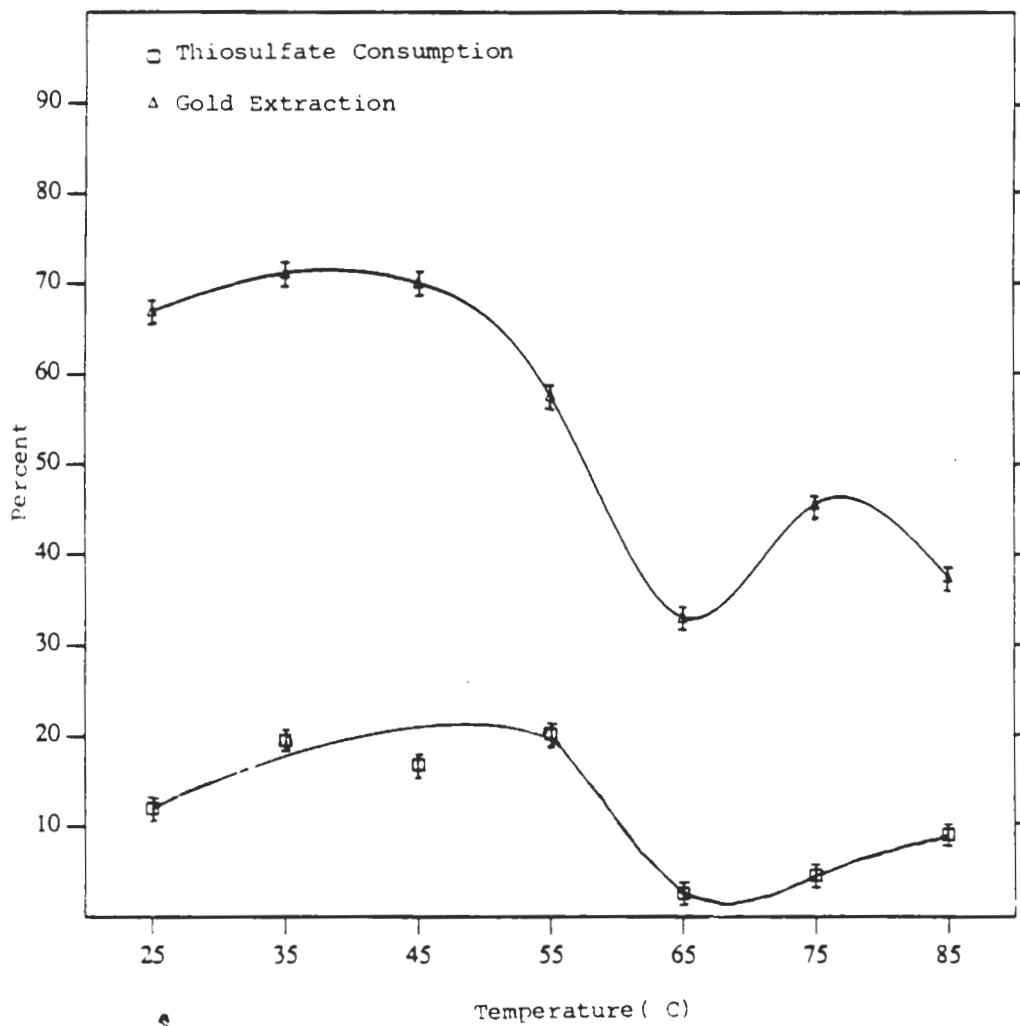


Figure 5- Effect of Temperature on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; Stirring Velocity = 33.3cm/s; $[S_2O_3^{2-}] = 0.71M$; $[SO_3^{2-}] = 0.22M$; $[Cu(II)] = 0.15M$; $[NH_3] = 3.00M$ (pH=10.5)

temperature. The general trend being an increase in consumption with increasing the temperature.

EFFECT OF AMMONIUM THIOSULFATE CONCENTRATION

The relationships between gold extraction and thiosulfate concentration, and that between the latter and thiosulfate consumption have been examined. The experimental conditions were: oxygen pressure = 103 kpa, time = 2hr, temperature = 55°C, stirring velocity = 33.3cm/s, $[\text{NH}_3] = 1.5\text{M}$ at pH of 10.0, $[\text{Cu(II)}] = 0.15\text{M}$, $[\text{SO}_3^{2-}] = 0.224\text{M}$. The experimental results are shown in figure 6. Using these conditions, increasing the thiosulfate concentration from 0.45M to 0.71M increased the extraction from 27 to 50 percent. Increasing the concentration to 1.2M, however, depressed the extraction to 41 percent, gold extraction was highest at 0.71M. Thiosulfate consumption is plotted in mole/l. Considering the experimental error, there is slight variation in consumption with increasing the thiosulfate concentration. Similar results were obtained at a different condition as is seen in Figure 7.

Considering that the optimum temperature was approximately 35°C, a series of experiments was also performed at this temperature and a pH of 10.5 with varying thiosulfate

