A SURVEY OF CONVENTIONAL AND NOVEL PROCESSES FOR THE TREATMENT OF REFRACTORY GOLD

by

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ABSTRACT

This thesis is a survey of treatments for the recovery of gold from refractory ores. An elucidation of the characteristics leading to gold retention and a definition for refractory gold ore is presented. From this survey of treatments are derived diagnostic conditions that may be useful in predicting the specific refractory characteristics of many ores.

Conventional processes are surveyed and include the refractory ore treatments that enjoy commercial application. Oxidative pretreatments, lixiviants for gold dissolution, and adsorption methods are discussed for both conventional and emerging or less known refractory gold ore treatments. These emerging methods may have seen considerable research and possibly some commercial application. Included are some alternate techniques that were developed for specific ores and that may have been superseded by less expensive or more general treatments.

Some diagnostic test conditions are proposed as being useful in elucidating the refractory components of non-cyanidable ores. Many pretreatments and lixiviating leaches are applied to sulfidic ores with cyanidation gold recoveries as low as 2.6% leading to extractions as high as 99%. Samples of moderately refractory carbonaceous ore with varying organic carbon and sulfide content are treated similarly and exhibit extraction gains from 66.5% to 92.3%

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1.0   INTRODUCTION

The intent of this thesis is to examine past and current hydrometallurgical treatments applicable to the recovery of gold from non-cyanidable deposits. A definition for refractory gold is developed and the factors that contribute to refractoriness are described. The background literature search surveys what is known about the causes of gold retention by ores and reports the state-of-the-art of refractory gold ore treatments.

In 1986, Dry and Coetzee concluded "at present the choice of a particular process route for the recovery of gold from any given refractory ore must be based on the results of empirical testing". This study summarizes conventional processes including the refractory ore treatments that enjoy commercial application. Oxidative pretreatments, lixiviants for gold dissolution, and adsorption methods are discussed for both conventional and emerging or less known refractory gold ore treatments. The emerging methods discussed may have seen considerable research and possibly some commercial application. Included are some alternate techniques that were developed historically for specific ores and that may have been superseded by less expensive or more general treatments.

Treatments of refractory gold ores can be categorized for the particular refractory property that they are designed to address. A further distinction can be made between treatments suitable for low grade ore and those more feasible for high grade or beneficiated ores and concentrates. The focus of this study is to examine whole ore treatment methods that may be generalized for diagnostic purposes.

Some diagnostic test conditions are proposed as being useful in distinguishing between the refractory components of non-cyanidable ores. This combined with mineralogical assessment may reduce the empirical testing requirements referred to by Dry and Coetzee.

A number of surveys of refractory gold ore treatments have been published in the last 30 years by various metallurgical institutes, engineering companies, groups of researchers and individuals. From the earliest survey reviewed (Nagy et al. 1966) to the most recent (Goode 1993) the consensus as to what constitutes a refractory gold ore appears to be any gold occurrence that cannot be substantially recovered by conventional cyanide leaching after grinding to a reasonable size (Mrkusic 1970). The cyanide leaching of gold has been an important hydrometallurgical process since its introduction in a patent by John MacArthur and Robert and William Forrest more than 100 years ago. An arbitrary level of extraction of 80% has been suggested as the division between free-milling and refractory gold ore (Fraser et al. 1991).

The interest in the recovery of gold from refractory ores is a function of prevailing gold price and, more recently, the growing inventory of refractory gold reserves coupled with the exhaustion of easily recoverable gold. A large resource of refractory gold tailings from conventional extraction circuits has provided an easily accessible feedstock for metallurgical test work in the past, but further process development is likely to be incorporated in the exploratory and feasibility study costs of new mine prospects. As new mineral occurrences are discovered it is often found that new problems in the gold extractive process are brought to light (Scheiner et al. 1972). It is necessary to consider the mineralogical characteristics, processing options and economic factors when designing suitable

treatments for specific refractory gold ores. Some preliminary attempts at developing a knowledge base for an expert system have been published (Foo et al., Pendreigh & Barratt, Henley, Major et al.) but a detailed and exhaustive examination of ore mineralogy and the chemistry of process altered residues, for use as a predictive model for gold extraction from refractory ore, remains to be done.

With the exception of the few strictly gravity concentration - direct smelting methods to produce dore, all gold recovery processes incorporate a hydrometallurgical step. The majority of the world's gold production requires the use of cyanide lixiviation and there exist a number of leaching or dissolution pretreatments as well as hydrometallurgical purification and stripping techniques that are applied to both free-milling and refractory gold ores. This study will focus primarily on hydrometallurgical processes with some cosideration of the pyrometallurgy of whole ore roasting.

2.0   LITERATURE REVIEW

- THE DEFINITION OF REFRACTORY GOLD ORE

There are many reasons for which an ore exhibits refractory behaviour. Often it is due to the nature and distribution of the gold itself, but the nature of the overall ore mineralization must be considered. It is often necessary to have a complete elemental as well as mineral analysis of an ore before the refractory nature can be understood. The major groupings of refractory behaviour are those where reactive minerals interfere with the cyanidation process, where physical locking of the gold within cyanide impermeable minerals prevents liberation and a special case where the cyanide or otherwise complexed gold is adsorbed onto a gangue component of the ore. Diagnostic leaching of specific minerals and their stability phases can often clarify the deportment of gold in an ore (Tumilty et al., Lorenzen & van Deventer).

2.1   Cyanicides

Cyanicides are those constituents of an ore that reduce the activity of cyanide for the purpose of gold dissolution. They are represented by three main phenomena and are characterized by the presence of reactive minerals in the ore that lead to cyanide consumption, irreversible cyanide degradation or the related case where reactive minerals cause excessive oxygen consumption interfering with the cyanidation process.

2.1.1 Cyanide Consumption

Metal cations, particularly copper (Stanley), can compete for the cyanide ligand, forming stable complexes and reducing the cyanide activity in solution (Hefter and May). The stabilities of some cyanide complexes are indicated in Table 1 (Hsu). The major loss of uncomplexed cyanide remains volatilization, particularly if the pulp pH approaches 9.4 (Stoychevski & Williams).

Table 1. Cyanide Species Dissociations

Cyanide Complex	Equilibrium Reaction	K_a or K_d
HCN, CN^-	$CN^- + H_2O = HCN + OH^-$	2×10^{-10}
$Zn(CN)_4^{2-}$	$Zn(CN)_4^{2-} = Zn^{2+} + 4CN^-$	10^{-16}
$Cd(CN)_3^-$	$Cd(CN)_3^- = Cd^{2+} + 3CN^-$	10^{-19}
$Ni(CN)_4^{2-}$	$Ni(CN)_4^{2-} = Ni^{2+} + 4CN^-$	10^{-21}
$Cu(CN)_3^{2-}$	$Cu(CN)_3^{2-} = Cu^+ + 3CN^-$	10^{-29}
$Fe(CN)_6^{4-}$	$Fe(CN)_6^{4-} = Fe^{2+} + 6CN^-$	10^{-35}
$Ag(CN)_2^-$	$Ag(CN)_2^- = Ag^+ + 2CN^-$	10^{-41}
$Fe(CN)_6^{3-}$	$Fe(CN)_6^{3-} = Fe^{3+} + 6CN^-$	10^{-42}

2.1.2 Cyanide Degradation

At potentials that can exist in cyanidation of auriferous sulfides it is possible to produce both cyanate and thiocyanate effectively reducing gold dissolution capacity.

$$2S_2O_3^{2-} + O_2 + 2CN^- = 2SCN^- + 2SO_4^{2-} \tag{1}$$

$$2FeS + O_2 + 2H_2O + 14CN^- = 2Fe(CN)_6^{4-} + 2SCN^- + 4OH^- \tag{2}$$

$$Fe_5S_6 + CN^- = SCN^- + 5FeS \tag{3}$$

$$CN^- + SO_2 + O_2 + H_2O \rightarrow CNO^- + H_2SO_4 \tag{4}$$
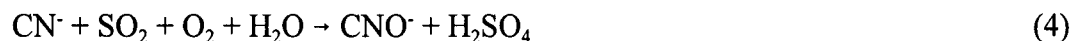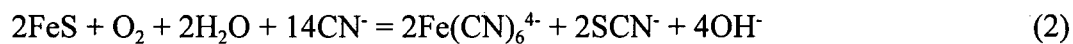
Copper is known to catalyze the oxidation of cyanide to cyanate although the reaction is slow at the pH normally encountered in cyanidation. This reaction in the presence of sulphur dioxide and a copper catalyst is the Inco SO_2-Air cyanide destruction process which is very effective at a potential of 110 mV_{SCE} at a pH of 10 (Devuyst et al.1982), equation (4) being essentially irreversible.

2.1.3   Oxygen Consumption

Elsner [1846] recognized that oxygen was essential to the dissolution of gold by cyanide solutions. Bodländer [1896] suggested that dissolution should proceed through a two step process with the formation of a hydrogen peroxide intermediate (Habashi):

$$2Au + 4NaCN + O_2 + 2H_2O = 2NaAu(CN)_2 + 2NaOH + H_2O_2 \tag{5}$$

$$2Au + 4NaCN + H_2O_2 = 2NaAu(CN)_2 + 2NaOH \tag{6}$$

$$4Au + 8NaCN + O_2 + 2H_2O = 4NaAu(CN)_2 + 4NaOH \tag{7}$$
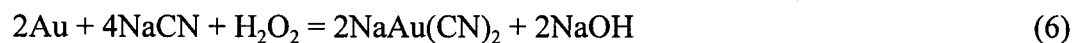
Minerals which decompose in the alkaline cyanidation solutions to form polysulfide anions, sulfoxy anions (polythionate, thiosulfate), thioarsenite, thioantimonite and various other oxidizable species

tend to compete for the dissolved oxygen necessary for gold dissolution (Nagy et al., Komnitsas and Pooley 1989). Pyrrhotite is particularly reactive as an oxygen consumer (Graham).

2.2   Occluded Gold

Siliceous refractory ores can lock gold into the gangue phase. This phase can be made up of microcrystalline quartz, chalcedony, chert, or amorphous forms of opaline silica. The gold can be present either as microfine metallic gold or auriferous sulfides occluded in the impermeable matrix (Hausen 1989). This type of gold occurrence can lead to size dependant refractoriness where gold cannot be liberated with a reasonable input of grinding energy.

An important variation of this is where the gold exists as a microfine dispersion or even a lattice substitution in a sulfide mineral. Submicron gold has been found in pyrite, arsenopyrite, pyrrhotite and chalcopyrite in levels up to 20 ppm (Chryssoulis et al. 1988) and 488 ppm in pyrite (Swash). Arsenic rich zones within a mixed sulfide matrix have been shown to contain as much as 6900 ppm gold (Claassen). To release this gold it is necessary to alter the matrix, generally by a destructive oxidation, since grinding to submicron levels is impractical. Auriferous sulfides represent the majority of refractory gold ores.

Other refractory gold ores contain gold solid solutions that have been found to resist dissolution by cyanide. These commonly contain gold compounds with tellurium, antimony, arsenic, selenium and occasionally lead or bismuth (Vaughan, Goode).

2.2.1   Tellurides

Solid solutions of gold in tellurium are resistant to extraction with cyanide.

Table 2. Principal Gold Bearing Tellurides

Calaverite	$AuTe_2$	Sylvanite	$AuAgTe_2$
Montbrayite	Au_2Te_3	Petzite	Ag_3AuTe_2
Krennerite	$(AuAg)Te_2$	Kostovite	$AuCuTe_4$

2.2.2   Antimonial Ores

Antimonial ores generally contain stibnite occluded gold and occasionally aurostibnite where the gold substitutes for antimony in the sulfide matrix. The dissolution of stibnite (Sb_2S_3) can lead to the formation of thioantimonites at high pH and it has been suggested that these can passivate a gold surface by adsorption (Stanley).

2.3   Preg-robbing Ores

Gold ores containing various forms of graphitic or organic carbon can actively adsorb the dissolved gold cyanide species. This leads to a portion of the gold remaining bound to carbonaceous particles and reporting to the tailings. The term preg-robbing was first used by the Carlin Gold Mining Company to describe highly active types of carbonaceous ores. The mere presence of carbon is not

a reliable indicator of preg-robbing capacity since it is necessary for the carbonaceous gold ore to be an active gold adsorber. The ore can be inherently active or become activated by thermal or oxidative treatments.

2.3.1 Carbonaceous Species

A variety of functional groups and active sites are available on active organic carbon including both oxidizing and reducing sites and many stereochemically specific surfaces. These sites have been partially described by infrared analysis to include most known simple aliphatic and aromatic combinations of carbon, oxygen and hydrogen with the presence of thiosulfate and amino functional groups also known. Organic (humic) acids are known to react with aurocyanide species preventing the complete recovery of gold from the ore (Afenya, Guay 1981).

Graphitic or crystalline carbon may provide the primary adsorption sites for aurocyanide and may cause a polymerization to $Au_4(CN)_5^-$ on the graphitic plate faces at acidic pH levels (Ibrado and Fuerstenau).

2.3.2 Clay Minerals

Some clay minerals such as limonite and halloyite have been observed to provide adsorption sites for the gold cyanide complex. A study of the K_2O content present as the mineral illite $((K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}((OH)_2H_2O))$ at one mine suggests that there is a correlation between

the clay content and the tendency of the carbonaceous ore to be "geo-activated" and become preg-robbing (Fuchs & Schurer). It is also possible that cationic sites for the adsorption of gold cyanide complex can be created by the initial adsorption of a cation from the dissolution of associated minerals.

3.0   LITERATURE REVIEW

- CONVENTIONAL REFRACTORY GOLD ORE TREATMENTS

The various reasons for which gold ores exhibit refractory behaviour have spawned a number of approaches to the problem. It is often found that a combination of the reasons for gold refractoriness are found in a single ore. When both refractory sulfidic minerals and active carbonaceous components exist the ore is referred to as doubly refractory. Once the nature of an ore's refractoriness has been determined an appropriate treatment method can be chosen from the variety that are available. The difficulty that often arises is the fact that most methods were originally developed as site specific remedies and may not translate well to a new ore type. Some generally successful hydrometallurgical treatments have found commercial application at multiple sites and can be considered conventional.

3.1   Oxidizing Pretreatments

Conventional methods for the oxidation of refractory gold ores and concentrates include both pyrometallurgical (roasting) and hydrometallurgical (pre-aeration, pressure oxidation, chlorine/hypochlorite oxidation, and bioleaching) pretreatments.

3.1.1 Pre-aeration

The pre-aeration of refractory gold ores is generally used as a method to oxidize cyanicides before cyanidation. It includes any method that improves oxygen contact with unstable minerals at ambient pressure by sparged air or well vortexed agitation. It can be used in the pretreatment of slurries or heaps (Bartlett) and is most often done in the presence of a caustic reagent such as lime or sodium hydroxide. There is evidence of the leaching of unstable pyritic minerals such as the pyrrhotites, marcasite and framboidal pyrite at high pH in a pre-aeration stage.

$$4FeS + 5O_2 + 4H_2O + 4OH^- = 4Fe(OH)_3 + 2S_2O_3^{2-} \tag{8}$$

$$4FeS_2 + 15O_2 + 16OH^- = 4Fe(OH)_3 + 8SO_4^{2-} + 2H_2O \tag{9}$$

$$S_2O_3^{2-} + 2O_2 + 2OH^- = 2SO_4^{2-} + H_2O \tag{10}$$
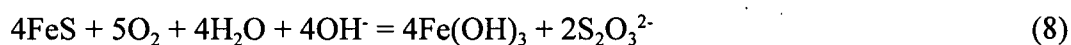
At the Golden Sunlight Mine, research has suggested that the pre-aeration of sulfides prevents the formation of thiocyanates and improves the free cyanide concentration in the gold dissolution stage (Lofftus et al.).

3.1.1.1 Cyanidation Promoters

It is common that the pre-aeration treatment also incorporates the addition of cyanidation promoters. These consist of soluble lead or mercury salts which remove sulfide from solution before it begins to consume oxygen. It has been concluded that lead in solution can activate passivated sulfur

complexed gold surfaces, prevent gold sulfide film formation and act as a sulfide oxidation intermediary through lead sulfide formation (Kondos et al.)

3.1.2 Pressure Oxidation

The extension of pre-aeration to higher temperatures and overpressures of oxygen is becoming a popular method for the pretreatment of refractory gold ores. Although the process in alkaline conditions is used commercially at the Mercur Mine, which has a high carbonate content ore, it is the acid process that is finding application around the world. An improvement in recovery is brought about by the oxidation of cyanicides and the destruction of the sulfide matrix which often occludes finely disseminated gold.

$$4FeS_2 + 14O_2 + 4H_2O = 4FeSO_4 + 4H_2SO_4 \tag{11}$$

$$4FeSO_4 + O_2 + 2H_2SO_4 = 2Fe_2(SO_4)_3 + 2H_2O \tag{12}$$

$$4FeS_2 + 15O_2 + 2H_2O = 2Fe_2(SO_4)_3 + 2H_2SO_4 \tag{13}$$

$$FeS_2 + 2O_2 = FeSO_4 + S° \tag{14}$$
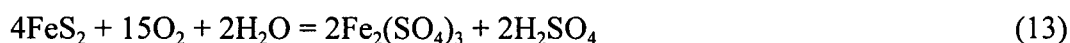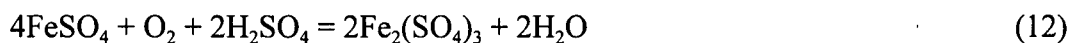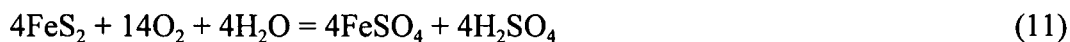
The elemental sulfur product is considered an intermediate (McKay & Halpern) and in high temperature pressure oxidation (>160°C), is itself oxidized.

$$2S° + 3O_2 + 2H_2O = 2H_2SO_4 \qquad\qquad (15)$$

Considerable research has been done to clarify the mechanism of sulfide oxidation and it is possible that elemental sulfur is a low potential reduction product from a true intermediate such as thiosulfate or a polysulfide (Mishra & Osseo-Asare).