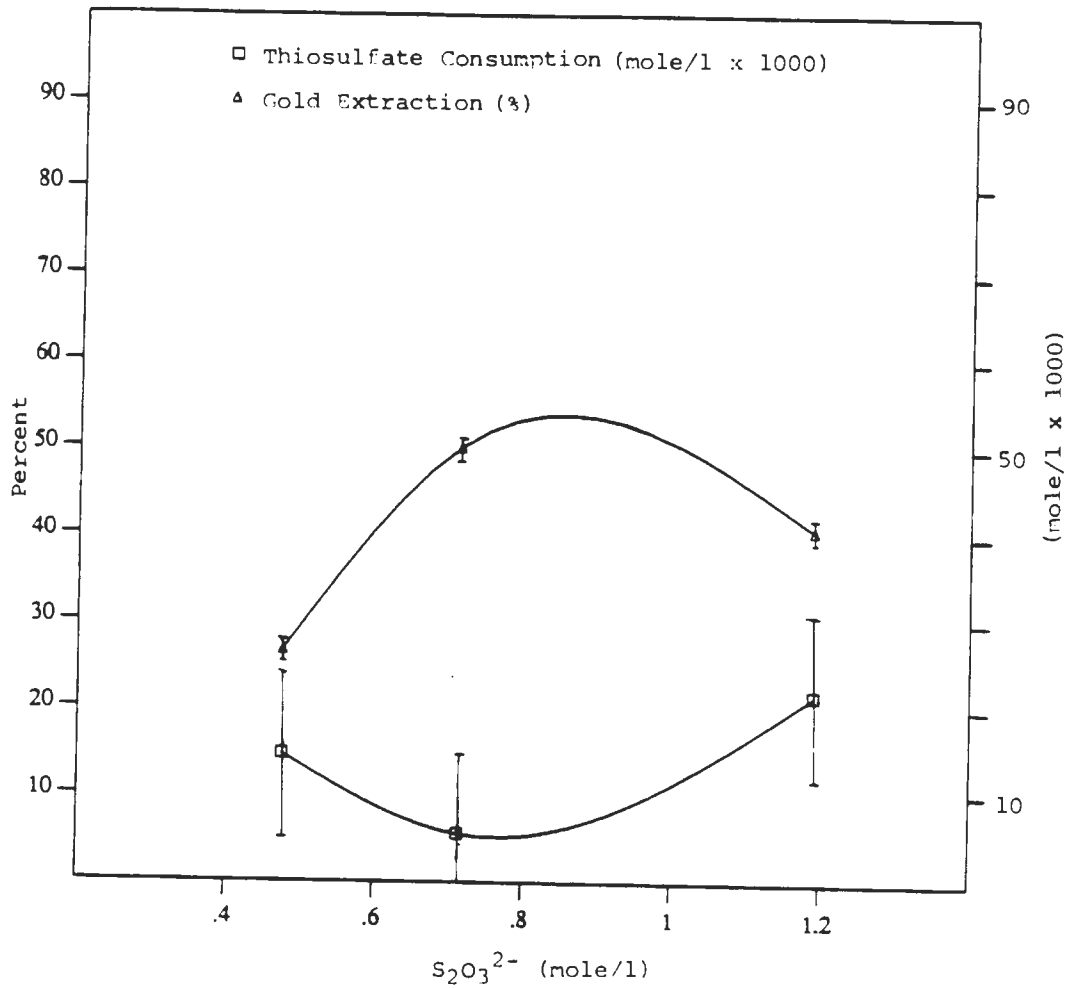


Figure 6- Effect of Ammonium Thiosulfate Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 55°C;
 Stirring Velocity = 33.3cm/s; $[SO_3^{2-}] = 0.22M$;
 $[Cu(II)] = 0.15M$; $[NH_3] = 1.5M$ (pH=10.0)

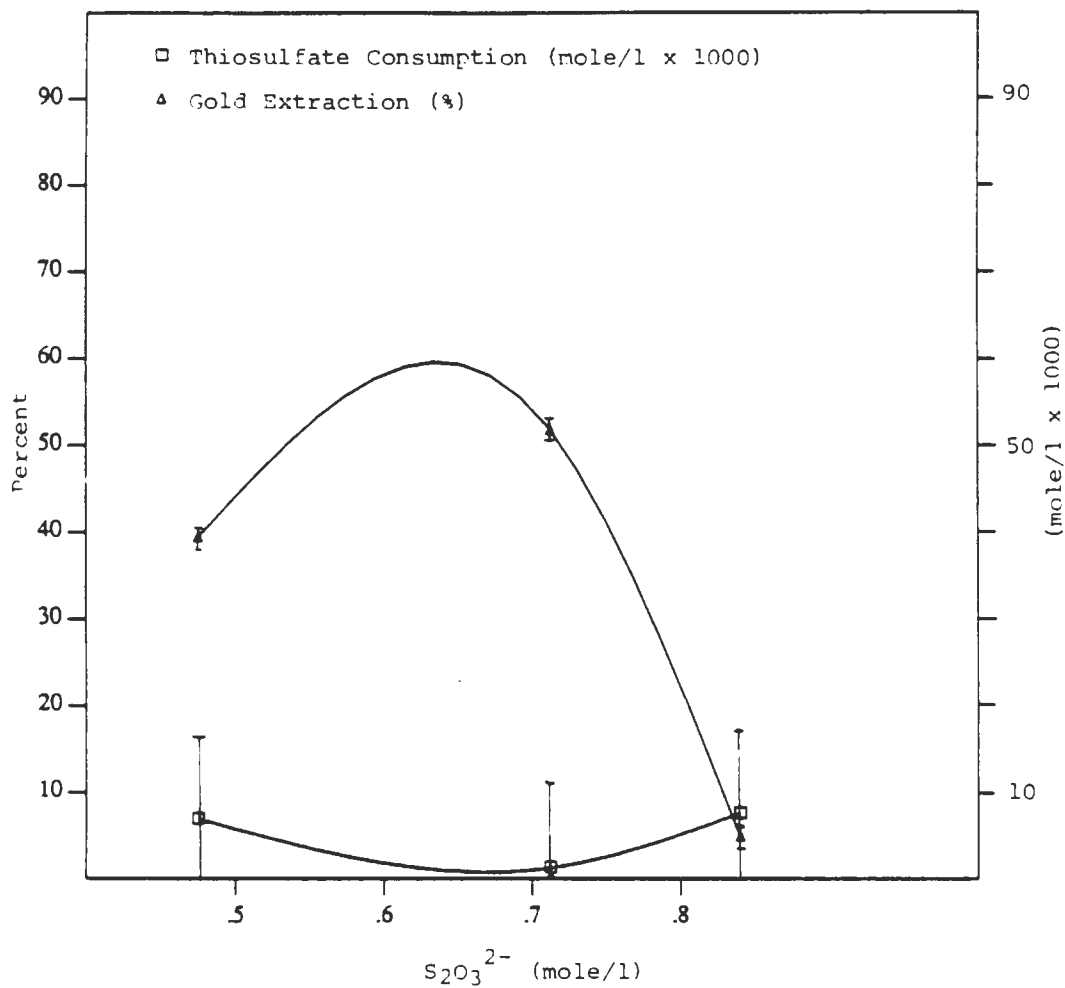


Figure 7- Effect of Ammonium Thiosulfate Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 55°C;
 Stirring Velocity = 33.3cm/s; $[SO_3^{2-}] = 0.10M$;
 $[Cu(II)] = 0.04M$; $[NH_3] = 0.3M(pH=9.5)$

concentration. The concentration of thiosulfate ranged from 0.09 to 0.88M. The results are shown in Figure 8. The experimental conditions were: oxygen pressure = 103 kpa, time = 2hr, stirring velocity = 33.3cm/s; $[\text{NH}_3] = 3.0\text{M}$ at pH of 10.5, $[\text{SO}_3^{2-}] = 0.10\text{M}$, $[\text{Cu(II)}] = 0.05\text{M}$.

It is seen from Figure 8 that gold extraction increased slightly with increasing thiosulfate concentration. At the low concentration of 0.088M thiosulfate, extraction of 64 percent was obtained, which was improved to 74 percent at 0.88M. The consumption curve, again is similar to Figures 6 and 7. Within the experimental error, the trend is a slight increase in actual thiosulfate consumption with an increase in thiosulfate concentration. Results of Figure 8 are plotted as percent thiosulfate consumption versus its concentration. This is shown in Figure 9. The percent consumption decreases with an increase in thiosulfate concentration. This is because the concentration increase is larger than the consumption increase.

EFFECT OF SULFITE CONCENTRATION

Ammonium sulfite is added to the leach solution to stabilize thiosulfate and prevent sulfide precipitation. Small quantities of sulfite are needed for the stability of thiosulfate. The effect of sulfite addition has been examined over

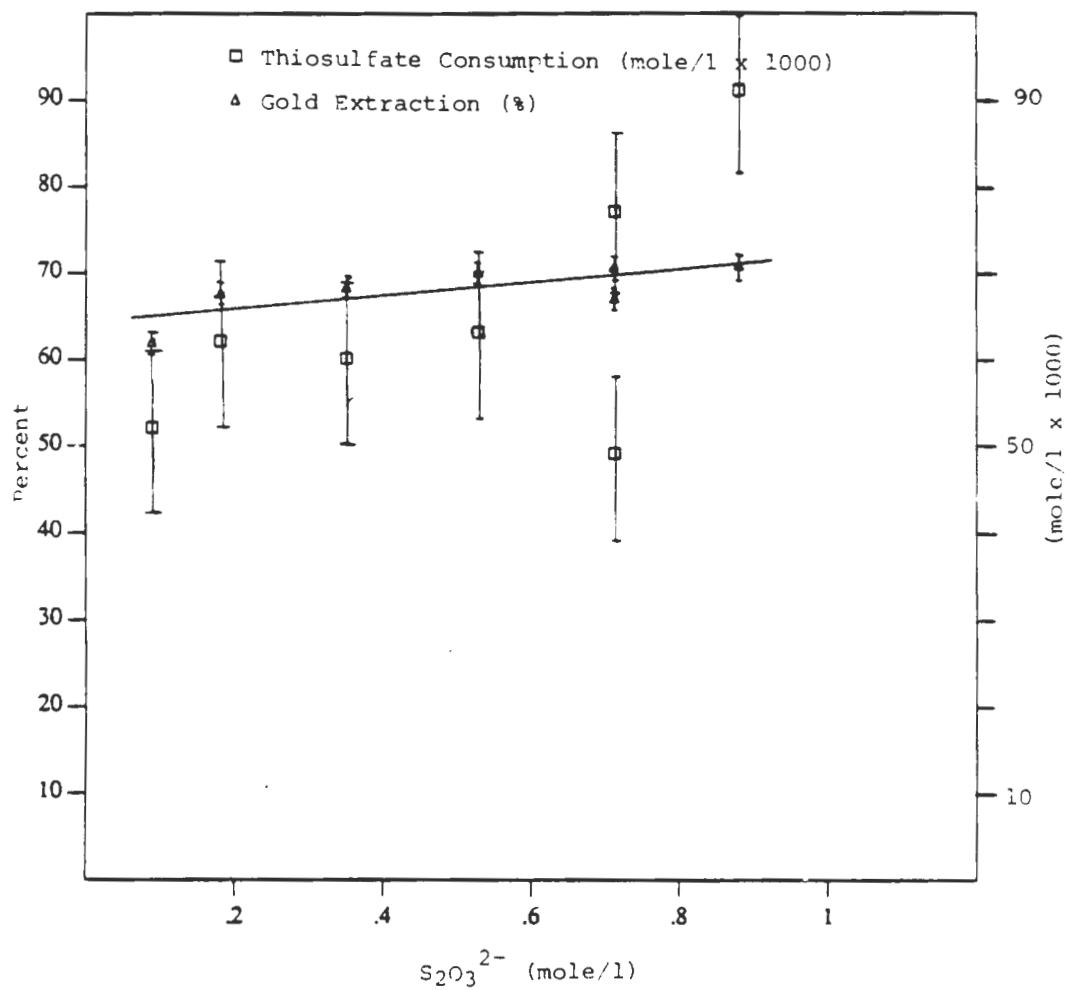


Figure 8- Effect of Ammonium Thiosulfate Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 35°C;
 Stirring Velocity = 33.3cm/s; $[SO_3^{2-}] = 0.10M$;
 $[Cu(II)] = 0.05M$; $[NH_3] = 3.0M$ (pH=10.5)

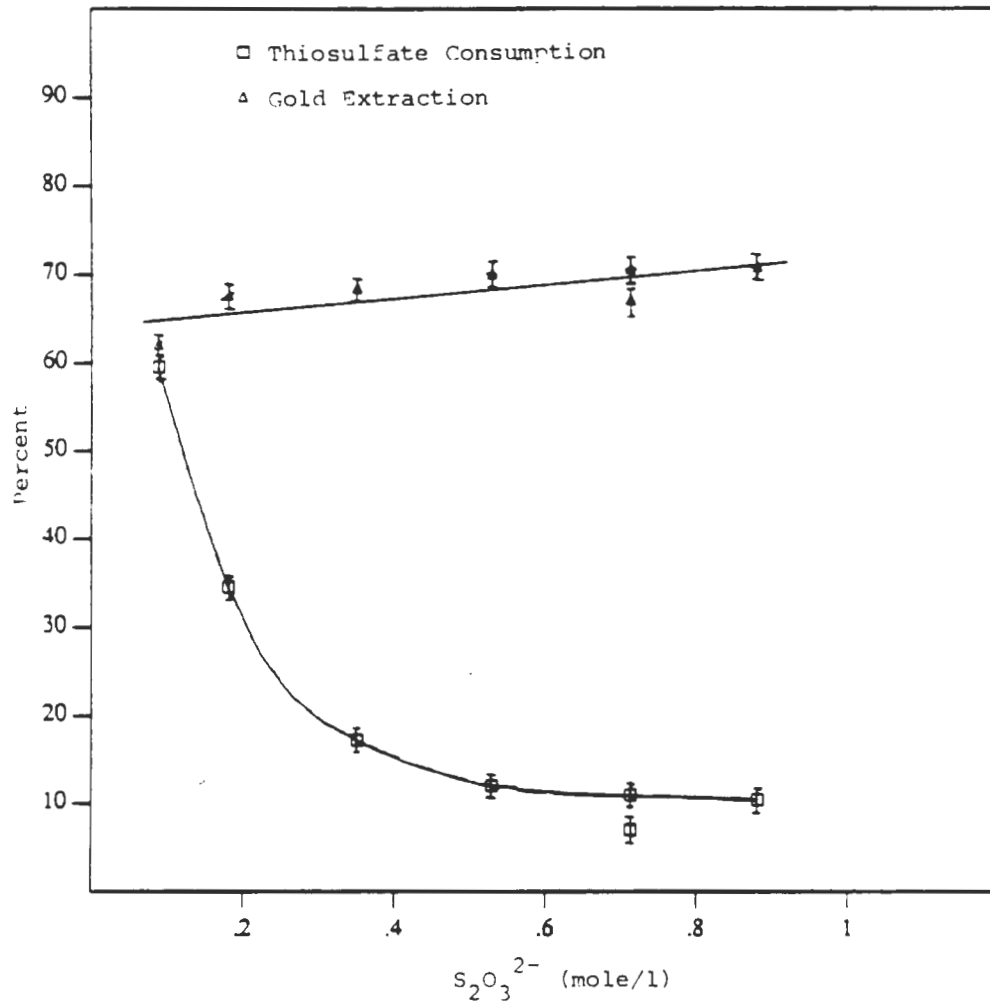


Figure 9- Effect of Ammonium Thiosulfate Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 35°C;
 Stirring Velocity = 33.3cm/s; $[SO_3^{2-}] = 0.10M$;
 $[Cu(II)] = 0.05M$; $[NH_3] = 3.0M$ (pH=10.5)

a range of 0.13M to 0.80M at pH 10.2, and thiosulfate concentration of 0.71M. Other conditions were: oxygen gage pressure = 103 kpa, time = 2hr, temperature = 55°C, stirring velocity = 33.3cm/s, [Cu(II)] = 0.04M, and [NH₃] = 1.5M.

The experimental results are shown in Figure 10. Under these conditions, the thiosulfate consumption decreased with increasing sulfite. Gold extraction increased up to 0.4M sulfite then gradually decreased. In order to further investigate the effect of sulfite, a series of experiments were performed at the optimum temperature of 35°C, pH of 10.5, and copper concentration of 0.15M, with the other conditions being the same. Sulfite concentrations ranged from zero up to 0.6M. The results are shown in Figure 11. Under these experimental conditions, sulfite did not have a significant effect on gold extraction. Thiosulfate consumption, however, decreased with increasing sulfite concentration. This is in accord with the previous results shown in Figure 10.