Further hydrolysis reactions of the ferric sulfate produce more acid, requiring a neutralization step before cyanidation can be carried out. This requirement for neutralization often represents a significant cost factor when a ready source of limestone is not close at hand.

$$Fe_2(SO_4)_3 + 2H_2O = 2Fe(OH)SO_4 + H_2SO_4 \qquad\qquad (16)$$

$$Fe_2(SO_4)_3 + 3H_2O = 2Fe_2O_3 + 3H_2SO_4 \qquad\qquad (17)$$

$$3Fe_2(SO_4)_3 + 14H_2O = 2H_3OFe_3(SO_4)_2(OH)_6 + 5H_2SO_4 \qquad\qquad (18)$$

A hydronium jarosite product can incorporate various metals from the reaction slurry. This can be the cause of poor silver recoveries in pressure oxidation processes since silver jarosite is relatively insoluble in the gold extraction stage (Berezowski & Weir 1983,1989).

Arsenopyrite exhibits some elemental sulfur production (Equation (23)) but the desired high temperature product in a fully oxidized system is ferric arsenate and sulfate (Papangelakis & Demopoulos 1990).

$$2FeAsS + 7O_2 + 6H_2O = 2FeAsO_4 \cdot 2H_2O + 2H_2SO_4 \qquad (19)$$

$$4Fe_7S_8 + 69O_2 + 10H_2SO_4 = 14Fe_2(SO_4)_3 + 10H_2O \qquad (20)$$

$$4FeS + 9O_2 + 2H_2SO_4 = 2Fe_2(SO_4)_3 + 2H_2O \qquad (21)$$
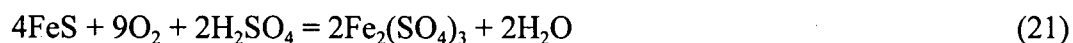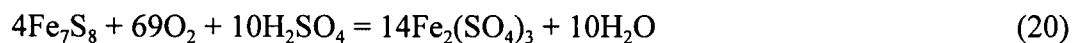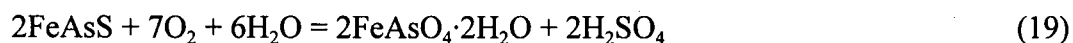
The pyrrhotite reactions approximated in equations (20) and (21) demonstrate a net acid consumption. All of these sulfides provide significant heat upon oxidation with arsenopyrite providing the most energy per mole of sulfide sulfur (Mason).

A combination of ultra-fine grinding of sulfide flotation concentrates followed by low intensity pressure oxidation has shown some success in refractory gold ore treatment and is marketed as the Activox™ process (Johnson et al.).

3.1.3   The Redox Process

The Redox process uses a nitric acid catalyzed oxygen leach to decompose refractory pyrite and arsenopyrite gold ores. The residue produced can be treated for gold extraction by conventional means such as cyanidation. It is because of the nitric acid catalysis that the leach is very rapid and complete.

Variants of the process have been developed to optimize the treatment of materials with a range of pyrite and arsenopyrite contents (Beattie et al.).

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3.1.3.1 Leach Chemistry

Pyrite (FeS_2) and arsenopyrite (FeAsS) are decomposed by nitric acid according to the following equations:

$$FeS_2 + 5\ HNO_3 = 1/2\ H_2SO_4 + 1/2\ Fe_2(SO_4)_3 + 5\ NO + 2\ H_2O \qquad (22)$$

$$FeAsS + 4.27\ HNO_3 + 1.4\ H^+ = Fe^{3+} + 0.8\ SO_4^{2-} + 0.2S +$$

$$H_3AsO_4 + 4.27\ NO + 4/3H_2O \qquad (23)$$
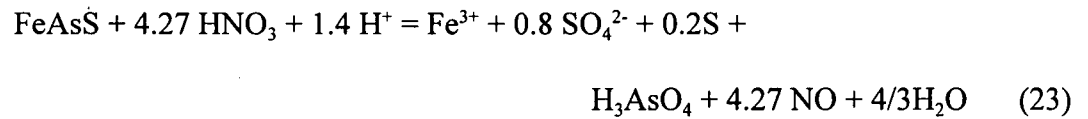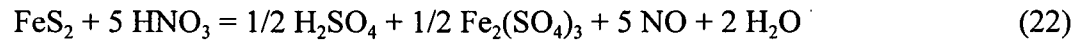
The stoichiometric consumption of nitric acid by pyrite is 2.63 kg/kg and sulfate is the only product of sulfur oxidation. The stoichiometric consumption of nitric acid by arsenopyrite is 1.65 kg/kg and the reaction has approximately a 20% yield of elemental sulfur.

Less stable minerals such as marcasite develop lower potentials at the surface and can decompose as follows:

$$FeS_2 + 4HNO_3 = Fe(NO_3)_3 + 2S + NO + 2H_2O \qquad (24)$$
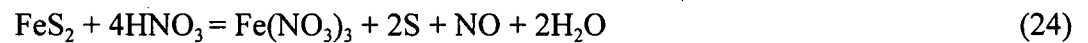
This produces elemental sulfur which is difficult to oxidize further at low potentials.

The product of nitric acid reduction is nitric oxide gas, NO. Nitric oxide reacts with oxygen to form

nitrogen dioxide, NO_2, which is absorbed as nitric acid. These reactions are shown in the equations

below:

$$6\ NO + 3\ O_2 = 6\ NO_2 \tag{25}$$

$$6\ NO_2 + 2\ H_2O = 4\ HNO_3 + 2\ NO \tag{26}$$

$$4\ NO + 3\ O_2 + 2\ H_2O = 4\ HNO_3 \tag{27}$$

The regeneration of nitric oxide to nitric acid can be carried out concurrent with the leach or as a

separate unit operation.

Overall, the reactions of pyrite and arsenopyrite with oxygen in the Redox process, are shown in the

equations (28) and (29):

$$FeS_2 + 15/4\ O_2 + 1/2\ H_2O = 1/2\ H_2SO_4 + 1/2\ Fe_2(SO_4)_3 \tag{28}$$

$$FeAsS + 3.2\ O_2 + 0.8\ H_2O + 1.4\ H^+ = Fe^{3+} + 0.8\ SO_4^{2-} + 0.2\ S° + H_3AsO_4 \tag{29}$$
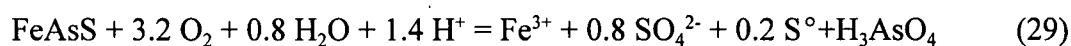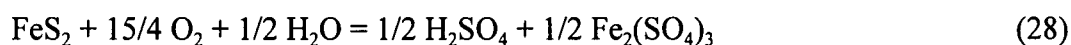
By these equations the stoichiometric consumption of oxygen is 0.62 kg/kg arsenopyrite and 1.00

kg/kg pyrite. The initial reaction with arsenopyrite consumes one equivalent of acid per mole of

sulfide reacted; pyrite generates one equivalent of acid.

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In the atmospheric pressure leach used for low grade sulfide ores the nitric oxidant is regenerated outside of the leach vessel so that the final stage of a continuous circuit is nitrate depleted. The high pressure autoclave process uses the in-situ regeneration of nitric acid to maintain the reaction of oxygen with a sulfide concentrate.

3.1.3.2 Precipitation Reactions

The Redox leach solubilizes iron, arsenic and sulfur, however, precipitation reactions can take place concurrently. These are favoured by high temperature and low solution acidity. The compounds precipitated include ferric arsenate, $FeAsO_4 \cdot 2H_2SO_4$; basic ferric sulfate, $Fe(SO_4)(OH)$; and hematite, Fe_2O_3. Silver can be precipitated as refractory silver jarosite, $AgFe_3(SO_4)_2(OH)_6$ resulting in metal value losses to the tails.

$$Fe^{3+} + H_3AsO_4 + 2\ H_2O = FeAsO_4 \cdot 2H_2O + 3\ H^+ \tag{30}$$

$$Fe^{3+} + 3/2\ H_2O = 1/2\ Fe_2O_3 + 3\ H^+ \tag{31}$$
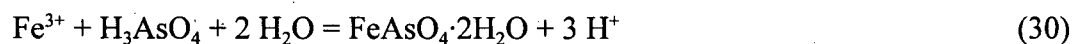
Both reactions produce three equivalents of acid.

The bulk of the sulfate must be removed from solution as gypsum or anhydrite. The calcium sulfate is produced by the addition of limestone to the leach slurry as shown in equation (32):

$$CaCO_3 + SO_4^{2-} + 2\ H^+ = CaSO_4 + CO_2 + H_2O \tag{32}$$
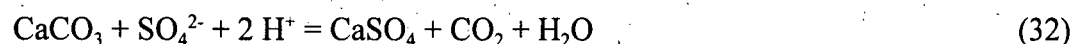
It was observed in pilot plant trials of the process at Bacon, Donaldson & Associates' test facility in 1988 (unpublished) that the precipitation of sulfate promotes the precipitation of ferric arsenate probably by removing sulfate ligands that compete for the necessary ferric iron in solution although the pH increase to 1.5 due to concurrent acid neutralization also plays a part.

The overall reaction for the oxidation of pyrite with the production of hematite proceeds as follows:

$$FeS_2 + 2CaCO_3 + 15/4O_2 = 1/2Fe_2O_3 + 2CaSO_4 + 2CO_2 \tag{33}$$
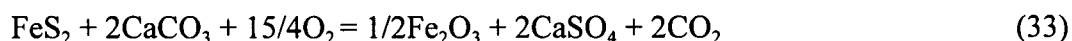
In this reaction there is no overall production or consumption of acid.

3.1.4   The Nitrox Process

This nitrate catalyzed oxidation method has been generally limited to the atmospheric pressure leaching of refractory gold ore using air as an oxidant. The nitric acid is regenerated within the reaction vessel in a manner similar to the high temperature Redox process (Van Weert et al.).

3.1.5   Chlorine/Hypochlorite Pretreatment

Sodium and calcium hypochlorite and gaseous chlorine additions to carbonaceous ore slurries (or their percolation through heaps) provide a more oxidizing environment than simple pre-aeration. The intent is to destabilize any existing gold organic complexes that would result in soluble loss

(Ahmadiantehrani et al., Scheiner et al. 1968) or to deactivate carbon species that would compete for the gold cyanide complex (Ahmadiantehrani & Hendrix). There is evidence that some of the auriferous iron sulfides are oxidized (Bayrakceken et al.) and this could enhance gold liberation which is in fact seen as some gold dissolution in the oxidizing chloride environment. The exact mechanism of gold complex adsorption onto organic and graphitic carbon species is still somewhat problematical so that the effect of chlorine species oxidation of carbonaceous gold ore is not entirely predictable. The oxidation of pyrite by hypochlorite would proceed electrochemically and the overall reaction could be described by:

$$FeS_2 + 7NaOCl + 2NaOH = FeCl_2 + 2Na_2SO_4 + 5NaCl + H_2O \tag{34}$$
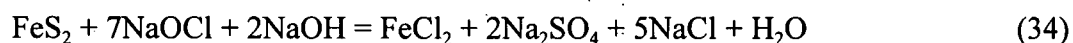
3.1.6   Bioleach Oxidation

Bacterial oxidation of pyrite and arsenopyrite is the major cause of acid rock drainage since the bacterial species responsible occur naturally in waste rock dumps and sulfidic tailings. Several commercial installations are now operating where the appetite of *Thiobacillus ferrooxidans* for pyrite or arsenopyrite is harnessed to provide a controlled oxidation of the refractory matrix. This results in gold liberation and improved gold recoveries in cyanidation (Brierly, Bruynstein, van Asweegan et al., Hackl, Harris & Brierly, Nagpal et al.).

An advantage of bioleaching is in the selective oxidation of high energy areas at the faults and defects of a pyritic sulfide matrix. For ores where microfine gold particles (500Å to 20μ) occupy

these faults and discontinuities in the pyrite mineral, it is not necessary to achieve complete oxidation to get high gold extraction. In the case for arsenopyrite where the gold is finely disseminated throughout the matrix, or pyrite/arsenopyrite co-occurrences where often the gold is enriched in the arsenic containing mineral, it is fortunate that the bio-oxidation rate is enhanced. Often, nearly complete oxidation is possible in treatment times as short as seven days (Spencer & Budden 1990, Komnitsas & Pooley 1990, Komnitsas & Pooley 1991, Karavaiko et al.).

The bacteria oxidize ferrous iron to ferric and sulfide sulfur to sulfate as a metabolic process. It is unclear as to the fate of elemental sulfur which results from the chemical leaching of the original pyrite feed by the bacterially produced ferric iron (Chaudhury et al.) although bacterially assisted oxidation is plausible.

$$4FeS_2 + 15O_2 + 2H_2O = 2Fe_2(SO_4)_3 + 2H_2SO_4 \tag{35}$$

$$Fe_2(SO_4)_3 + FeS_2 = FeSO_4 + 2S° \tag{36}$$

$$2S° + 3O_2 + 2H_2O = 2H_2SO_4 \tag{37}$$
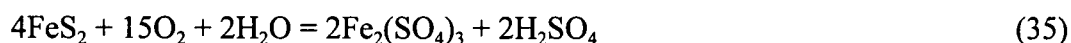
Other bacterial species such as *Thiobacillus thiooxidans* and higher temperature *Sulfolobus acidocaldarius*, have been tested for their amenability to refractory gold ore treatment (Liu et al.). The optimization of bioleach methods is empirical due to difficulties in direct bioassay determinations since the techniques for speciation and culture population determination are not as yet well defined. Visual enumeration, protein assay and infrared methods are in development (Ralph). In amenability testing, the variables of concern are culture type, nutrition requirements,

oxygen transport, temperature and time of leach to effect gold liberation. The measureable variables are mixing power input, pH, potential, pulp density and dissolved metal content. It has been suggested that careful control of pH is necessary to avoid precipitation of basic iron/arsenic/sulfate compounds (Barrett & Hughes) which with other jarosite formation can interfere with gold liberation.

Bioleach treatment of carbonaceous refractory gold ore does not always address the preg-robbing problem. Newmont has developed and patented the use of species of the *Pseudomonas*, *Achromobacter* and *Arthrobacter* genera to modify or block the gold adsorption in active carbonaceous gold ore (Brierly & Kulpa). The use of diesel oil, ostensibly as a nutrient source for the bacteria in the Newmont process, has been used on its own as a carbon blinding agent.

3.1.6.1 Bioheapleach

Cyanide heapleaching technology for low grade oxidized ores is well established (McLelland et al., Pantelis & Ritchie). The use of bacterial agents in a pre-oxidation step for low grade refractory ore is a natural extension of uncontrolled oxidation exhibited in sulfidic waste rock and tailings. The controlled process generally requires an acidification and nutrient addition to get the bacterial growth phase started and to enhance overall kinetics. As in tank bioleaching, the *Thiobacillus ferrooxidans* and other chemoautotrophs derive sustenance from the sulfidic matrix. The result is liberation of the occluded gold by oxidation and dissolution of pyrite (Chaudhury et al.). The use of cyanide as lixiviant is complicated by the iron compound precipitation upon neutralization to a pH amenable

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to cyanidation. There has been some test work to examine the use of thiourea at low pH in this application.

The use of bioheapleach with sulfide ingesting species will not completely relieve carbonaceous ores of a tendency to preg-rob (Afenya). The design of the heap to maximize solution percolation and encourage bacterial activity is the subject of a number of patents. Agglomeration using an inoculum provides an effective distribution of bacteria and improved percolation for cyanide, thiourea or thiosulfate extraction methods (Brierly & Hill).

3.2   Lixiviation

The recovery of gold from refractory ore would ideally involve the use of a solvent that extracts only the gold, leaving the gangue intact. Indiscriminate solvents such as aqua regia lead to complicated purification requirements. The most common lixiviant for the dissolution of gold is cyanide, usually as a sodium or potassium salt. Other gold complexing agents have been developed to avoid the problem of cyanicides, to provide additional oxidizing potential to liberate and dissolve occluded gold, and to dissolve gold in the acid environment created by some of the stronger oxidizing pretreatments. Other developments in lixiviants are focused on combining the pre-oxidation and dissolution steps into one operation by using oxidizing species capable of gold complexation in solution. Cyanide gold extraction provides the benchmark for evaluating refractory ore treatments.

3.2.1 Cyanide Lixiviation

The accepted mechanism for the dissolution of gold with cyanide is thought to proceed with the formation of a peroxide intermediate.

$$2Au + 4NaCN + O_2 + 2H_2O = 2NaAu(CN)_2 + 2NaOH + H_2O_2 \qquad (38)$$

$$2Au + 4NaCN + H_2O_2 = 2NaAu(CN)_2 + 2NaOH \qquad (39)$$

$$4Au + 8NaCN + O_2 + 2H_2O = 4NaAu(CN)_2 + 4NaOH \qquad (40)$$
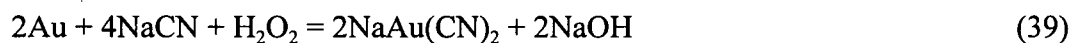
Once the appropriate pre-oxidation steps have been taken to liberate refractory gold it is possible to achieve a dissolution rate of 1.8 mg cm^{-2} h^{-1}. In the case of very fine gold liberated from a sulfide matrix the extraction rate is enhanced by the large surface area of the exposed gold particles (Haque 1992). The cyanidation stage is sometimes simply limited by kinetics where the gold surface is in some way passivated or where cyanide is consumed by base metal complexation. Methods of enhancing the dissolution kinetics are the use of intensive cyanidation techniques such as a pressurized leach or high concentration cyanide solutions (Kontopoulos & Stefanakis 1990).