EFFECT OF COPPER CONCENTRATION

The effect of Cu(II) concentration was examined over a range of 0.04M to 0.15M. The experimental conditions were as follows: oxygen pressure = 103 kpa, time = 2hr, temperature = 55°C, stirring velocity = 33.3cm/s, [NH₃] = 1.5M at pH of

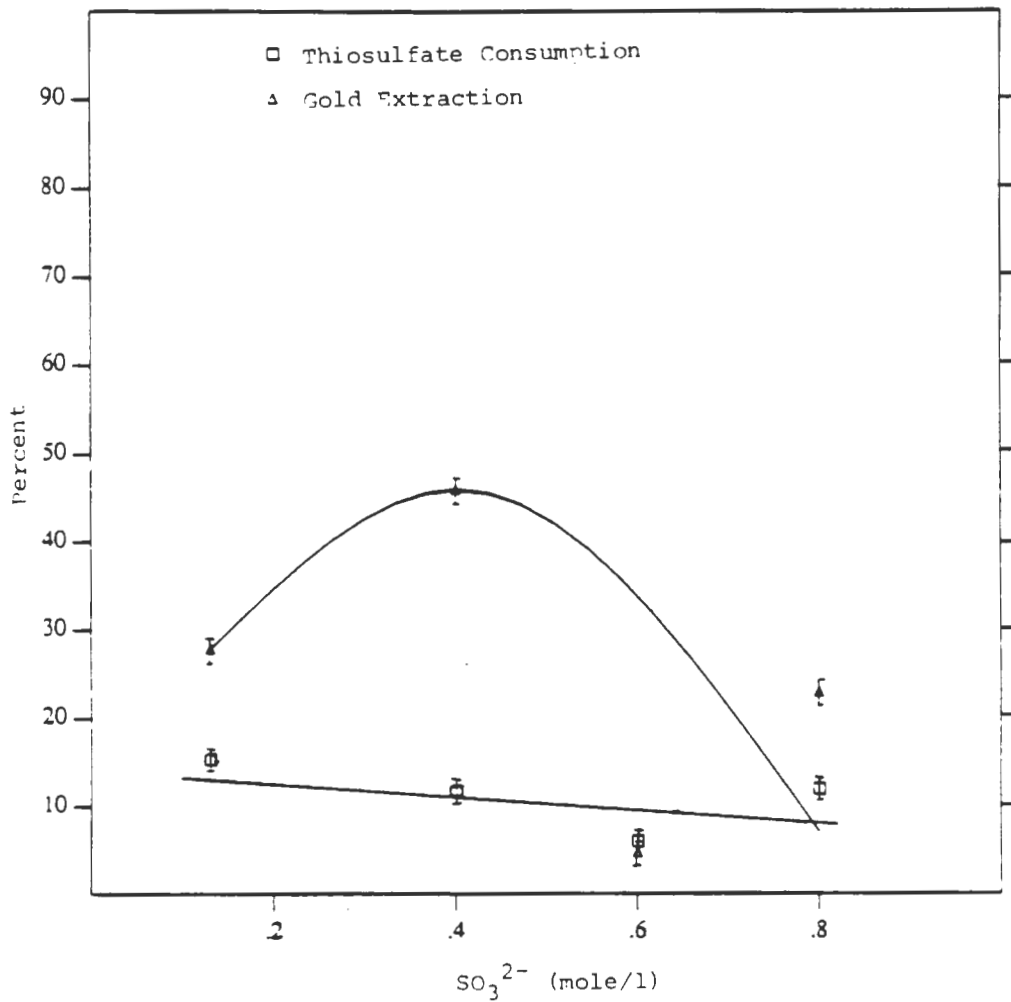
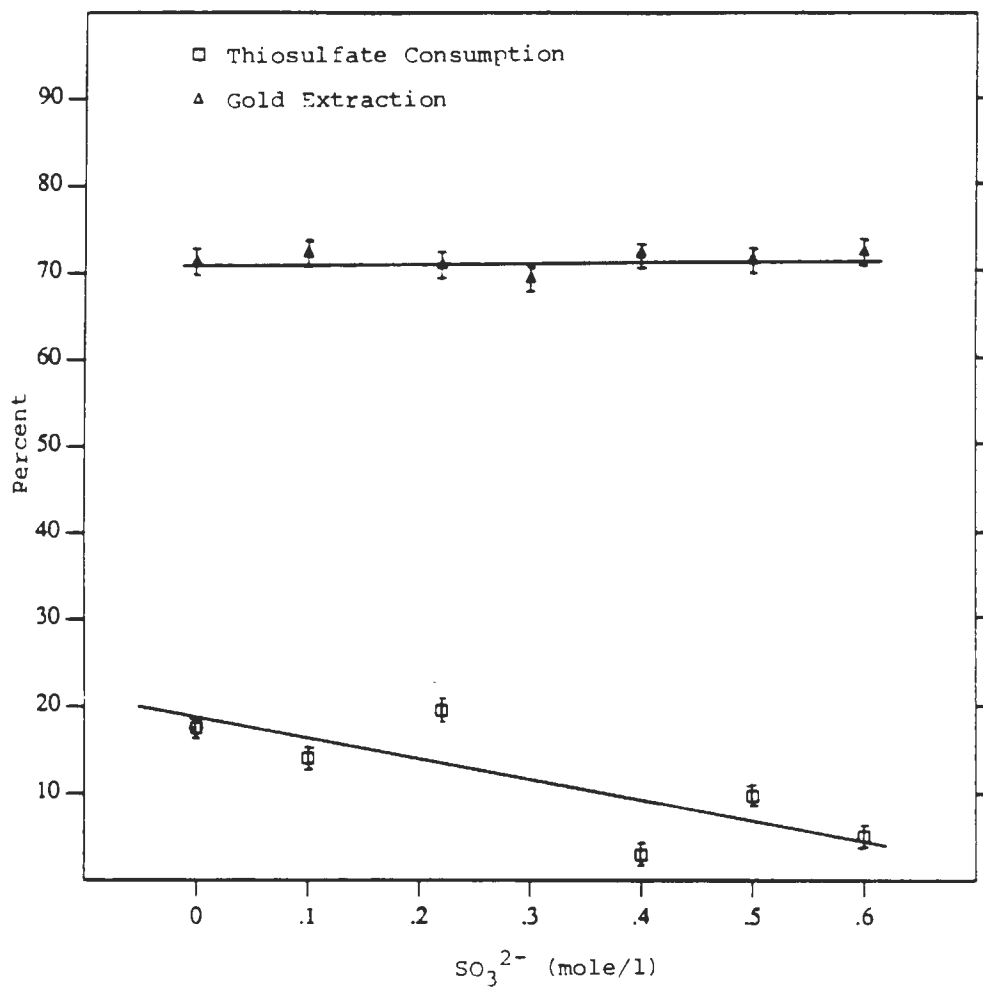


Figure 10- Effect of Ammonium Sulfite Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 55°C;
Stirring Velocity = 33.3cm/s; $[\text{S}_2\text{O}_3^{2-}] = 0.71\text{M}$;
 $[\text{Cu(II)}] = 0.04\text{M}$; $[\text{NH}_3] = 1.5\text{M}(\text{pH}=10.0)$



aFigure 11- Effect of Ammonium Sulfite Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 35°C;
 Stirring Velocity = 33.3cm/s; $[\text{S}_2\text{O}_3^{2-}] = 0.71\text{M}$;
 $[\text{Cu(II)}] = 0.15\text{M}$; $[\text{NH}_3] = 3.0\text{M}$ (pH=10.5)

10.2, $[S_2O_3^{2-}] = 0.712M$, $[SO_3^{2-}] = 0.40M$. The experimental results are shown in Figure 12. Gold extraction decreased from 46 percent at 0.04M to 33 percent at 0.10M, but again improved to 51 percent at 0.15M. In order to further investigate the effect of copper concentration, experiments were carried out under different conditions. These were: temperature = 35°C, pH = 10.5, oxygen pressure = 15psi, time = 2hr, stirring velocity = 33.3cm/s, $[S_2O_3^{2-}] = 0.712M$, $[SO_3^{2-}] = 0.224M$. The experimental results are shown in Figure 13. Copper concentrations ranged from 0.05 to 0.20M. It was found that variation in gold extraction were not significant. The thiosulfate consumption curve paralleled the extraction pattern, and there is no major change in consumption with changing the copper concentration.

EFFECT OF TIME

The effect of time on the extraction of gold and the consumption of thiosulfate was examined over a range of 0.5 to 4 hours. The experimental results are shown in Figure 14. The experimental conditions were as follows: oxygen pressure = 103 kpa, temperature = 35°C, stirring velocity = 33.3cm/s, $[NH_3] = 3.0M$ at pH of 10.5, $[S_2O_3^{2-}] = 0.712M$, $[SO_3^{2-}] = 0.224M$, $[Cu(II)] = 0.15M$.

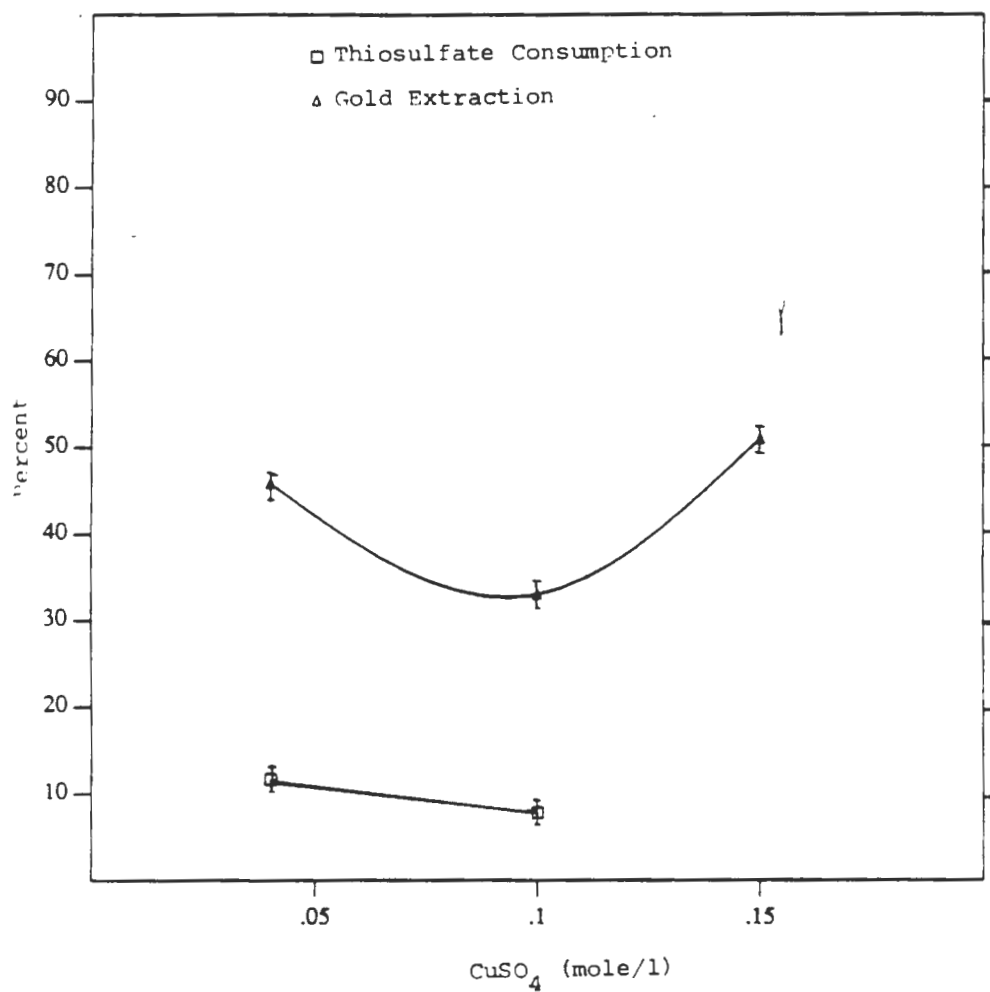


Figure 12- -Effect of Copper Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 55°C;
Stirring Velocity = 33.3cm/s; $[\text{S}_2\text{O}_3^{2-}] = 0.71\text{M}$;
 $[\text{SO}_3^{2-}] = 0.40\text{M}$; $[\text{NH}_3] = 1.5\text{M}$ (pH=10.0)