3.2.2 Pressure Cyanidation

A pipe reactor has been installed by Consolidated Murchison Mine to treat an antimonial gold concentrate. The increased oxygen activity at pressures as high as 13.5 MPa prevents the oxygen consumers present from interfering with cyanidation. Temperatures up to 60°C are shown to improve

the kinetics of cyanidation whereafter decomposition of cyanide becomes a concern (Davis & Patterson, Muir et al.). The method has limited use where the gold is occluded in a sulfide matrix since much of the original sulfide remains intact at the low temperatures and moderate pH of this operation.

3.2.3 Thiourea Lixiviation

The use of thiourea for the direct extraction of gold has many intrinsic advantages over cyanidation of refractory ores where cyanide consumption is the major problem. An important advantage comes from its enhanced kinetics, said to be 10-12 times greater than cyanide (Charley). The thiourea is oxidized by an added oxidant such as ferric iron or hydrogen peroxide to formamidine disulfide which serves as a selective oxidant for gold or silver.

$$2CS(NH_2)_2 + 2Fe^{3+} = NH_2(NH)CSSC(NH)NH_2 + 2H^+ + 2Fe^{2+} \tag{41}$$

$$2Au^\circ + 2CS(NH_2)_2 + NH_2(NH)CSSC(NH)NH_2 + 2H^+ = 2Au(CS(NH_2)_2)_2^+ \tag{42}$$

$$Au^\circ + Fe^{3+} + 2CS(NH_2)_2 = Au(CS(NH_2)_2)_2^+ + Fe^{2+} \tag{43}$$
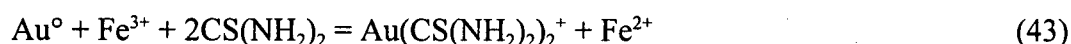
The major advantage of thiourea is that it dissolves gold in acidic solutions and may be used to treat the pre-oxidized acid pulps without a neutralization or solid-liquid separation stage (Deschenes, Gabra, Hiskey 1983, Raudsepp & Allgood, Yen & Wyslouzil). In general thiourea has found limited commercial application in part due to the difficulty in potential control to prevent the irreversible oxidation of the formamidine disulfide to elemental sulfur resulting in reagent consumption. There

is some indication that thiourea is carcinogenic and this has led to prohibition of its use in some

countries (Yen & Pindred 1989).

3.2.4 Chlorine

The use of chlorine for the dissolution of gold was known before the advent of cyanidation.

Dissolved chlorine, hypochlorous acid and hypochlorite ion are all capable of oxidizing gold in a

chloride medium. The predominant complex is $AuCl_4^-$ (Figure 1.). The aurous complex, $AuCl_2^-$ can

exist in high chloride activity solutions (Hiskey & Atluri 1988) but tends to disproportionate to gold

metal and the auric complex.

Pressure leaching with hypochlorite in a chloride solution followed by a hot hydrochloric acid leach

extracted in the range of 75-98% of the gold from an ore for which cyanidation recovered only 20%

(Yen & Pindred 1989), but hypochlorite consumption was prohibitively high for the best gold

recovery. This is a general problem with chlorine systems since the potential is higher than necessary

to effect gold dissolution and pyrite oxidation. The nature of the leach solution prevents the use of

inexpensive conventional materials for leach vessels often requiring all wetted surfaces to be made

of titanium.

3.2.5 Bromine

The ability of bromine to leach gold was the subject of a patent in 1881 but like chlorine leaching, it was supplanted by cyanidation. The development of the K-process (assisted by the involvement of the Dead Sea Bromine Group) and the manufacture of bromo-hydantoins by Great Lakes Chemicals has lead to renewed interest in bromine gold leaching. The K-process has been said to leach gold at rates up to 100 times that of cyanide (Capps & Cameron) while avoiding the dissolution of copper. The process of gold dissolution is enhanced by the presence of both hypobromous acid and bromide according to:

$$2Au° + 3HOBr + 5Br^- + 3H^+ = 2AuBr_4^- + 3H_2O \tag{44}$$
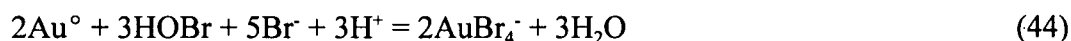
The potential-pH diagram for the gold-bromide system predicts a stability for the $AuBr_2^-$ species as well (Figure 2.). Tests with bromo-hydantoins as a source of hypobromous acid for gold dissolution used a roasting oxidation pre-treatment to avoid exorbitant reagent consumptions (Dadgar). As in cyanidation, the presence of oxidizable substances such as humic acid or soluble sulfides leads to high reagent consumptions (Sergent).

3.2.6 Iodine

The use of iodine as a gold lixiviant is the subject of a patent (McGrew & Murphy) and the applicability to refractory gold ores is the case where mild oxidation conditions are adequate to

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liberate the gold. The I_3^-/I^- couple at 10^{-3} M provides a potential of 0.71 volts which is sufficient to oxidize gold in the presence of iodine (Hiskey & Atluri 1988). The complex formed is the most stable of the halides examined and exists at lower potentials and higher pH's (Figure 3) than either the chloro- or bromo-gold complexes (Tran & Davis 1991).

3.2.7 Other Lixiviants

There are several other oxidants or complexing agents for gold that operate in aqueous solution at a variety of potentials and pHs. The sulfoxy species such as the strongly oxidizing Caro's acid, (Lakshmanan), and the complexing lixiviant thiosulfate (Kerley 1981 & 1983, Langhans et al.) have been applied to various low grade or refractory gold ores. Sulfurous acid formed by the addition of sulfur dioxide is the subject of a patent claiming the formation of a soluble gold sulfite complex that can be adsorbed by resin in the presence of a chelating compound (EDTA) and air as an oxidant (Touro et al.). Malononitrile, a dinitrile of formula $CH_2(CN)_2$ and its many derivatives, can complex gold in a manner analogous to thiourea without the problem of reagent oxidation to elemental sulfur (Heinen et al. 1970, Scheiner & Lindstrom).

Table 3 summarizes the optimal pH and potential regimes for the production of stable gold complexes for a variety of lixiviants. Figures 1-3 demonstrate the gold halide stability areas in the form of Eh-pH diagrams (Hiskey & Atluri).

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Table 3. Gold Lixiviants

Lixiviants	Complex	pH	Eh V$_{SHE}$
Cyanide	$Au(CN)_2^-$	10-11	-0.6
Thiourea	$Au(CS(NH_2)_2)_2^+$	2	0.4
Chloride	$AuCl_4^-$	<3	1.0-1.2
Bromide	$AuBr_4^-$	4-6	0.7-0.9
Iodide	AuI_4^-	4-10	0.7
Thiosulfate	$Au(S_2O_3)_2^{3-}$	> 7	0.1
Polysulfide	AuS^-	> 10	0.5
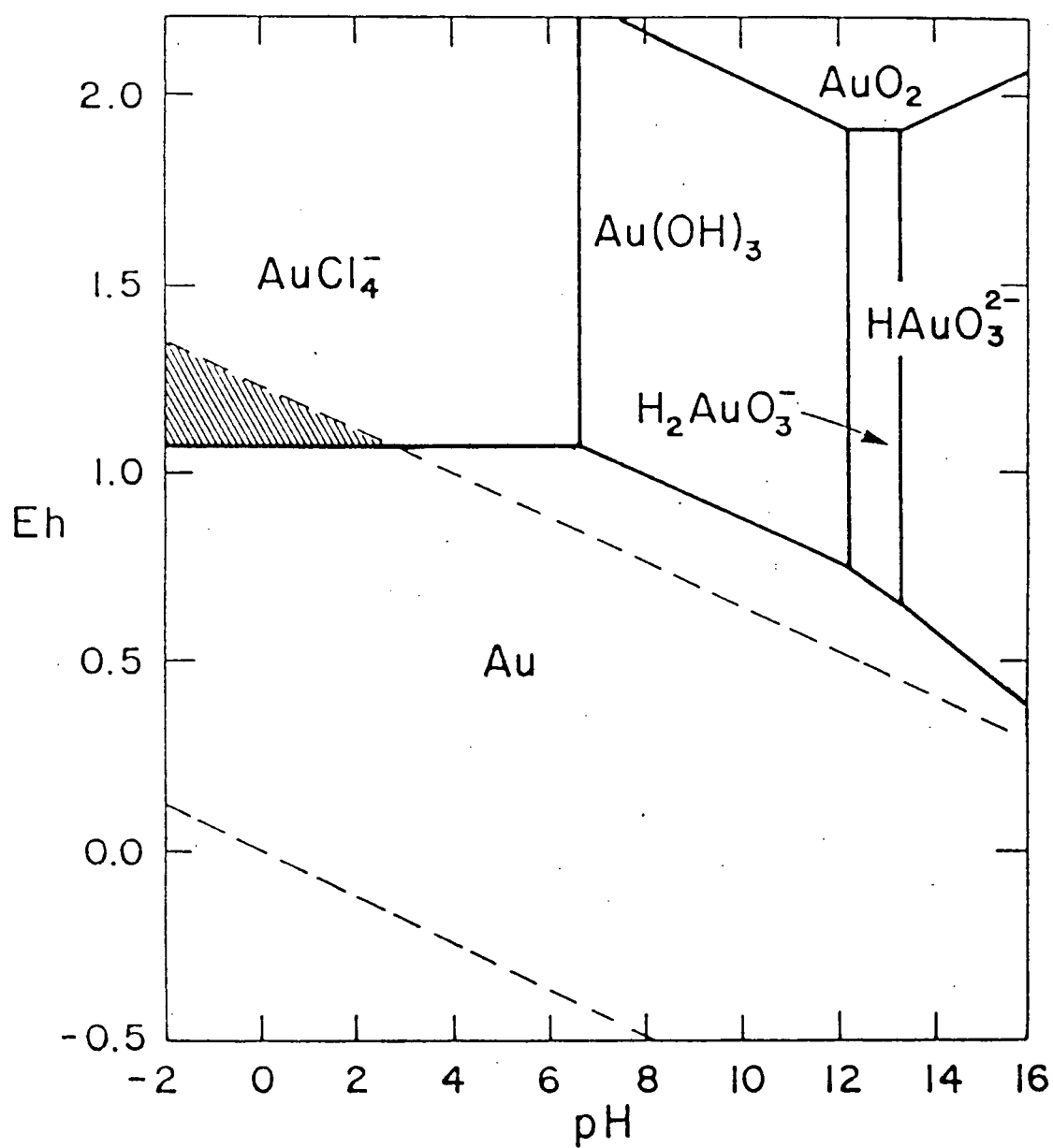
Figure 1.   Potential-pH equilibrium diagram for the gold-chloride system for [Au] = 10^{-5} M and [Cl] = 10^{-2} M
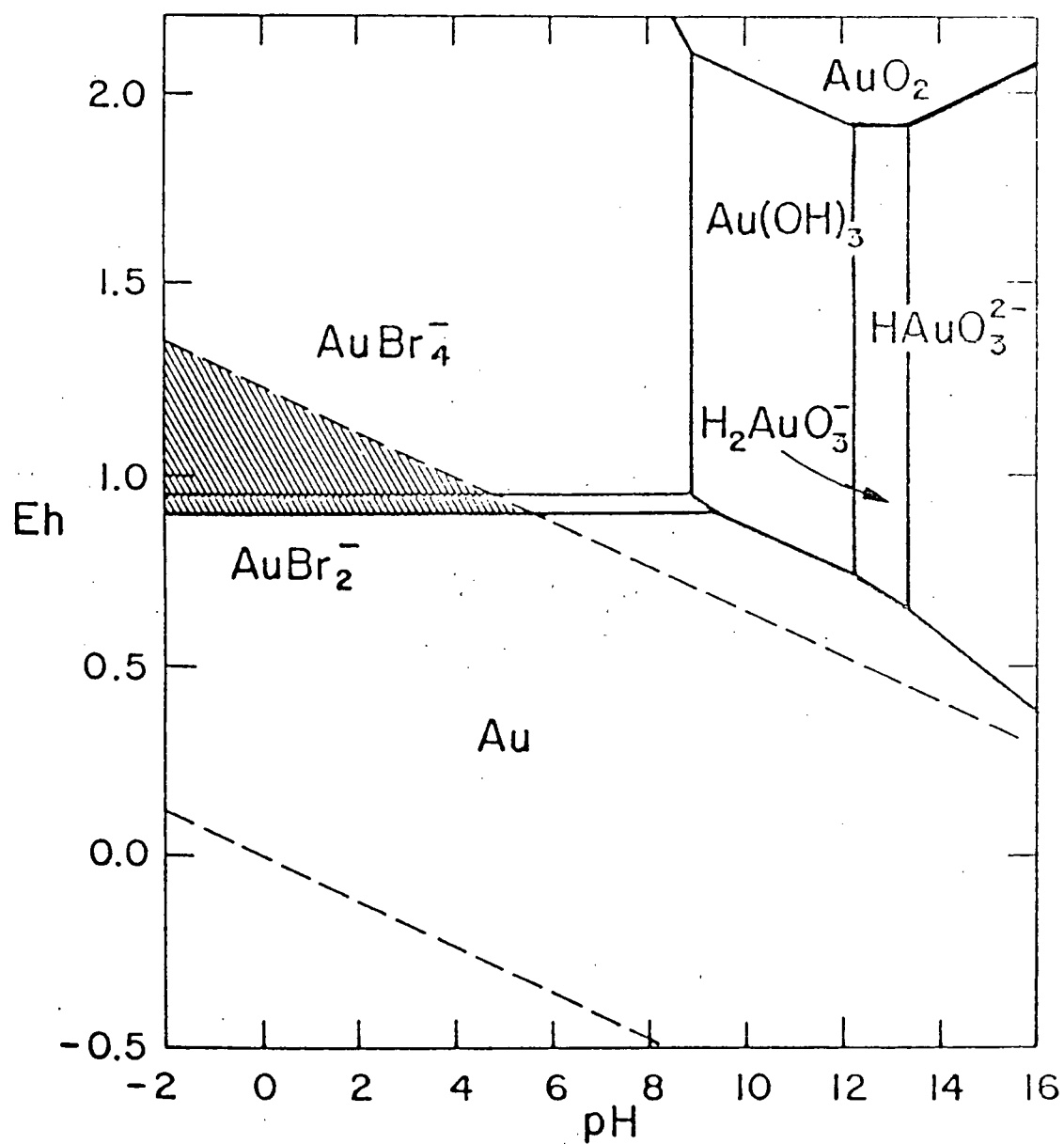
Figure 2.   Potential-pH equilibrium diagram for the gold-bromide system for [Au] = 10^{-5} M and [Br] = 10^{-2} M

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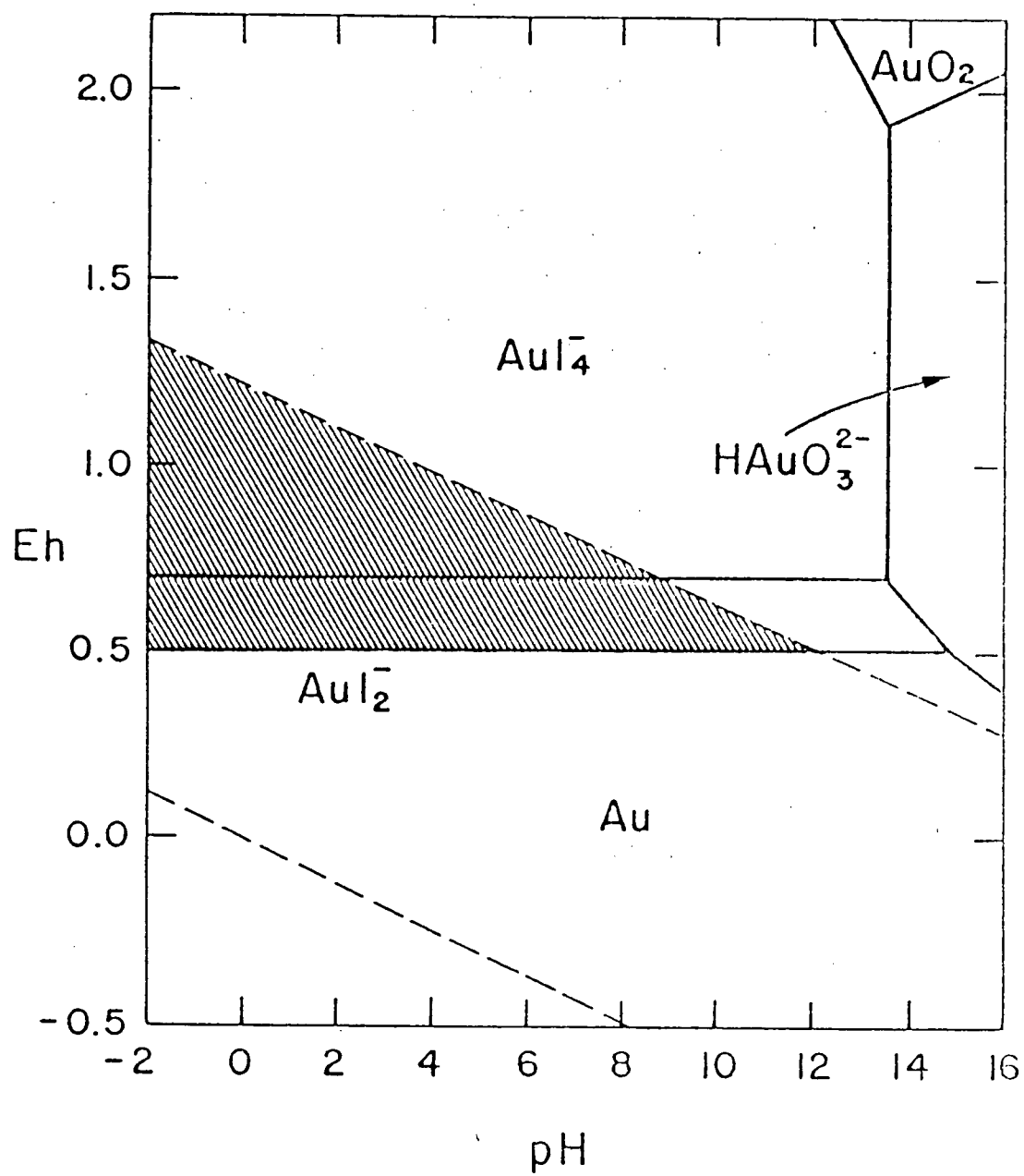
Figure 3.   Potential-pH equilibrium diagram for the gold-iodide system for [Au] = 10^{-5} M and [I] = 10^{-2} M

3.3   Adsorption

The use of carbon-in-leach (CIL) and carbon-in pulp (CIP) is well established (Ibrado & Fuerstenau). These techniques provide a large active surface area for the adsorption of the aurocyanide complex and can compete with carbonaceous refractory ore preg-robbing constituents (Guay 1981, Nendick). CIL has been effective where the refractory ore contains active carbon by competing for the gold complex as it is formed. Both techniques are useful when humic or fulvic acids are the problem since these vegetable source acids may complex gold and are in turn adsorbed by the activated carbon (Lee et al.).

Alternate competing adsorbents for the recovery of gold from carbonaceous gold ore would include various ion exchange resins suitable for use in resin-in-leach circuits. The resins are not prevented from use in alternate lixiviant systems since thiourea and halogen complexes can be adsorbed as well.

3.3.1   Resin-in-leach

Strong base quaternary amines have been tested for use in treating double refractory ores where cyanide is used as a complexing agent (Hiskey & Atluri 1989). The results suggest that the resin competes effectively with the carbonaceous preg-robbing component of the ore and only the portion of the gold that remains uncyanidable is lost to tailings. Several resins are available for gold adsorption and can likely accomodate the various natures of the oxidized gold complex solutions that

may result from ore specific treatments (Green et al.). This would indicate the use of competing resins in slurries where the oxidative method used to liberate gold from a sulfide matrix has activated a carbonaceous component causing preg-robbing.

3.3.2 Carbon-in-Chlorine Leach

Research done by the U.S. Bureau of Mines examined the carbon-in-chlorine leach (CICL) concept to combine an oxidative pre-treatment with lixiviation and recovery from solution (Greaves et al.). The conclusion was that some sulfide oxidation and preg-robbing deactivation can take place in the chlorine leach but that the carbon adsorption is not efficient in the uncontrolled potential regimes experienced. It may be possible to design a process where the potential and pH is optimized at the end of the leach stages to maintain the chloro-gold complex in solution until contacted with carbon in a carbon-in-chlorinated pulp (CICP).This would conserve oxidizing potential for the leaching stage and avoid the consumption of oxidant in the adsorption stage.

4.0   SCOPE AND OBJECTIVES

The description of a gold ore as refractory depends simply on the ease by which its precious metal content can be extracted with conventional cyanidation. The common benchmark used by industry is 80% gold extraction. This means that the reasons for which gold is not extractable can still be present and account for the remaining 20%. The literature search identifies many reasons for gold retention.

It is reasonable for the ore processor to assess treatments on the basis of economic feasibility in the pursuit of higher gold extractions. The assessment process can involve extensive testwork of any of a variety of treatment processes discovered in the literature search. It is apparent that different processes can apply to different ores even those within the various zones of a single orebody.

The following testwork will attempt to develop diagnostic approaches to differentiating the multiple reasons for refractoriness in a selection of sulfidic and doubly refractory carbonaceous ores. The approach uses a comparison of gold extraction by cyanide to determine the effectiveness of any particular treatment. The conditions under which the treatments are applied are derived from the literature and are not generally constrained by reagent consumptions nor are they optimized except in a general fashion. The treatments include pyrometallurgical and hydrometallurgical pretreatments for gold liberation as well as gold dissolution tests with various lixiviants.

5.0 RESEARCH RESULTS AND DISCUSSION

5.1 Feed Characterization

The ore received for the purposes of experimentation was represented by drillcore, blasthole and millfeed samples exhibiting various degrees of refractory behaviour. The samples represented two distinct minesites and encompassed varied subtypes from those locations. The characterization of the samples involved analysis of the pertinent elements, some chemical compound approximations by assay and inference, a consideration of mineralogy, an examination of physical state and finally diagnostic leaching to estimate the level of refractory behaviour and reagent consumption. From this feed characterization an experimental plan was developed to examine the response of the varied ore types to a variety of conventional and novel treatments intended to improve the extraction of gold.

5.1.1 Sample Identification

The first sample received came from the Cordex Exploration Company and was identified as drillcore D-231 from the Secret Pass zone of the Daisy property in Nevada. It was divided into selections of five foot mineralized intersections of drillcore from the 340 to 410 foot range. Further samples came from the Pinson Mining Company in Nevada and included mill feed from operations on June 9, 1993 and two blasthole samples identified as BH 2841 and BH 2844.

The samples were sorted and five were chosen for treatment on the basis of inferred mineralogy and grade of precious metal with overall consideration for the refractory nature of the ore. The Secret Pass drillcore represented a very refractory, low carbonate, low organic carbon, moderately high sulfide content ore. The two mineralized intersections chosen for treatment represent low and higher grade gold samples of the overall deposit and are identified in Table 4 by their drillcore interval (SP340, SP405). The Pinson mill feed is a composite sample containing high carbonate carbon, significant organic carbon, somewhat less sulfide than the drillcore samples and exhibits mildly refractory behaviour (Pinson). The blasthole samples have varied carbon levels with significant carbonate, moderate sulfide, widely differing gold contents and moderate refractory behaviour (2841, 2844).

The carbon assays were performed with commercial Leco induction furnace equipment using infrared detection for total carbon and carbonate carbon with the difference assigned to organic carbon. Total sulfur and sulfate sulfur were determined gravimetrically. Sulfide sulfur was determined with the use of a Leco induction furnace with a titrimetric analyzer using weak acid leached (sulfate depleted) samples.

Table 4. Refractory Ore Head Analysis

Sample	Carbonate Carbon %	Organic Carbon %	S^{2-} %	SO_4 %	S total %	CN Extract %	CIL Extract %
SP340	-	0.01	3.83	0.18	3.89	2.6	
SP405	-	0.07	3.69	0.15	3.87	13.3	
Pinson	2.92	1.35	1.33	-	1.35	71.8	
2841	1.52	3.78	1.45	0.03	1.43	74.7	79.2
2844	1.53	0.64	1.20	0.09	1.28	66.5	67.0

The cyanidation results (CN Extract) and cyanidation with carbon results (CIL Extract) are from diagnostic cyanide leaches with five gram per litre sodium cyanide solutions. The limited extractions confirm the refractory nature of the ores although only two can be considered extremely refractory.

5.1.2   Head Sample Analysis

Sample analysis involved blending and riffle splitting of the finely crushed bulk samples into one kilogram charges for subsequent rod mill grinding. Further splitting of these minus 3/8 mesh feed samples produced 250 gram splits which were pulverized with steel media and analyzed by conventional means with highlights of the results recorded in Table 5.

Table 5. Elemental Ore Analysis

Sample	Ag ppm	As ppm	Au ppm	Ca %	Cu ppm	Fe %	Hg ppm	Mg %	Sb ppm	V ppm
SP340	0.4	728	1.3	0.28	29	4.19	1	0.04	6	4
SP405	1.6	1195	3.6	0.48	30	3.9	3	0.09	12	10
Pinson	0.8	208	4.49	2.28	94	2.06	11	0.06	6	59
2841	0.6	148	2.13	6.07	573	1.75	5	0.09	4	38
2844	0.6	602	6.99	2.41	123	2.15	17	0.64	8	41

From the elemental analysis of the feed samples by inductively coupled plasma (ICP), some conclusions could be drawn as to the nature of the ore mineralization. In all cases, silver represents less than twenty five cents per tonne of ore and is of minor economic consideration. The arsenic may provide a clue as to whether the presence of arsenopyrite contributes to the low gold recoveries. Assuming that all the arsenic exists as arsenopyrite leads to the results recorded in Table 6 which indicate that only a small percentage of the sulfide sulfur can exist as arsenopyrite. It is plausible that the gold can be associated with an arsenious sulfide phase if the relationship seen in some South African ores is duplicated (Claassen). The gold values in the various ores range from $15-88 US per tonne (at $300 US per oz.) and this suggests that a low cost, simple process will be required to economically extract the precious metal values. The calcium content is an indicator of the potential for acid consumption in any low pH treatment when it is present as a carbonate and becomes a nuisance when it leads to the precipitation of gypsum in processes in which oxidation of sulfide to sulfate is necessary. Table 6 estimates the maximum calcium carbonate content of each ore. The copper content of these ores is generally low and should not contribute significantly to cyanide

complexation or oxidation and is likely to be of limited use in copper catalyzed processes. The mercury does not impede the recovery of gold to any great extent but becomes an environmental consideration in choosing an extractive process since levels in excess of 10 ppm in two of the samples will require attention in a commercial process. The magnesium content is often an indicator of dolomite and impacts a process where the rate of neutralization of acid is significant since dolomite is typically less reactive than calcite. The dissolved magnesium in hydrometallurgical processing of sulfides often contributes to the recirculating load of metals in any closed circuit acid process due to the solubility of the sulfate. The antimony levels are enriched with respect to the average earth's crust level of 1 ppm but are still low and would require an intensive search by microprobe to show any passivation of gold particle surfaces or any compound formation that may affect gold extraction with cyanide. The consequence of this evaluation is that the ores contain a number of elements that can contribute to refractory behaviour but they are present in relatively minor amounts.

Table 6. Derived Mineralogy

Sample	FeAsS max %	S_{FeAsS} / S^{2-} %	$CaCO_3$ max %
SP340	0.16	0.8	0.7
SP405	0.26	1.4	1.2
Pinson	0.05	0.7	5.7
2841	0.03	0.4	12.8
2844	0.13	2.2	6.0

5.1.3   Mineralogy

The higher levels of contained sulfides in the drillcore samples permits an examination of mineralogy through the use of a Siemen's Kristalloflex X-ray diffractometer (XRD) which results in the scans appended as XRD SCANS. The method suffers from a lack of resolution for minerals present in amounts less than 5%. The major minerals proved to be quartz and potassium feldspar. The predominant sulfide is pyrite and due to the resolution limits no other sulfides are readily identifiable. A comparison of the SP340 and SP405 patterns shows the possibility of a clay component in the SP405, most likely kaolinite and clinochlore.

A sample of the SP405 ore was ground to a P_{80} of 78 microns in a steel rod mill and a stirred slurry cut was taken for examination under a Carl Zeiss reflecting light microscope. The sample preparation involved panning out the higher density minerals to produce a gravity concentrate of about 5 grams from a 50 gram sample. This concentrate was then mounted in epoxy and polished in stages ending in a one micron diamond paste polished finish. Examination at 128x magnification confirmed the presence of pyrite, quartz and silicate minerals such as potassium feldspar. There was minor evidence of hematite rimmed pyrite indicating that there had been only limited oxidation and this may indeed have been an artifact of the sample preparation. Infrequent occurrences of well polished whitish grains that could be arsenopyrite were encountered. The pyrite existed as mostly liberated anhedral and euhedral grains but there was some evidence of smaller pyrite grains trapped in silicate gangue. Any gold associated with the encapsulated pyrite would be unavailable for cyanide extraction.

5.2   Pretreatment

5.2.1   Experimental Design

To test the amenability of oxidative treatments for the modification of refractory gold ores it is reasonable to first apply conditions that are, as much as possible, unconstrained by reagent consumption or kinetics to help identify the types of refractory behaviour. Physical occlusion is reduced by grinding to a practical degree of fineness generally 80% passing a 200 mesh (74μ) screen. A highly oxidizing pressure leach of the Redox variety ensures sulfide oxidation is complete and provides a comparison for the likely more feasible partial oxidation treatments. Carbon activity and the degree of preg-robbing responds variably to hydrometallurgical treatments but can be eliminated in a dry thermal pretreatment or oxidizing roast.

A variety of possible approaches for the treatment of the ores was chosen for testing and is represented in Figure 4. Standard ore preparation methods provided feed stock for the various pretreatments. Comparison of the effectiveness of the pretreatments was made using a diagnostic cyanidation method that somewhat masked the effect of residual cyanicides by using a generous dose of cyanide. This largely determined the comparative degree of gold liberation for the pretreatment methods. Alternate lixiviants including thiosulfate and the halides were tested for gold extraction from ground ores, oxidized residues, and in some cases the oxidation pretreatment pulps. This was

to aid in the investigation of combined oxidation and lixiviation in a continuous process without filtration.

Stirred tank tests were conducted using a variety of oxidizing agents to reduce the activity of carbonaceous fractions and liberate gold from the sulfide matrix while removing oxygen consumers. Some methods provide oxidation potential without direct complexing or lixiviating ability (Redox, peroxide, persulfate) while others combine the oxidation reaction for sulfides with the ability to directly complex in-situ liberated gold (hypochlorite/chloride).

The general procedure for the hydrometallurgical leach tests was to agitate slurries adequately to ensure solids suspension. This was typically 250-400 rpm for both Parr autoclave tests and glass resin kettle or beaker tests where solids were suspended but splashing was avoided. Acid additions generally started with any indicated sulfuric acid first followed by heating to the desired temperature and then adding the indicated oxidant. Nitric acid addition to high temperature tests was done through a pressurized stainless steel 'bomb' through which oxygen could be passed to ensure full reagent addition. Nitrogen and oxygen used for headspace makeup or air displacement were medical grade. Acid consumption, when quoted on a weight to weight basis with respect to ore, refers to bottle strength acid.
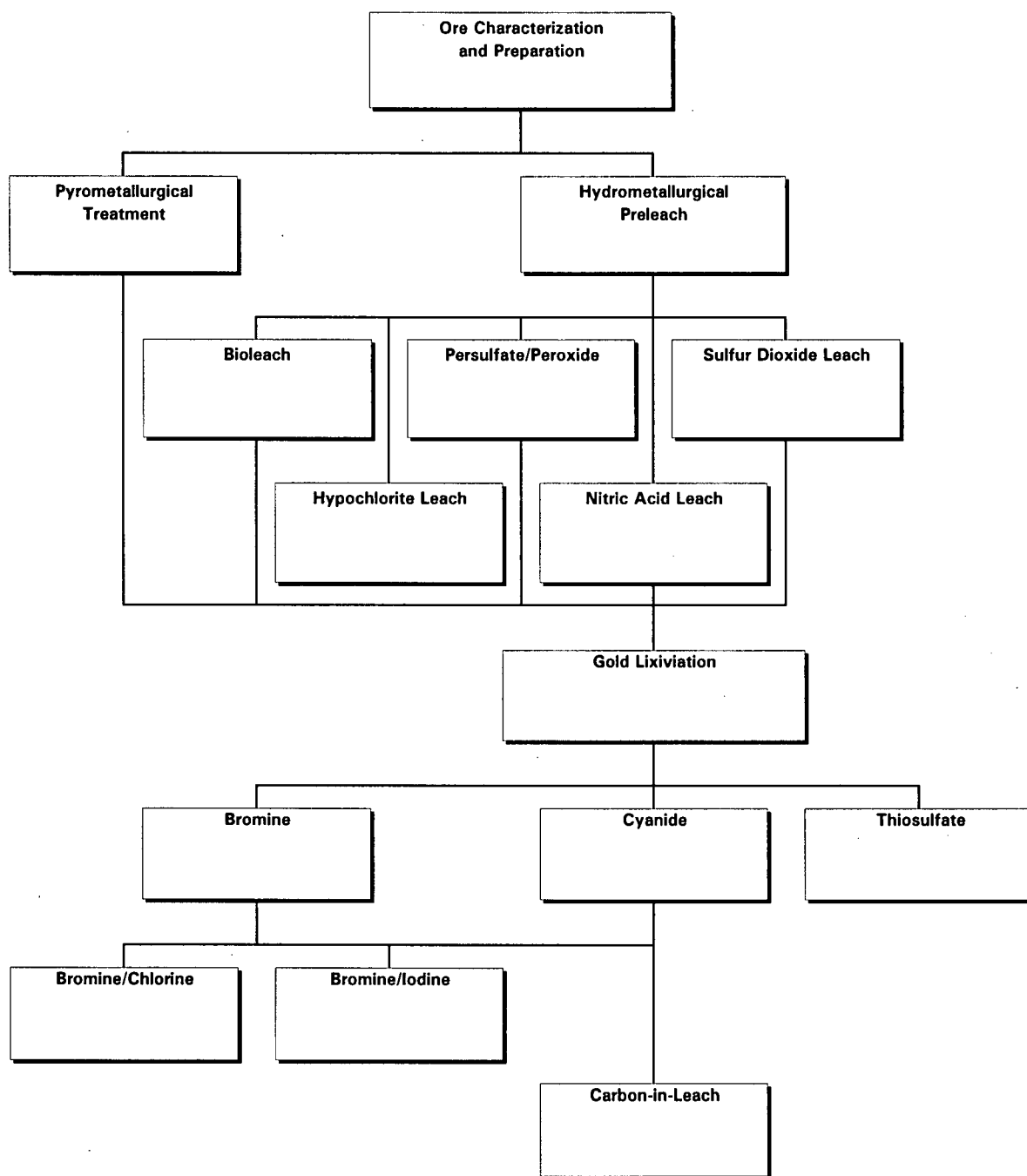
Figure 4. Experimental Plan

5.2.2 Grinding

The ore samples were cone crushed to one quarter inch size and then split into 1 kilogram charges for wet grinding. A steel mill with an approximately 30 kilogram rod charge was used to grind one kilogram charges of each of the ore samples at 66% solids. The ground pulps were washed out of the rod mill using Vancouver tapwater, settled and decanted to approximately 50% solids. Duplicate stirred samples were analyzed for particle size and the remaining slurry stored in sealed containers. The target was to achieve the common mill grind of 80% passing a 200 mesh Tyler screen. The results in Table 7 are averages of the indicated test grinds and on the whole ranged from 69 to 84% passing a 200 mesh screen for the samples used in the testwork. The one exception is the intentionally fine grind of the SP405 ore (G9) used to compare mechanical liberation with the oxidation methods.