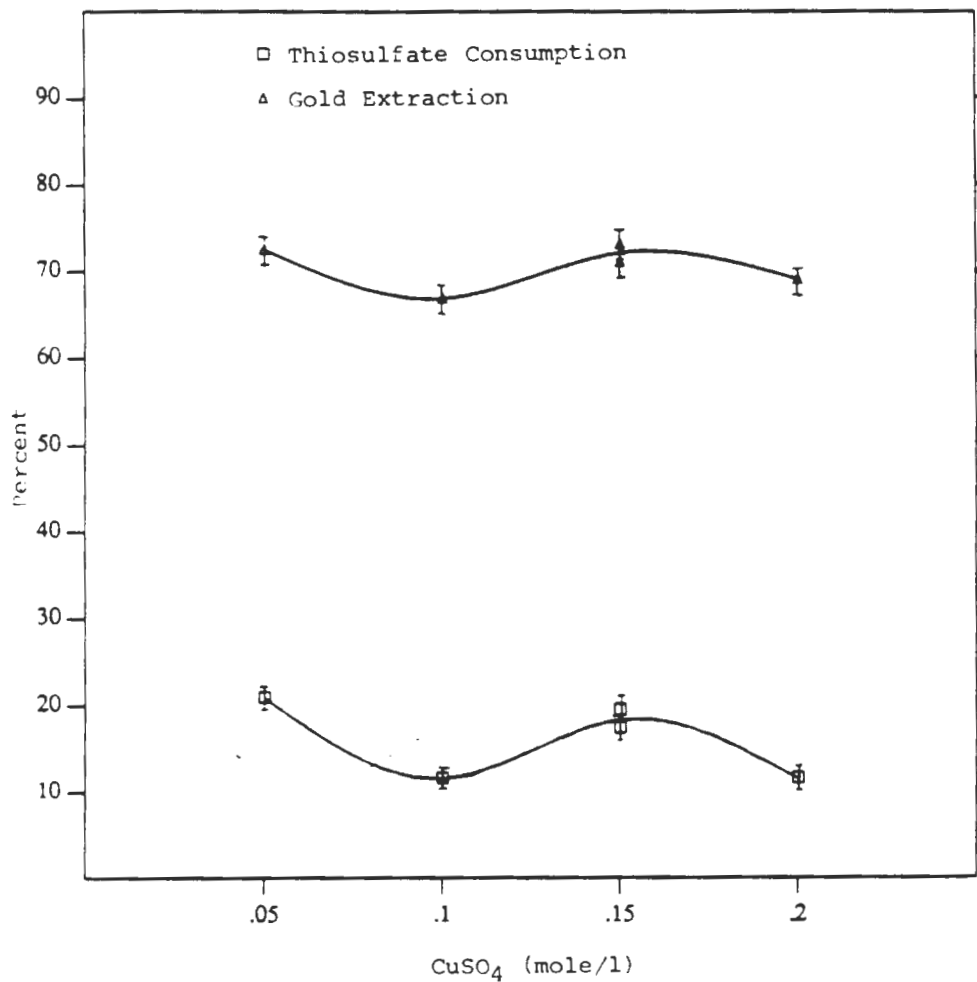


Figure 13- Effect of copper Concentration on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; time = 2hrs; temperature = 35°C;
Stirring Velocity = 33.3cm/s; [S₂O₃²⁻] = 0.71M;
[SO₃²⁻] = 0.22M; [NH₃] = 3.0M(pH=10.5)

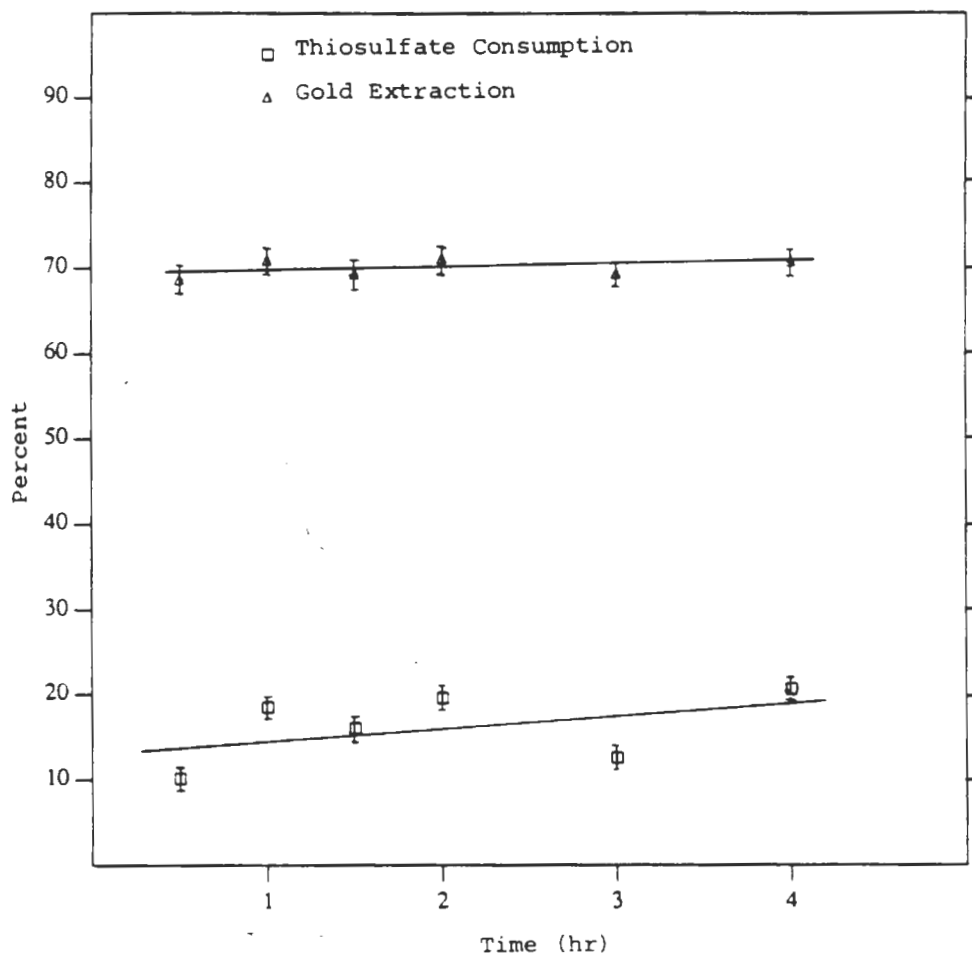


Figure 14- Effect of Time on the Gold Extraction and the Thiosulfate Consumption

Oxygen = 103kpag; temperature = 55°C; Stirring Velocity = 33.3cm/s; $[S_2O_3^{2-}] = 0.71M$; $[SO_3^{2-}] = 0.22M$; $[Cu(II)] = 0.15M$; $NH_3 = 3.0M$ (pH=10.5)

The reaction was determined to be fast and reached completion after one half hour. The thiosulfate consumption, however, increased slightly even though there was no further increase in gold extraction. This can be due to decomposition of thiosulfate during the extra time.

EFFECT OF OXYGEN PRESSURE

The effect of oxygen pressure was determined experimentally. The pressure of oxygen ranged from atmospheric to 206 kpa. The experimental conditions were as follows: time = 2hr, temperature = 35°C, stirring velocity = 33.3cm/s, $[\text{NH}_3] = 3.0\text{M}$ at pH of 10.5, $[\text{S}_2\text{O}_3^{2-}] = 0.712\text{M}$, $[\text{SO}_3^{2-}] = 0.10\text{M}$, $[\text{Cu(II)}] = 0.05\text{M}$. The experimental results are shown in Figure 15. It is seen that neither the gold extraction nor the consumption are much affected with changing oxygen pressure.

LEACHING OF OTHER TYPES OF ORES

Besides the carbonaceous ore, different types of ores were also examined for leaching with thiosulfate. The experimental conditions were: oxygen pressure = 103 kpa, time = 2hr, temperature = 35°C, stirring velocity = 33.3cm/s, $[\text{NH}_3] = 3.0\text{M}$ at pH of 10.5, $[\text{S}_2\text{O}_3^{2-}] = 0.18\text{M}$, $[\text{Cu(II)}] = 0.15\text{M}$, $[\text{SO}_3^{2-}] = 0.10\text{M}$. The experimental results