Table 7. Grind Data

Test #	Feed	Grind Time min	P_{80} μ	-#200 %	Au g/t
G1	SP340	15	131	63.0	1.3
G2,G3,G4,G18	SP340	20	88	69.0	1.3
G5,G7,G17	SP405	20	78	76.9	3.6
G9	SP405	30	37	96.2	3.6
G8,G10	Pinson	20	71	81.7	4.5
G11,G13,G15	2841	20	63	85.0	2.1
G12	2844	20	50	90.0	7.0
G14,G16,G19	2844	18	62	84.0	7.0

The fine grinding of ores does not always result in an improved gold extraction. The sub-micron gold in many refractory ores remains occluded by the sulfide matrix or gangue minerals. There is the further complication of adsorption of dissolved gold on gangue minerals, particularly clays that can provide adsorptive surfaces for the gold complex. All of the direct cyanidations are summarized in Table 8. The tests were performed at 6% pulp solids and 5 gram per litre sodium cyanide additions.

Table 8. Cyanidation of Ground Ore

Feed type	Feed grind	P80 μ	Test #	CN Ext %
SP340	G4	88	CG4	2.6
SP405	G5	78	CG5	13.3
SP405	G9	37	CG9	22.9
Pinson	G8	71	CG8	71.8
2841	G11	63	CG11	74.7
2844	G12	62	CG12	66.5

Grinding the SP405 ore to a P_{80} of 37 microns gained a further 9.6% gold extraction. This is not likely to improve to a feasible level of extraction by further grinding alone.

5.2.3   Diagnostic Lixiviation

Direct cyanidation of the various ores using a diagnostic procedure confirmed their refractory nature. The cyanidation conditions used throughout this testwork were 5 grams per litre of sodium cyanide at 6% solids density and pH maintained between 10.5 and 11 with 10% lime slurry with any variations noted along with the extraction results. The length of the tests ranged from 18 to 22 hours

depending more on the time to achieve a steady pH in excess of 10.5 than concern about the rate of gold extraction in the last few hours of the leach.

Carbon-in-leach (CIL) tests used 1.0 dry gram (typically 33 kg/tonne of ore) of Norit extruded peat source carbon rods added in a water saturated form to ensure activity. These rods resisted attrition and were easily removed from the leached pulps with a 20 mesh screen. These leaches could also be referred to as carbon-in-pulp (CIP) tests when carbon additions occur sometime after the cyanide is added. The active carbon addition process was used to compete with natural preg-robbing components, particularly in the organic carbon containing ores, and provides a qualitative assessment of adsorptive activity. The summary of unoxidized ore tests in Table 9 suggests that marginal improvement in gold recoveries can be expected for the 2841 and 2844 ores with the use of active carbon in the leach.

Table 9. Ore Cyanidation Reagent Consumption

Feed type	Test #	CN Cons. kg/tonne	Lime Addn. kg/tonne	CN Ext. %	CIL Ext. %
SP340	CG4	31.4	4.4	2.6	
SP405	CG5	31.9	1.5	13.3	
Pinson	CG8	24.3	2.2	71.8	
2841	CG11	14.0	2.2	74.7	
2841	CIPG11	16.6	2.2		79.2
2844	CG12	12.5	2.2	66.5	
2844	CIPG12	11.9	2.9		67.0

The high cyanide consumptions (CN Cons) for all of the tests on untreated ore are partially an artifact of the test conditions since the solution cyanide strength is high for the pulp density used.

The consumptions are determined by titrating the free cyanide and weak acid dissociable cyanide availale at the end of the test with silver nitrate. These conditions were used to assure residual free cyanide for overnight tests. The exceedingly high cyanide consumptions for the SP340 and SP405 ores reflect their high sulfide content and indicate that cyanide is being complexed by gangue mineral solutes, adsorbed by clay components and /or reacting to form cyanates and thiocyanates. The lime additions reflect the amount of 20% finely ground lime slurry that was used to ensure a pH in excess of 10.5 before cyanide was added to the leach. The stated quantities are reasonable and are useful as a baseline for comparison to the added expense engendered by acid pretreatment neutralizations.

5.2.4 Bioleach Oxidation

The oxidation of the refractory gold ores by a mixed culture of *Thiobacillus ferrooxidans, Thiobacillus thiooxidans* and *Leptospirillum ferrooxidans* in both shakeflask and stirred reactor configurations was undertaken. Bioleach oxidation has been applied successfully in stirred tank reactors and is being developed as a low cost heap leach option.

Shakeflask tests used 30 gram charges of the wet ground refractory gold ore feed, 100 grams of 9K nutrient solution as per the formula published by Silverman and Lundgren [1959] (Torma) sufficient quantities of 6M H_2SO_4 to lower the slurry pH to less than 2.7 and a measured charge of inoculum resulting in approximately 20% solids slurries. The primary inoculum consisted of mixed culture grown on gold-free pyrite concentrate in 9K nutrient solution at 10% solids. Sequential tests allowed

the use of active culture grown on the refractory gold ores themselves to inoculate subsequent tests. The tests were stirred and incubated at 35°C in a rotary shaker. The oxidation-reduction potential (Eh) and the hydrogen ion activities (pH) were monitored using silver chloride reference electrodes with platinum or glass working electrodes respectively. Table 10 summarizes the measured variables of the shakeflask tests.

Table 10. Shakeflask Bioleach Parameters

Test #	Feed type	Feed grind	Feed g	9K g	H$_2$SO$_4$ 6M ml	Inoculant	Time days	Temp C	pH init.	pH final	Final Eh mv SHE
B1	SP340	G4	30	100	0.0	Pyr	12	35	2.13	1.52	733
B2A	SP405	G5	30	100	0.0	Pyr+10ml B1	22	35	2.63	1.16	670
B3A	SP405	G5	30	100	0.0	Pyr+10ml B1	22	35	2.63	1.19	673
B4	SP340	G4	30	100	1.0	Pyr	21	35	1.54	1.40	437
B5	SP405	G7	30	100	0.0	Pyr	26	35	1.78	1.03	738
B6	Pinson	G10	30	100	0.3	Pyr	26	35	1.95	1.45	733
B7	2844	G14	30	100	2.0	Pyr	18	35	1.93	1.33	740
B9	SP405	G17	30	100	1.0	Pyr	54	35	1.37	1.27	695
B10	SP405	G17	30	100	0.0	Pyr	17	35	2.35	1.19	701

The first three tests treated the sulfidic ores and when the biological activity lag period reached 12 days for the higher grade (SP405) feed the tests were re-inoculated with the active culture growing on the lower grade sulfidic ore (test B1 using SP340). Subsequent tests with the sulfidic ores were acidified to a pH below 2.5 before inoculation.

Larger tests using 200 grams of refractory gold ore feed were performed in a covered, heated, one litre resin kettle with air injected below the agitator. Each test was inoculated with the entire pulp of a shakeflask test conducted using the same refractory gold ore feed. The larger size of the stirred-tank tests allowed samples to be taken at intervals to attempt to correlate residence time and the

measured solution parameters to improved gold extraction. Table 11 summarizes the measured variables of the stirred-tank tests.

Table 11. Stirred Tank Bioleach Parameters

Test #	Feed type	Feed grind	Feed g	9K g	H_2SO_4 6M ml	Inoc.	Time days	Temp °C	pH	Eh mV SHE
B8	2844	G16	200	600	21.0	B7	0	35	2.65	
B8.1							7	35	2.30	417
B8.2							20	35	1.99	638
B8.3							33	35	1.96	718
B11	SP340	G18	200	600	0.0	B10	0	35	1.94	
B11.1							12	35	1.56	678
B11.2							19	35	1.30	730

The lag period varied for the ore types and even varied over the drill core intervals in these unoptimized tests. All samples demonstrated bioleach activity after lag periods of 2 to 11 days. Figure 5 shows a typical pH and solution potential response to bacterial activity. The diagnostic cyanidation results for the bioleached, filtered and washed residues are summarized in Table 12.
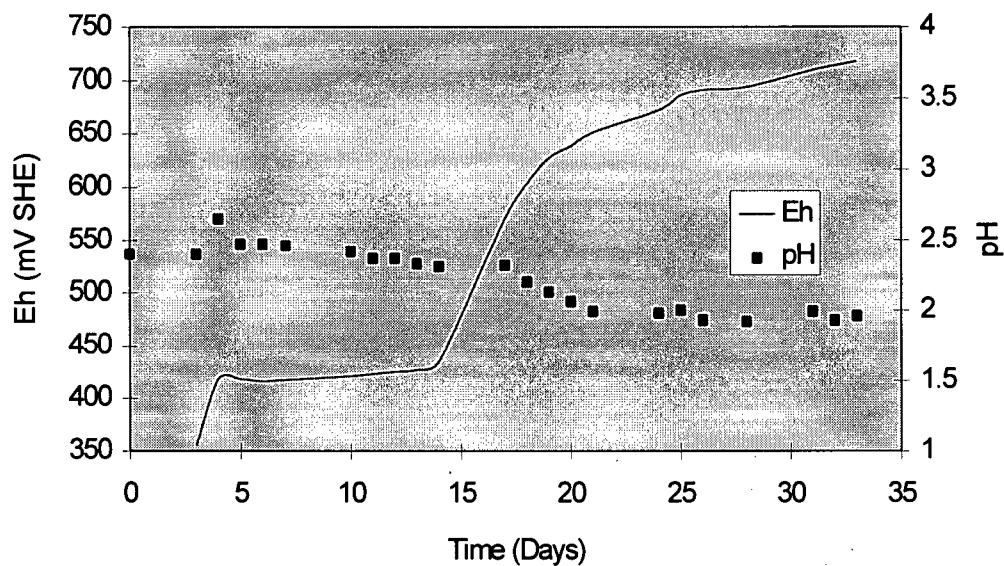
Figure 5.   Carbonaceous ore stirred tank bioleach

Table 12. Bioleach Results

Test #	Feed type	Time days	Temp C	pH final	Eh mV SHE	CN Ext %	CIL Ext %	Fe ppm
B2A	SP405	22	35	1.16	670	98.1		
B5	SP405	26	35	1.03	738	99.0		
B6	Pinson	26	35	1.45	733	83.2		
B8.1	2844	7	35	2.30	417		85.6	6750
B8.2	2844	20	35	1.99	638		86.0	4130
B8.3	2844	33	35	1.96	718		85.0	
B11.1	SP340	12	35	1.56	678		94.7	
B11.2	SP340	19	35	1.30	730		94.7	

The SP405 ore contains the highest levels of arsenic at 1195 ppm and has the greatest chance of

recovery improvement by bioleaching due to the preferential attack on arsenopyrite and the common

association of gold with the arsenic in refractory gold ores. The ore contains insignificant carbon

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and with no tendency to preg-rob the cyanide gold extractions are spectacular. Similarly the SP340 ore has an improvement in extraction from 2.6 to 94.7% at 12 days in the bioleach reactor.

Pinson ore showed a moderate improvement from 71.8 to 83.2% gold extraction suggesting that optimization of the bioleach conditions may be necessary or that the carbon component is active and unaffected by the bioleaching pretreatment. The related 2844 carbonate ore showed significant improvement (from 66.7 to 85.6% gold extraction) after 7 days of leaching and achieving a pH of 2.3 and a potential of 616 mV SHE. These conditions suggest only partial oxidation of the pyrite. Further oxidation coupled with oxidation product precipitation over a further 26 days resulted in no significant change in gold extraction. The solution iron had dropped from 6.7 to 4.1 grams per litre by the 20th day and the pH had levelled out at 2.

5.2.5 Thermal Pretreatments

Considerable use has been made of pyrometallurgical techniques in the treatment of refractory gold ore. The complete oxidation of both the sulfide and carbon components often eliminates all of the refractoriness of an ore, although occlusion of gold in dense hematite calcines is possible in roasts performed at temperatures greater than 800°C (Osmanson). This is an example of process induced refractory behavior and can usually be solved by an attritioning or grinding treatment after roasting.

Since properly designed roasters are very effective at converting refractory gold ore into a feed amenable for cyanidation, the feasibility of this technology is determined by environmental and to

a lesser extent, energy costs. The scrubbing of roaster gasses for the removal of SO_2 is a problem associated with thermal treatments that have an oxidizing component. As well, any thermal process must address the volatilization of toxic metals such as mercury, selenium and arsenic. The required development of higher efficiency scrubbers and improved electrostatic precipitators is part of the environmental cost of roaster operation.

A phenomenon observed in the non-oxidative roasting of auriferous pyrite and arsenopyrite is the migration of gold. Gold particles in the sub-micron size range coalesce and become detectible by scanning electron microscopy in the roaster product (Swash & Ellis). This would suggest that a non-oxidative thermal process may be successful in liberating gold from a sulfide matrix by either conversion of that matrix or effective liberation of the gold. The volatilization of sulfur from pyrite would result in a pyrrhotite or troilite product containing coarser gold particles. This new sulfide matrix is likely to be less refractory without further treatment but can also be dissolved readily in a mild acid leach prior to an alkaline cyanide gold extraction.

To test this concept a tube furnace was built to allow roasting to be conducted in the absence of oxygen. Iron filings were added to possibly adsorb or combine with the liberated sulfur in the production of pyrrhotite from pyrite. The roast was conducted in alumina sample boats.The choice of temperature and roast time at temperature is guided by research that suggests pyrite rapidly converts to pyrrhotite under inert atmosphere at 600-650°C (Grimsey & Aylmore). Further, direct conversion of arsenopyrite and pyrite to magnetite or hematite does not allow gold coalescence and tends to occlude the fine gold. The best conditions would convert the auriferous sulfides to porous

pyrrhotite or troilite with a magnetite rim and coalesced gold blebs to provide leachable gold and reduced pyrrhotite reaction with cyanide. If sulfur is available as SO_2, troilite can convert to pyrrhotite at 330°C on cooling. Between 560 and 590°C pyrite converts to troilite under inert or low sulfur atmospheres (Hausen).

Carbon content in the ore requires oxidation before appreciable sulfur oxidation occurs (Chen & Reddy). Roasting in the presence of lime at a temperature of 500°C results in the retention of sulfur as calcium sulfides, sulfites and sulfates while there is evidence that the transition to pyrrhotite still occurs (Hagni et al.).

Organic carbon components including humates are mostly pyrolized by 350°C generating varying amounts of carbon dioxide and low molecular weight hydrocarbons depending on the available oxygen. The carbon dioxide of calcium carbonate begins to dissociate at temperatures as low as 550°C. This suggests that even dead roasts under inert atmospheres can remove problematic carbon content from the ore. Highly agitated oxidizing roasts are effective refractory gold ore pretreatments since the particle temperature is more closely controlled and kinetics are enhanced by better bulk gas/solid contact.

Table 13. Roasting Conditions and Results

Test #	Feed source	Feed	Temp °C	Time hr	Atmos	Feed g	Iron g	Res g	Wt Loss %	CN Ext %
T1	SP405	G7	500	4	N$_2$	30.00	3.13	32.62	1.54	52.4
T2	SP405	G7	640	4	N$_2$	30.05	3.00	32.37	2.06	50.5
T3	SP405	G7	600	19	air	41.00	0.00	39.58	3.46	73.3
T4	2844	G12	700	4	N$_2$	32.08	0.00	30.31	5.52	82.9
T5	2844	G12	720	18	N$_2$	32.55	0.00	30.76	5.50	81.5
T6	2844	G12	500	21	N$_2$	32.24	0.00	31.63	1.89	77.1
T7	2841	G11	750	4	N$_2$	32.47	0.00	29.39	9.49	66.2

Tests of low carbon /high sulfide ore (SP405) were examined for enhanced pyrrhotite production by attempting to capture the pyritic sulfur on finely divided iron at temperatures where pyrite converts to pyrrhotite. The samples were purged, roasted and cooled under a nitrogen atmosphere. Gold extraction from the 500°C test residue slightly exceeded that of the 640°C residue. The X-ray crystallographic analysis using a Siemens D5000 diffractometer did not find much alteration in the low temperature residue, but indicated significant pyrite loss and the presence of troilite in the 640°C roast. Roasting SP405 for 19 hours at 600°C in the presence of air resulted in much improved gold extraction and X-ray analysis indicated residual pyrite, a possible ferric arsenate and no troilite.

Roasting the higher carbon/moderate sulfide ore (2844) for 4 and 18 hours under nitrogen at 700-720°C resulted in similar gold extractions but the 21 hour roast at 500°C suggests that carbon content impedes the beneficial effects of roasting at lower temperatures. A high temperature non-oxidizing roast of the high organic carbon ore (2841) actually reduces the gold extraction possibly due to hematite occlusion resulting from pyrite oxidation by small amounts of CO as well as the activation of remaining carbon components as adsorption sites.

Further investigation may reveal that a thermal test under a non-oxidizing atmosphere can distinguish between organic carbon refractory ores that are thermally activated, arsenopyrite/pyrite refractory ores and naturally preg-robbing ores. This can be useful where crystallographic analysis is inconclusive due to low mineral content.

5.2.6 Nitric Acid Leach

The ambient pressure stirred tank version of the Redox process was designed for application to low grade ores primarily as a sulfide oxidizing method. The typical procedure is to use stoichiometric quantities of nitric acid oxidant based on the sulfide content under an inert atmosphere to achieve nearly complete oxidation without redissolving NO_2 formed in the headspace by contact of NO with O_2 (Equation (27)). The intent is to achieve nitrate free pulps. The pressure leach of low grade ore in a titanium Parr autoclave at temperatures exceeding 200°C is likely to achieve complete sulfide oxidation due to the recycling of the oxidation catalyst. The Redox process has variable results with active carbon. It is likely that humic substances are eliminated in the high potential, high temperature leach, but it is unlikely that oxidation of graphitic carbon is complete. Further, precipitation reactions can occlude precious metal as in the case of silver jarosite formation.

High temperature autoclave tests are summarized in Table 14. The acids used were ACS reagent grade and are quoted at bottle strength. A two litre Parr autoclave with titanium wetted parts contained the reacting mixture. Oxygen overpressure of 100 psi above the steam pressure ensured

high oxidizing potentials. Samples were taken after rapid cooling and filtering of the residues and leached using the diagnostic cyanidation method.

Table 14. High Temperature Redox Results

Test #	Feed	Solids %	HNO₃ kg/t	H₂SO₄ kg/t	Time hr	Temp °C	pH	CN Ext %
R1	SP340	20	460	80	0.5	195	<1	94.7
R2	SP405	20	460	100	0.5	195	<1	99.0

The two autoclave tests at high temperature and extreme acidity were used to treat the very refractory sulfidic ores. The residues were readily cyanided to very low tails suggesting that silicate occlusion of gold is minimal and that the refractory behaviour is due entirely to sulfide occlusion since little or no carbon is associated with the ore.

Table 15. Low Temperature Redox Results - Sulfidic Ore

Test #	Feed	Feed Grind	HNO₃ kg/t	H₂SO₄ kg/t	Time hr	Temp °C	pH	Eh Pt/AgCl	Solids %	CN Ext %
R3	SP340	G4	540	38	2.0	85	0.3	>800	26.4	97.4
R5	SP340	G4	540	38	2.0	85	0.2	900	39.2	97.4
R11	SP340	G18	540	38	0.5	85	0.0	715-800	28.5	97.4
R12	SP340	G18	540	38	1.0	85	-1.4	700-830	28.6	94.7

Low temperature, ambient pressure stirred tank tests using excess nitric acid to effect sulfide oxidation were universally effective at liberating the gold content. Although this process can generate elemental sulfur from arsenopyrite oxidation, the low arsenic content of the ore (728 ppm) ensures that the amounts would be small and according to these results, ineffective at occluding gold.

Sulfuric acid additions were based on acid consumption titrations to pH 1.5 at 60°C. This acidification ensured free acid availability in the early part of the leach. The low temperature Redox process leaches are conducted under a nitrogen blanket ostensibly to prevent re-entrainment of NO_2 as nitrate but all of the low temperature tests used excess nitric acid based on sulfide stoichiometry. Although reagent intensive, the Redox process results define the maximum gold extractions that can be achieved with sulfidic refractory gold ores.

Table 16. Low Temperature Redox Results - Carbonaceous Ore

Test #	Feed	TOC %	HNO_3 kg/t	H_2SO_4 kg/t	Time hr	Temp C	pH	Eh Pt/AgCl	CN Ext %	CIL Ext %
R4	Pinson	1.35	188	74	2.0	85	0.6	790	84.7	90.1
R7	Pinson	1.35	188	73	1.5	85	<1	400-690	90.1	
R7	Pinson	1.35	188	73	3.0	85	<1	400-690	84.0	88.5
R6	2841	3.78	225	149	2.0	85	1.0	735-775	70.2	72.6
R8A	2844	0.64	186	100	1.0	85	0.7	706		72.4
R9	2844	0.64	186	100	0.5	85	0.5	719		79.4

In leaching carbonate carbon containing ores in high acid, care must be taken to ensure adequate head space is available to contain the frothy pulp. The three carbon containing ores have similar sulfide content and are distinguished only by their range of organic/graphitic carbon (TOC) content. The Pinson ore is roughly the average of the other two and demonstrates refractory behavior midway between the others before treatment. The anomaly is that the lowest TOC containing ore (2844) is the most refractory. Behaviour such as this prevents simple carbon analysis from predicting the degree of gold retention. In general , shorter leach times in nitric acid pulps improved gold recovery

reflecting the destruction of the sulfide matrix effect on liberation. As leach times lengthen this ore type demonstrated reduced gold extractions by what appears to be a carbon activitation.

Pinson Millfeed Redox
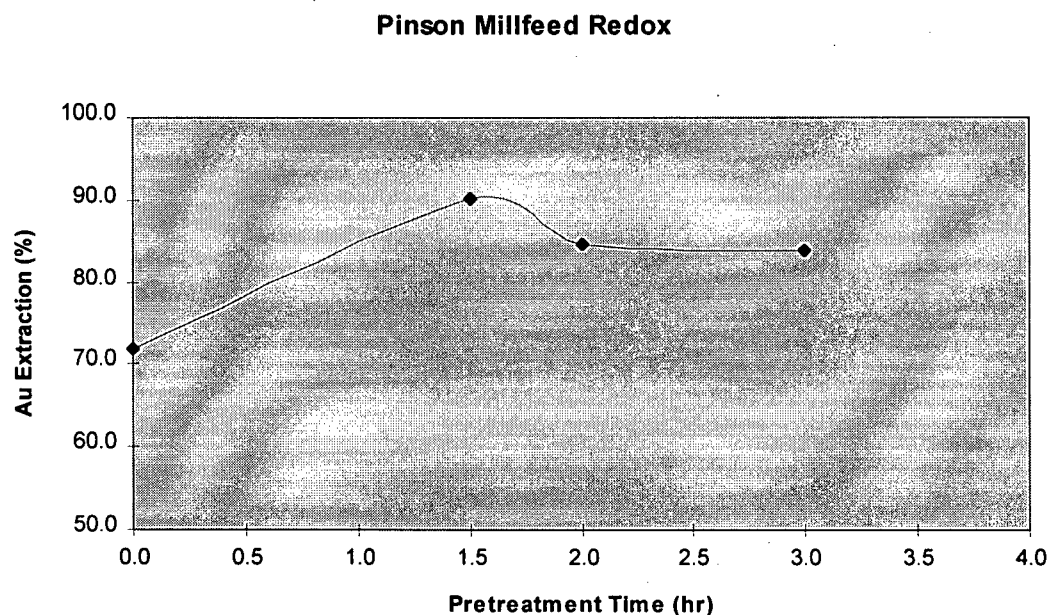
Figure 6.   Carbonaceous ore showing evidence of process activation

The nitric acid leach differentiates between sulfidic refractory and doubly refractory or active carbon containing ores. For sulfidic ores, gold that is not extracted after the Redox leach can probably be considered silica encased. For organic carbon containing ores, the lower extraction after longer leach times may be indicative of development of more active graphite or carbon rich organic components. Further examination of the carbon species after the Redox leach may clarify this relationship.

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5.2.7 Sulfoxy Species from Sulfuric Acid, Peroxide and Persulfate

The application of oxidizing potential to ore slurries by means of surrogates such as the nitrosyl ion in the Redox process can be extended to sulfoxy species. The thermodynamically predicted solution species resulting from sulfur oxidation are almost always sulfate in aqueous solutions due to the practically irreversable nature of sulfate production. In practice, a variety of metastable thio species exist in solution and can react with reduced sulfur present in the form of metal sulfides. The dissolution process is complex and is influenced by the conductivity of the sulfide, galvanic effects of mixed mineral phases and the genesis of metal depleted and passivated zones.

The oxidation of pyrite is thought to proceed through a thiosulfate intermediate which can disproportionate to elemental sulfur and sulfate. The mechanism by which this is possible can be developed by considering the semiconducting nature of pyrite. Semiconductors are characterized by valence bands and conduction bands separated by a quantum mechanically forbidden energy band or band gap. The energy required to promote an electron from the valence to the conduction band affects the mechanism of dissolution. Two types of charge carriers exist in semiconductors, electrons in the conduction band and holes in the valance band.

The electronic structure of pyrite allows a crystal field interpretation of the iron to sulfur bonding scheme (Figure 7). It is proposed that pyrite resists dissolution because the upper energy levels of its valence band are occupied by non-bonding t_{2g} molecular orbitals. A band gap of 0.9 eV exists between this upper valance band and the anti-bonding e_g^* energy level of the conduction band. This

suggests that electron transfer to the conduction band, which is equivalent to a hole injection into the upper valance band, cannot contribute to bond breaking in the solid (Crundwell) since it is non-bonding orbitals that are affected. The consequence of the crystal field splitting is that pyrite is less easily oxidized.
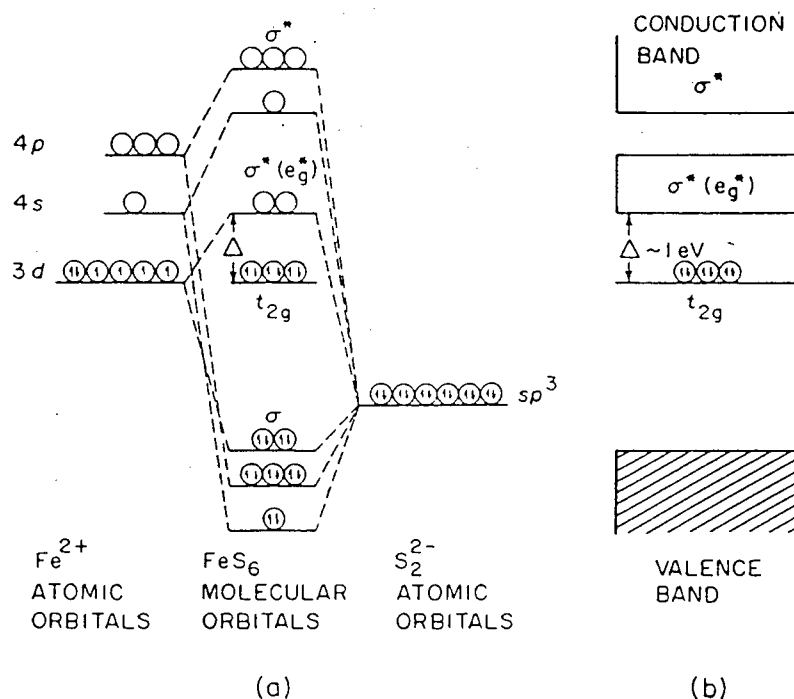
Figure 7.   (a) Relationship between atomic orbitals, molecular orbitals, and the band molecular orbital diagram for the FeS_6 cluster. (b) Band model for FeS_2 (from Osseo-Asare)

The dissolution of pyrite may begin with the injection of a hole into the t_{2g} valance band by an oxidant. This hole may react with water at the surface:

$$H_2O + h^+ = H^+ + OH\bullet \tag{45}$$

The hydroxyl radical formed is very reactive and the couple with the hydroxide anion has an estimated 2.95 volt SHE potential. This has sufficient energy to react with the bonding orbitals.

Mishra & Osseo-Asare propose the intermediate formation of an adsorbed thiosulfate species via a series of steps:

$$FeS_{2(S)} + H_2O + h^+ = Fe(OH)S_{2(S)} + H^+ \qquad (46)$$

This is a net adsorption of a hydroxide anion at a ferric iron surface site induced by the hole injection. This is followed by a migration to a sulfide lattice site:

$$Fe(OH)S_{2(S)} = FeS_2(OH)_{(S)} \qquad (47)$$

Surface hydroxylation:

$$FeS_2(OH)_{(S)} + 3H_2O + 3h^+ = Fe(OH)_2 \bullet S_2(OH)_2 + 3H^+ \qquad (48)$$
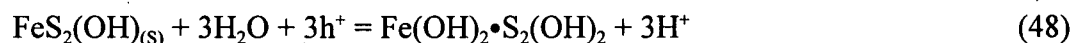
is followed by oxidation of the sulfide:

$$Fe(OH)_2 \bullet S_2(OH)_2 + 2h^+ = Fe^{2+} + S_2O_3^{2-} + 2H^+ + H_2O \qquad (49)$$
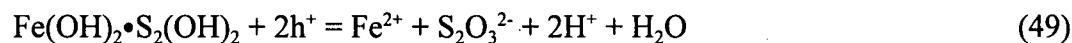
The net result is the production of an adsorbed thiosulfate which can disproportionate to elemental sulfur and a bisulfite anion:

$$FeS_{2(S)} + 3H_2O + 6h^+ = Fe^{2+}_{(AQ)} + S_2O_3^{2-}_{(ADS)} + 6H^+_{(AQ)} \qquad (50)$$

$$S_2O_3^{2-}{}_{(ADS)} + H^+ = S_{(ADS)} + HSO_3^-{}_{(AQ)} \tag{51}$$
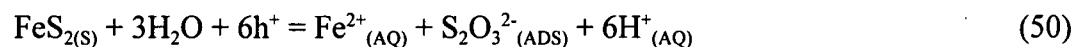
Since the thiosulfate can be oxidized to sulfate through polythionate and sulfite intermediates, this mechanism avoids the requirement of oxidizing any elemental sulfur to sulfate. If the thiosulfate formed by oxidation of pyrite by this route could be stabilized in solution then gold extraction could be effected by this in-situ lixiviant.

The potentials measured for some sulfoxy and peroxide species suggest that there is sufficient oxidation potential to dissolve pyrite and change the nature of carbon species. The use of Caro's (peroxomonosulfuric) acid formed by the cold addition of hydrogen peroxide to concentrated sulfuric acid, has been examined as an oxidant for sulfides (Lakshmanan et al.) and the use of Oxone powder containing peroxomonosulfate has been studied as an oxidative enhancement to the cyanidation of auriferous pyrite (Stoychevski & Williams). The oxidizing strength of sulfur dioxide/oxygen mixtures in the presence of transition metal ions was the subject of a study done of high pH metal oxidations that postulated the formation of salts of Caro's acid (Devuyst et al.).

The reduction potentials for peroxydisulfate and hydrogen peroxide are adequate for the purpose of sulfide oxidation as follows:

$$S_2O_8^{2-} + 2H^+ + 2e^- = 2HSO_4^- \qquad E^\circ = 2.12 \text{ V} \tag{52}$$

$$S_2O_8^{2-} + 2e^- = 2SO_4^{2-} \qquad E^\circ = 2.01 \text{ V} \tag{53}$$

$$H_2O_2 + 2H^+ + 2e^- = 2H_2O \qquad\qquad E^\circ = 1.78 \text{ V} \tag{54}$$
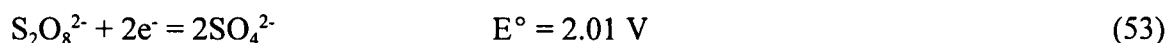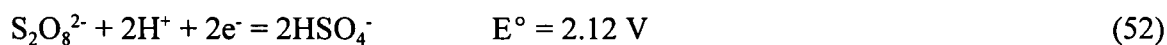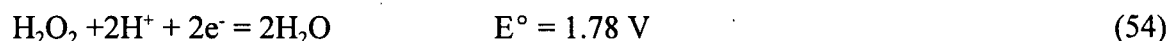
Hot concentrated sulfuric acid is an oxidizing agent according to:

$$5H_2SO_4 + 2e^- = H^+ + SO_2 + 4HSO_4^- + 2H_2O \tag{55}$$

The oxidation potential is lost in dilute solution. The addition of hydrogen peroxide to a sulfuric acid slurry may result in metastable sulfoxy species forming at the surface of a metal sulfide and leading to dissolution without elemental sulfur remaining to passivate the mineral surface.

To test this concept, ore samples were leached in titanium Parr autoclaves where an overpressure of oxygen or nitrogen in psi is indicated in Table 17. Tests in contact with air were done at ambient pressure in a stirred glass vessel. Hydrogen peroxide was added as a 30% solution in a quantity designed to maintain a potential in the range of 650 mV based on contact tests with silica sand in sulfuric acid slurry. The potentials achieved in the stirred pulps at pH 0-1 were certainly adequate to oxidize pyrite but the reaction kinetics are considerably lower than the nitric acid system.