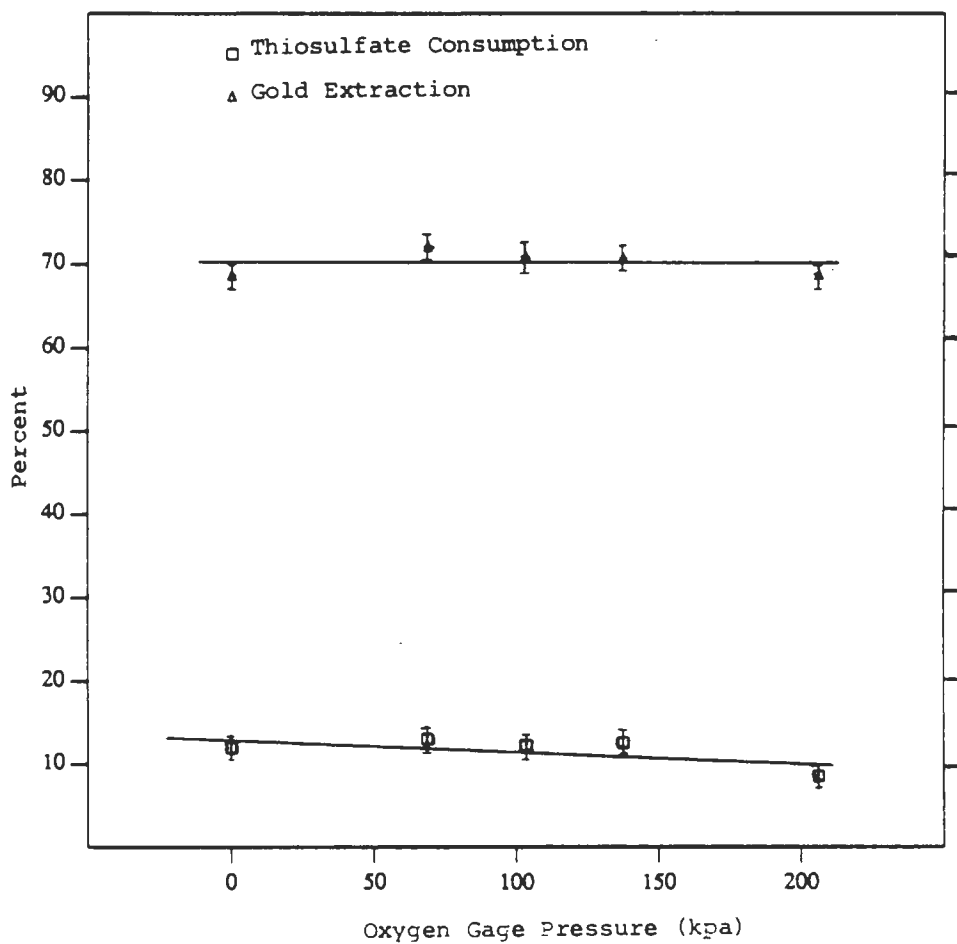


Figure 15- Effect of Oxygen Pressure on the Gold Extraction and the Thiosulfate Consumption

Time = 2hrs; temperature = 35°C; Stirring Velocity = 33.3CM/S; $[S_2O_3^{2-}] = 0.63M$; $[SO_3^{2-}] = 0.10M$; $[Cu(II)] = 0.05M$; $[NH_3] = 3.0M(pH=10.5)$

are shown in Table 5. It is seen that even at the low concentration of thiosulfate (0.18M) high recoveries are obtained, except for the sulfide ore which resulted in a poor extraction; only 18 percent. The two oxide ores from Freeport and Pinson were leached again with concentrated thiosulfate solutions of 0.71M. Recovery improved to 81 percent for both ores from 61 and 77 percent, respectively. Under the same conditions the carbonaceous ore resulted in 71 percent extraction which is within the experimental error from 69 percent. The results are shown in Table 6.

COMPARISON OF PRICES

Extraction results were compared to a cyanide bottle roll test. The results are shown in table 7. Cyanide leaching of the Freeport pink ore has lead to a poor recovery of 10 percent, while thiosulfate at low concentration of 0.088M has resulted in 64 percent recovery of gold. Based on this and reagent consumption, the costs of extraction for the two processes was calculated.

Table 5- Gold extraction from different types of ores with thiosulfate:

type of ore	pH	% Au Extracted	% (S ₂ O ₃ ²⁻) Consumed
carbonaceous	10.6	68.9	30.0
Freeport oxide	10.5	60.6	34.4
Gooseberry	10.5	56.8	27.2
Duval sulfide	10.5	18.1	29.4
Pinson oxide	10.5	77.2	32.8

O₂ pressure = 103 kpag; time = 1hr; temperature = 35°C;
 stirring velocity = 33.3cm/s; [NH₃] = 3.0M at pH of 10.5;
 [S₂O₃²⁻] = 0.18M; [Cu(II)] = 0.05M; [SO₃²⁻] = 0.10M.

Table 6- Comparison of carbonaceous and oxide ore leaching with thiosulfate:

type of ore	pH	% Au Extracted
carbonaceous	10.5	70.9
Freeport oxide	10.5	81.0
Gooseberry	10.5	81.4

O₂ pressure = 103 kpag; time = 2hr; temperature = 35°C;
 stirring velocity = 33.3cm/s; [NH₃] = 3.0M at pH of 10.5;
 [S₂O₃²⁻] = 0.712M; [Cu(II)] = 0.15M; [SO₃²⁻] = 0.224M.

Table 7- Comparison of thiosulfate autoclave leaching with cyanide bottle roll leaching:*

	thiosulfate leaching	cyanide leaching [#]
gold in ore (oz/ton)	0.43	0.43
ore (g)	100	100
initial lixiviant concentration (M)	0.088	0.010
$[S_2O_3^{2-}]/[Au]$ or $[CN^-]/[Au]$ in solution	2.36E3	2.74E2
% gold Extraction	64	10
% lixiviant consumption	61.3	41.1
lixiviant consumption (g/ton)	3.6E4	37.7
reagent cost (\$/ton ore)	17.6	1.05
reagent cost/oz gold extracted (\$/oz)	69.6	23.3

* Data from cyanide were provided by Mr. Fritz Sawyer of the University of Nevada Reno.

NaOH usage is also considered in cyanide leaching (22.7 g/ton). For autoclave leaching Copper, sulfite, and NH_4OH usage is negligible.

DISCUSSION

In order to better interpret the experimental results obtained in this study, it is worth while to analyze the leaching system. The leach solution is a heterogeneous system. Only a small percent of ore is being dissolved in solution. After leaching is completed there is still more than 90 percent of the solid remaining in solution. Therefore the chemistry of leaching is a combination of homogeneous and heterogeneous processes. The solution behavior can not be predicted mainly by the reactions that are based on homogeneous solutions.

Moreover, the ore is a very complex carbonaceous ore. The previous information [18] on carbonaceous ore suggests that carbonaceous matter consists of 1) a predominantly elemental carbon component which behaves like activated carbon and therefore adsorbs gold from solutions, 2) heavy hydrocarbons which do not interact with gold, and 3) organic acids, which can form complexes with dissolved gold. According to this argument each or all of these phenomena can occur in the system. For the ore, however, the only information available concerning the carbonaceous matter is that its carbon content is 4.9 percent, with the organic carbon being 2.5 percent. The type of organic matter is unknown.

Considering these facts, effects of variables on gold extraction and thiosulfate consumption are studied.

The general trend of gold extraction with pH was found to be a sharp increase in extraction with increasing pH. According to reaction (1), however, gold dissolution is inversely proportional to pH, its increase shifts the reaction to the left. As mentioned before, this is a complex heterogeneous system, and other controlling steps such as adsorption are a possibility. The result suggests, therefore, that gold extraction from carbonaceous ore with thiosulfate is controlled by physical adsorption and not chemical equilibrium.

Ammonium hydroxide was added to adjust the pH of the solution. The ammonia content of the thiosulfate leach solution should be controlled so that there is a sufficient amount of free ammonia in the leach solution with a high ratio of free ammonia to soluble metal [2]. Adequate free ammonia causes the formation of $[\text{Cu}(\text{NH}_3)_4]^{2+}$ in solution, which has a characteristic blue color [11]. The fact that solutions of low pH (9.5) were colored clear to pale yellow after leaching, indicates the absence of $[\text{Cu}(\text{NH}_3)_4]^{2+}$ in solution. The percent gold extraction from these solutions was also poor. Solutions of higher pH, however, resulted in a deep blue color after leaching, which shows the existence

of $[\text{Cu}(\text{NH}_3)_4]^{2+}$. These solutions resulted in much higher gold extraction.