Tests with the sulfidic ores (SP340, SP405) showed only minor improvement in gold extraction after a leach time of 45 minutes. The tests with Pinson ore established that gold extraction improvement was a result of the added oxidant and acidification, since an inert atmosphere test at 50°C lead to similar extraction improvements as those of a test under 100 psi overpressure of oxygen conducted

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over 2 hours. Similar tests with 2841 ore actually showed little or no effect under oxygen overpressure and slight improvement under nitrogen after 4 hours of pretreatment.

Table 17. Peroxide Oxidation

Test #	Feed	H_2O_2 kg/t	H_2SO_4 kg/t	Atm psi	Time hr	Temp C	pH	Eh mvPt/AgCl	CN Ext %	CIL Ext %
P2	Pinson	150	300	O_2#100	2.0	50	0.8	513	82.4	88.5
P3	Pinson	150	300	N_2#100	2.0	50	0.3	503	84.0	86.3
P4	2844	200	300	air	1.5	27	0.5	620-660		82.1
P5	2844	200	300	O_2#100	2.0	50	0.8	502	87.1	92.3
P6	2841	200	300	O_2#100	4.0	50	1.0	493	72.6	79.0
P7	2841	200	300	N_2#100	4.0	50	0.9	492	79.0	83.9
P10	SP405	200	300	air	0.75	22	0.6-1.2	480-620	28.6	33.3
P11	SP340	200	300	air	0.75	22	0.3-0.5	540-630	18.4	15.8
P12	2844	200	100	air	2.0	22	1.1-1.5	480-636		82.5
P15	2844	200	300	O_2#100	2.0	80	<0.4	510		86.8

A series of tests with the most refractory carbonaceous sample (2844) provides a cursory examination of the effects of temperature on gold liberation. The dotted line in Figure 8 indicates
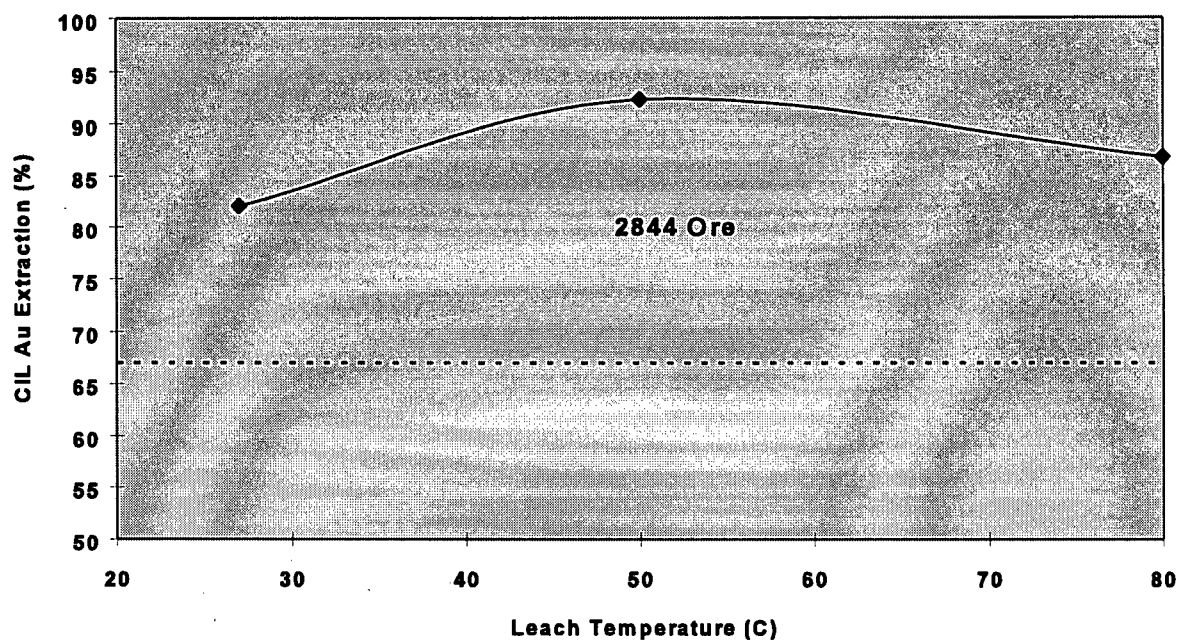
Figure 8.   CIL gold recoveries for 2844 ore after peroxide leaching

the extraction without pretreatment. Peroxide leaching appears to be beneficial in short contact time

tests (1.5 to 4 hours) with carbonaceous refractory gold ores. The acid addition was reduced to 100

from 300 kilograms per tonne in a 2 hour test in contact with air and the extraction by CIL was not

changed significantly.

5.2.8   Oxidation with Hypochlorite in Brine

Chlorination of gold ores was the primary recovery technique before the advent of cyanidation.

Roasting ores with chloride salts lead to chloride volatilization methods, leaching in high potential

brines with chlorine formed soluble gold chloride complexes, pressure leaching with hypochlorite and oxygen as a pretreatment before cyanidation and mild chlorine/hypochlorite pre-aeration to destabilize organic gold complexes in carbonaceous ores have all been developed on an ore specific basis. The use of chlorine to remove the sulfur content of coal is an effective example of pyrite oxidation. All of these methods suffer from loss of oxidant since the chlorine is non-specific in the substances it will oxidize. Chlorine dissolves in water according to:

$$Cl_2 + H_2O = HOCl + H^+ + Cl^- \tag{56}$$

At least one pathway for the chlorination of pyrite is:

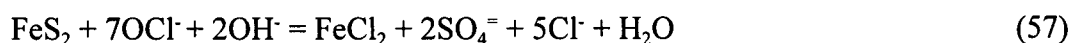$$FeS_2 + 7OCl^- + 2OH^- = FeCl_2 + 2SO_4^= + 5Cl^- + H_2O \tag{57}$$

In acid solutions the chlorine content is limited by the solubility of the gas, at high pH the chlorine is stabilized as the hypochlorite anion OCl^-. At high pH the metals are predominantly precipitated as their hydroxides or exist as oxyanions and are unavailable as surrogate oxidants. This suggests a process whereby the availability of chlorine oxidation potential can be combined with active metal oxidation reduction couples and likely increase the kinetics of oxidation of metal sulfides. A substance commonly used to retain 'free' chlorine in swimming pools at neutral pH is cyanuric acid. It exists as a triazine tautomeric ring structure with a stabilization analogous to a keto-enol formation. It has a greater resonance energy than a benzene ring and less tendency for the ring structure to enter into any reactions. Cyanuric acid or 1,3,5-triazine-2,4,6(1H,3H,5H)-trione

$((CNOH)_3)$ is characterized by the chemistry of the three functional groups attached to the C_3N_3 ring and is of interest in its chlorinated form. Treating ore in a pH range of 5 to 8 with chloride, hypochlorite and cyanuric acid is the subject of a patent (Baughman et al.).

Treatment of the carbonaceous refractory gold ores occurred in contact with air at ambient pressure or in a Parr autoclave under 50 psi of oxygen overpressure as noted in Table 19. The oxidant for this series of tests was calcium hypochlorite added as a solid so that pulp densities could be kept at 25% solids. All of the tests were filtered and washed, followed by cyanidation, to gauge the effect of the high oxidation potentials observed. The recorded extractions do not separately account for gold complexed in the chlorination stage as they were based on pretreatment feed assays.

Table 19. Chlorination of Carbonaceous Ore With Cyanuric Acid Added

Feed	Oxidant kg/t	$(CNOH)_3$ kg/t	NaCl kg/t	Additives	Additives kg/t	Time hr	Temp C	pH	Eh mvPt/AgCl	CN Ext %	CIL Ext %
2841	50	8.0	400	NaOH	12.0	20.0	45	7.4-8.9	300-750	77.6	79.1
2841	50	8.0	500	O_2	50psi	18.5	22	5.9	340	74.2	79.1
2844	50	8.0	400	NaOH	4.3	21.5	48	7.1-8.5	490-890	77.6	
2844	50	8.0	500	O_2	50psi	20.5	30			77.6	75.6
Pinson	50	5.0	100	NaOH/HCl	3/7.4	4.0	22	8.0	1000	77.9	
Pinson	50	8.0	400	NaOH	6.4	22.0	48	6.8-8.0	400-950	80.9	
Pinson	50	8.3	500	HCl/O_2	6.2/100psi	20.0	50	6.1-8.0	290	77.9	

Minor improvements are seen in the two most refractory ores (2844, Pinson). The extractions by cyanide and CIL are similar for the residues where both methods were used suggesting that some preg-robbing activity has been negated.

Tests using similar conditions and reagent additions for the sulfidic ores were unsuccessful in improving the gold extractions. The results were insensitive to chloride addition, time in leach, temperature and oxidant (Table 20).

Table 20. Chlorination of Sulfidic Ore With Cyanuric Acid Added

Feed	Oxidant	Oxidant kg/t	(CNOH)₃ kg/t	NaCl kg/t	Additives	Additives kg/t	Time hr	Temp C	pH	Eh mvPt/AgCl	CN Ext %
SP405	Ca(OCl)₂	116	4.17	0	NaOH	17.5	2	22	8.0	900	24.8
SP405	Ca(OCl)₂	123	4.17	200	NaOH	11.2	2	40	8.0	880	23.8
SP405	Ca(OCl)₂	116	4.17	0	NaOH	13.0	2	55	8.0	870	29.5
SP405	Ca(OCl)₂	46	8.33	0	NaOH	31.5	4	22	8.0	350	58.1
SP405	NaOCl	23	8.33	0	NaOH	10.7	4	22	8.0	920	39.0
SP405	Ca(OCl)₂	50	8.33	333	NaOH/HCl	6.8/13	4	22	8.0	990	27.6
SP405	Ca(OCl)₂	50	8.00	400	NaOH/HCl	6.7/7.5	22	22	6.5-8.0	1000	26.7
SP405	Ca(OCl)₂	50	8.33	500	HCl/O₂	9.1/100psi	20	50	1.7-8.0	503	39.0

The one test with an interesting result was the 20 hour leach under 100 psi of oxygen overpressure at 50°C. The final pulp was measured at 503 mV and a pH of 1.7 suggesting acid generation that would be expected to come from sulfide oxidation. The poor extraction result would suggest that considerable elemental sulfur was also formed and re-passivated the contained gold. The cyanidation test reagent consumption of 21.2 kilograms per tonne of ore for sodium cyanide is high but not unusual for these tests.

5.3   Lixiviation

5.3.1   Thiosulphate

The use of thiosulfate in the recovery of gold has been researched by many (Berezowski & Sefton, Langhans et al., Zipperian et al.) and is the subject of several patents (Berezowski et al. 1978, Kerley Jr. 1981 & 1983, Perez et al. 1987). Each patent further refines the operating conditions for a successful refractory gold ore treatment with the tendency towards higher pH as time passes. As the pH increases, so too does the influence of the ammonia component as a gold lixiviant.

A major benefit of the use of thiosulfate is the observation that the highly negatively charged complex does not adsorb readily on active carbon. The immediate benefit is the elimination of preg-robbing characteristics for carbonaceous ores. The conditions suggested for the thiosulfate leach are likely to have a destructive effect on unstable sulfides and this may be adequate to solve the sulfidic gold occlusion effect in some doubly refractory gold ores. The reactions of interest in this system are:

$$4Au + 8S_2O_3^{2-} + O_2 + 4H^+ = 4Au(S_2O_3)_2^{3-} + 2H_2O \qquad (58)$$

$$Au + 5S_2O_3^{2-} + Cu(NH_3)_4^{2+} = Au(S_2O_3)_2^{3-} + 4NH_3 + Cu(S_2O_3)_3^{5-} \qquad (59)$$
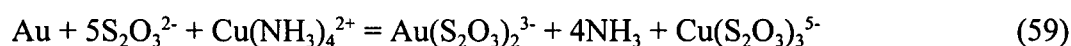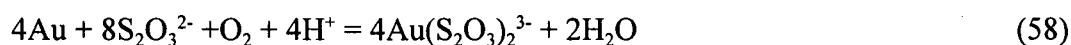
The cupric tetrammine complex acts as an oxidant for gold and the Cu^+/Cu^{2+} takes part in an equilibrium of complexed species.

$$2Cu(NH_3)_4^{2+} + 6S_2O_3^{2-} + H_2O = 2Cu(S_2O_3)_3^{5-} + 8NH_3 + 0.5O_2 + 2H^+ \qquad (60)$$
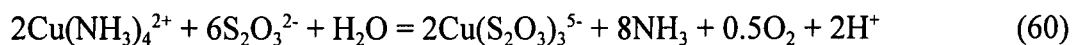
Sulfite added to the system generates thiosulfate from soluble sulfide and possibly metal sulfide surfaces with the generation of elemental sulfur.

$$3SO_3^{2-} + 2S^{2-} + H_2O = 2S_2O_3^{2-} + 6OH^- + S \qquad (61)$$
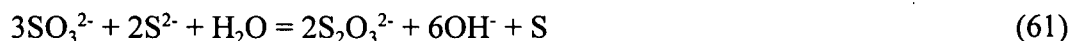
Although written here as an oxidation that removes gold lixiviant strength, the reduction potential for tetrathionate to thiosulfate is 0.08 V.

$$2S_2O_3^{2-} + O_2 + 2H_2O = S_4O_6^{2-} + 4OH^- \qquad (62)$$

Reduction of tetrathionate to thiosulfate and sulfate generates acid and controls the loss of thiosulfate to oxidation which is unfortunately catalyzed by copper.

$$SO_3^{2-} + 2OH^- + S_4O_6^{2-} = 2S_2O_3^{2-} + SO_4^{2-} + H_2O \qquad (63)$$
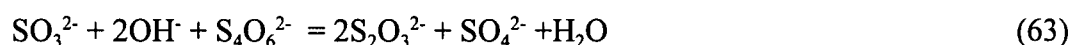
Test conditions suggested by the patents and other research (Hemmati et al.) appear to be somewhat ore specific with regards to thiosulfate consumption, but in general the kinetics are faster than cyanide, the temperature should not exceed 65°C and copper should be present in the few grams per litre range for pulp densities ranging from 10-50%. The ammonium thiosulfate concentrations range

from 10 to 400 grams per litre. Equilibrium conditions that favour the gold thiosulfate complex require a pH less than 8.8 for solutions that are 1M for thiosulfate and ammonia (Zipperian et al.).

A scoping test was conducted with 60 grams per litre ammonium thiosulfate, 1 gram per litre copper sulfate, 15% solids for 8 hours at 50°C with sulfur dioxide at or near saturation and using bottle strength ammonia (28% NH_3) to maintain a pH in the vicinity of 9 for the highest organic carbon containing ore in this study (2841). The result was a significant reduction of gold extraction from 79.2% for CIL extraction to less than 35%. Subsequent tests used pre-oxidized ore feeds (low temperature Redox or bioleach) to compare the sulfur dioxide saturated/ammonia pH control system for thiosulfate leaching to the more conventional cyanidations.

Table 21. Thiosulfate Lixiviation

Feed type	Preoxid^n Method	NH_4TS kg/t	$CuSO_4$ kg/t	NH_4OH kg/t	Solids %	Control	Time hr	Final pH	TS Ext %	CN Ext %
2841	none	350	20	247	14.1	SO_2	8	7.8-9	32.3	79.2
2841	Redox	350	20	143	14.3	SO_2	8	7.6-10.4	79.0	72.6
2844	Bioleach	700	40	848	7.4	$Na_2S_2O_5$	3	9.1	87.7	85.0
2844	Redox	350	20	903	12.9	SO_2	8	6.4-9.1	77.5	72.4
2844	Redox	350	20	475	13.6	SO_2	6	8.81	85.6	79.4
SP340	Redox	350	20	903	12.9	SO_2	6	7-8.3	97.4	97.4

Some slight gains in extraction were seen for all of the oxidized residues when thiosulfate was used as a lixiviant with the exception of the sulfidic residue for which there was not much room for improvement. It is likely that the difference in the extractions between the cyanide and thiosulfate tests can be ascribed to residual carbon activity.

5.3.2   Halides

One of the disadvantages of acid oxidations as a pretreatment for refractory gold ores is the necessity of neutralization or a solid /liquid separation before an alkaline cyanidation for gold recovery. The halides form stable complexes with gold at low pH and may be suitable for treating oxidized pulps directly.

Bromine as a gold lixiviant has been tested using liquid bromine as well as 1,3-dibromo-5,5-dimethylhydantoin which generates hypobromous acid in solution in combination with bromide salts (Equation (44)). For iodine it is the triiodide which can act as oxidant:

$$2Au + I_3^- + I^- = 2AuI_2^- \qquad (64)$$

Mixed halide systems have the economic advantage of replacing the more expensive iodide or bromide salts with chloride. A commercial mixed halide treatment is known as the INTEC Copper Process (Everett).