Thiosulfate consumption is also dependent on pH. A thiosulfate solution is more stabilized at high pH. As seen from Figures 3 and 4, the consumption decreases with increasing pH. This result is in accord with reactions (5) and (7) which both are inversely proportional to pH.

The influence of temperature on the recovery of gold was studied at temperatures ranging from 25 to 85°C. In the investigations by Tozawa et al. [4], on dissolution of metallic gold in thiosulfate, gold dissolution was found to increase with increasing temperature. They found that gold dissolution reached a maximum at 65°C then gradually decreased above that temperature, but increased again at 100°C. Highest gold dissolution occurred at 65°C. In this study, however, thiosulfate extraction of gold from carbonaceous ore followed a different pattern with respect to temperature. In the temperature range studied, two maxima and one minimum were observed. The two maxima were at 35 and 75°C, and the minimum at 65°C. For this ore the optimum temperature was at 35°C.

The reactions in this system all are equilibria which are temperature dependent, either chemical reactions or physical

adsorptions. A temperature change therefore causes a variation in gold extraction. As far as the thiosulfate consumption is concerned the general behavior is an increase in consumption with increasing temperature. This is because thiosulfate decomposes more quickly at higher temperatures, again due to dependence of the equilibrium reactions on temperature.

Experiments on thiosulfate concentration showed that there is only a slight increase in extraction with increasing thiosulfate concentration (in the range of 0.088 to 0.88M). The thiosulfate consumption also generally increases with increasing concentration. It was shown that sulfite addition stabilizes the thiosulfate solution and decreased the consumption. This is in accord with reactions (6) and (7) by which thiosulfate decomposition is suppressed.

Copper acts as a catalyst in the dissolution of gold in ammonical thiosulfate solution. Experiments were done in order to determine the concentration effect of copper on gold extraction and thiosulfate consumption. Thiosulfate consumption and gold extraction followed the same pattern, and they were not greatly affected by varying the copper concentration. This suggests that reactions (8) and (9) do not play a significant role in extraction. It should also

be noted that the ore contains copper, and the effect of changing copper concentration may not be observable.

Effects of partial pressure of oxygen and leaching time were also studied. The experiments on leach time showed that the reaction was fast and had reached completion after one hour. Thiosulfate consumption, however, increased slightly with time which may be due to thiosulfate decomposition. Experiments on oxygen partial pressures showed that there are no changes either in extraction or consumption with increasing the oxygen pressure.

Besides the carbonaceous ore, oxide and sulfide ores were also studied for leaching with thiosulfate. The results of leaching gold from oxide ore yielded a good extraction (81 percent). Oxide ores are not refractory ores and they usually result in much higher extraction with cyanide. Leaching gold from sulfide ore yielded a poor recovery of only 18 percent. Sulfide ores are known as refractory ores and are not amenable to cyanidation either. Leaching of gold from carbonaceous ore, which is also a refractory ore, resulted in a good extraction of about 70 percent. The results in table 7 show a poor extraction of gold from carbonaceous ore with cyanide (10 percent).

The fact that thiosulfate extracts gold from carbonaceous ore and cyanide does not is due to their gold complex adsorptive properties. The gold thiosulfate complex adsorbs less on carbonaceous ore than does the gold cyanide complex. In sulfide ores, on the other hand, gold occurs external to and disseminated within sulfides[19]. Such gold can not be exposed by grinding and the leach solution may not be in contact with the gold in the ore. Moreover, some sulfides tend to decompose in solution, consuming thiosulfate and inhibiting precious metal extraction. As is seen from our experimental results in Table 5, thiosulfate consumption was high and gold extraction was poor. This may be due to consumption of thiosulfate by sulfide materials. Another possible explanation for the low extraction of gold from sulfides is that the gold in solution could precipitate as sulfides. Moreover, every ore must be evaluated on an individual basis so as to find the optimum conditions for extraction. The sulfide ore in this experiment was leached under the same conditions as the carbonaceous ore. While these conditions were suitable for extraction from carbonaceous ore, better extraction may have been obtained under other conditions for sulfide ores.

A comparison of the costs for cyanide bottle roll test and thiosulfate autoclave leaching was made (table 7). There is a significant difference in percent extraction with using

thiosulfate or cyanide, 10 and 64 percent respectively. However, it should be considered that the experimental conditions for the two methods are quite different. Most important being that the concentration of thiosulfate is nine times that of cyanide.

This calculation shows that reagent cost of thiosulfate, because of higher reagent consumption, is more than that of cyanide. Even though ammonium thiosulfate is cheaper in actual price than cyanide (\$0.22/lb for $(\text{NH}_4)_2\text{S}_2\text{O}_3$, \$0.71/lb for NaCN). The reagent cost per oz of gold extracted in solution is \$70 in the case of thiosulfate and \$23 in the case of cyanide. However, these data are based on the reagent costs only and do not include the cost for handling the ore and equipment. Even though more money is expended to extract gold with thiosulfate, but less of the gold remains in tailings. In the case of cyanide, 90 percent of the gold is sent to the tails. Considering all these factors, ammonium thiosulfate is more advantageous than cyanide leaching of gold from carbonaceous ore.

CONCLUSIONS

This thesis has dealt with the autoclave leaching of gold from carbonaceous ore using thiosulfate as a lixiviant and determination of thiosulfate in the leached solution. Determination of thiosulfate in presence of sulfite and sulfate was successfully measured using the FIA with an electrochemical detection method. The measurements were found to be very accurate and data were reproducible. The method is fast and allowed injection and detection of samples within 10s. Moreover, there are no interferences from either sulfite or sulfate ions.

Thiosulfate ion in the leach solution undergoes a variety of complex degradation reactions in addition to the reaction with gold and side reactions with other metals in the ore. Although identification of any of the possible products were not attempted, analysis of the solution for $[S_2O_3^{2-}]$ showed that under certain conditions roughly 90 percent of the thiosulfate still existed in solution. After the recovery of the dissolved precious metals (e.g. zinc precipitation) from the leaching liquor, the remaining ammonium thiosulfate can be recycled and reused.

The investigation has determined the effects of such variables as temperature, pH, time, oxygen pressure,

ammonium thiosulfate, copper sulfate, and ammonium sulfite concentrations on gold extraction and thiosulfate consumption. Addition of sulfite helped to stabilize the solution and decreased thiosulfate consumption. The consumption also decreased considerably at high pH. Decomposition was found to accelerate at high temperatures. The optimum conditions for obtaining the highest extraction were obtained. Increasing thiosulfate concentration increased extraction slightly, however, low concentration of 0.088M resulted in 64 percent extraction. The optimum temperature was 35°C, and the optimum pH was 10.5. Time and oxygen pressure did not effect the extraction.

Price calculations based on the reagent consumption suggest that the thiosulfate process is economical. A comparison of cyanide with thiosulfate leaching of gold from carbonaceous ore shows that thiosulfate is chemically better than cyanide, since it can result in seven times more percent gold extraction. Even though the cost per oz of gold extracted was higher in the case of thiosulfate, we can conclude that in general thiosulfate is more advantageous than cyanide for extracting gold from carbonaceous ore. This is because the amount of gold in disposed ore and the cost of processing should also be considered.

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