Tests are generally shorter for gold extraction with halides due to enhanced kinetics. Two kinetic series are plotted in Figure 9 along with their cyanidation extractions before pretreatment.
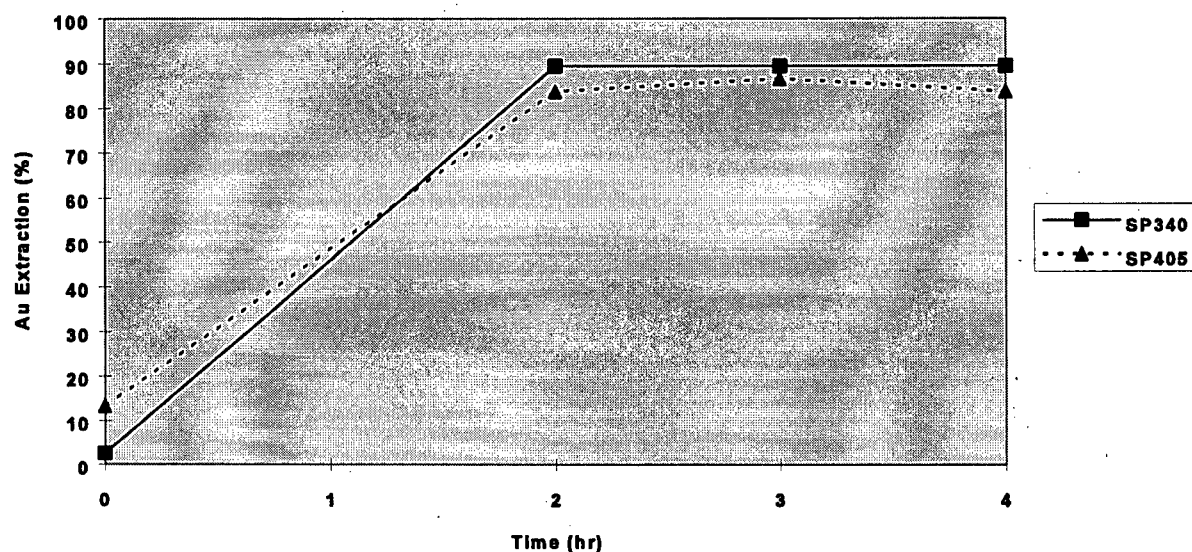
Figure 9.   Gold extraction at 12 kg/t Br$_2$ and 6 kg/t KBr.

The results of these tests suggest that a leach time of 2 hours or less would be sufficient.

Table 22. Mixed Halide Gold Extraction

Feed type	Test #	Br$_2$ kg/t	KBr kg/t	H$_2$SO$_4$ kg/t	KI kg/t	NaCl kg/t	Time hr	Temp C	Final pH	Final mV	3hr Ext %
SP405	BR14A	50	20	62.8			3	40	1.3	579	94.0
SP405	BR14B	50	20	61.4		200	3	40	1.2	894	94.8
SP405	BR14C	50	40	82.6		200	3	40	0.9	937	96.5
SP405	BR14D	50	20	62.0	40		3	40	1.1	857	99.1
SP405	BR14E	50	20	71.2			3	40	1.1	880	95.7

At least one study suggests that oxidizing potential is lost in a mixed system of iodide and bromine

by the formation of Br$_2$I$^-$ (Pesic & Sergent) but test results using a Redox process oxidized residue

demonstrated that this combination provided the best extraction (Table 22 test BR14D). The last test of the series incorporated active carbon on the recommendation of one study (Pesic et al.) and the results were not significantly different than the test without carbon (95.7 vs 94.0% gold extraction).

Direct bromination of Redox leached pulps for SP340 ore gave virtually identical results to the kinetic leach test for this sulfidic ore. The oxidized pulp was diluted to 15% solids and cooled to 50°C after 1 hour of low temperature Redox leaching. Both one and two hour bromine leach samples resulted in gold extractions similar to those seen for a filtered and washed Redox residue.

Table 23. Bromine/Bromide Gold Extraction

Feed Type	Test #	Br_2 kg/t	KBr kg/t	H_2SO_4 kg/t	Time hr	Temp C	Gold Extraction %			
							1hr	2hr	3hr	4hr
SP340	BR12	12	6	3.7	4	40		89.5	89.5	89.5
SP340	BR15	10	5	38	3.5	50	90.0	90.0		

The immediate benefit of these results is that there is the possibility of eliminating one filtration step where acidic oxidation of sulfide is indicated as the refractory ore pretreatment of choice.

6.0   CONCLUSIONS

1.   The chemistry of refractory gold ores is made complex by their heterogeneity which is promulgated in two major refractory characteristics; occlusion and adsorption.

2.   Occlusion can be physical or kinetic, and these in turn can be natural or process induced while adsorption is generally reversible /kinetic and is also natural or process induced.

3.   Natural occlusion is a function of the geochemical genesis of the ore whereas process induced occlusion can be caused by dense oxide phase development in roasting, surface passivating film formation from incomplete oxidation of sulfide or precipitant formation in leaching, and/or passivation by adsorbed non-auriferous complexes in lixiviation.

4.   Natural adsorption or preg-robbing is related predominantly to the complexing of gold by the graphitic or organic components of the ore before the ore is treated whereas process induced adsorption occurs when ore components become active due to increases in surface area or components are oxidized to an adsorption active state with respect to the lixiviant in use, leading to retention of the normally soluble gold complex.

5.   The degrees of refractoriness in gold ore can reasonably be distinguished as either economical or uneconomical to exploit.

6. The heretofore static analyses and characterization tests for ores can be augmented with kinetic evaluations using standard conditions that can often apportion refractory behaviour into its individual causes:

a) Bioleaching can determine the quantity of gold occluded by arsenopyrite and leachable sulfides that may be available for extraction by partial oxidation.

b) Nitric acid (Redox) oxidation at temperatures higher than 185°C can determine the amount of gold that remains occluded by acid insoluble silicates in sulfidic ores.

c) Nitric acid (Redox) oxidation at 85°C liberates sulfide occluded gold and can activate the non-hydrometallurgically oxidizable carbon in doubly refractory ores.

d) Inert atmosphere roasting below 650°C liberates gold that is occluded by a sulfide matrix as well as that adsorbed by humic substances but can create active graphitic adsorption sites.

e) Thiosulfate leaching can potentially determine the propensity of an ore to re-adsorb gold complexes with cyanide, thiourea or the halides due to the low uptake of thiosulfate gold complexes by active carbon.

f) Bromine or mixed halide lixiviation provides a quicker test for gold solubility than cyanidation due to enhanced kinetics and can be used as a rapid test for degree of gold liberation for partial oxidation pretreatments.

7. The plant metallurgist has available many diagnostic procedures to distinguish the mechanisms for the resistance of an ore to leaching and can use these to circumvent costly

empirical studies combined with sophisticated analytical techniques by the use of simple

amenability tests.

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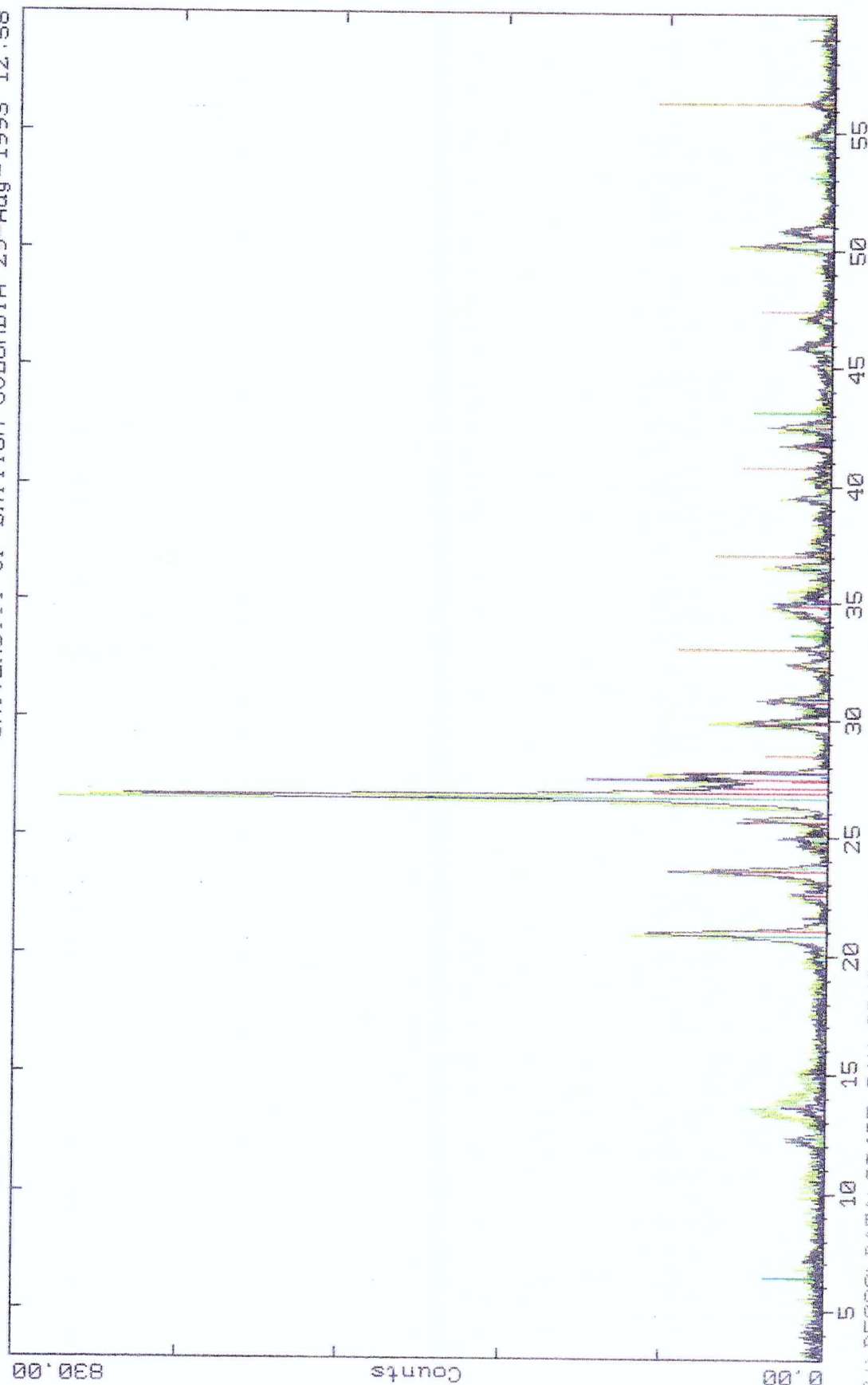
APPENDIX

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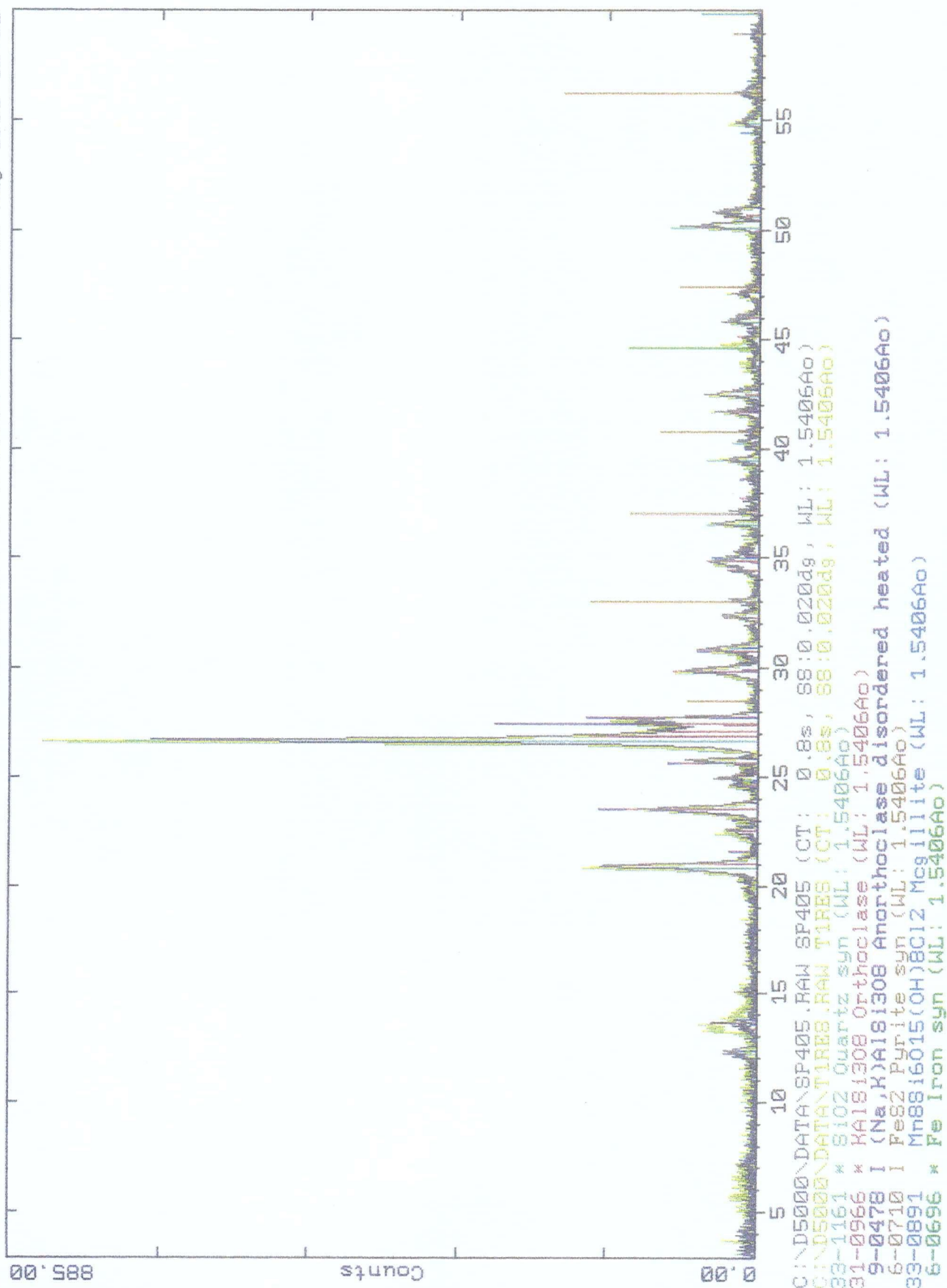
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6-0710 I FeS2 Pyrite syn (WL: 1.5406Ao)
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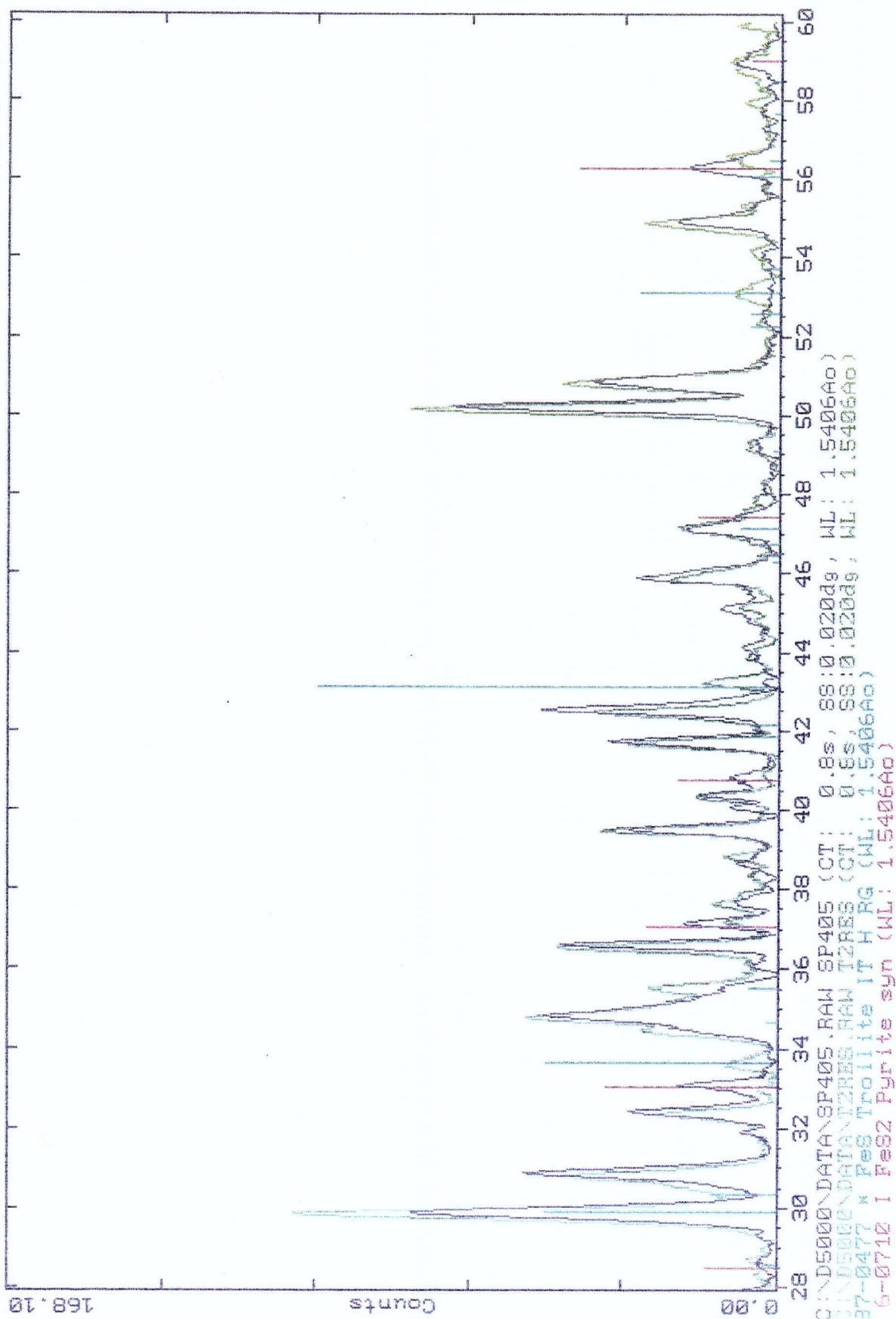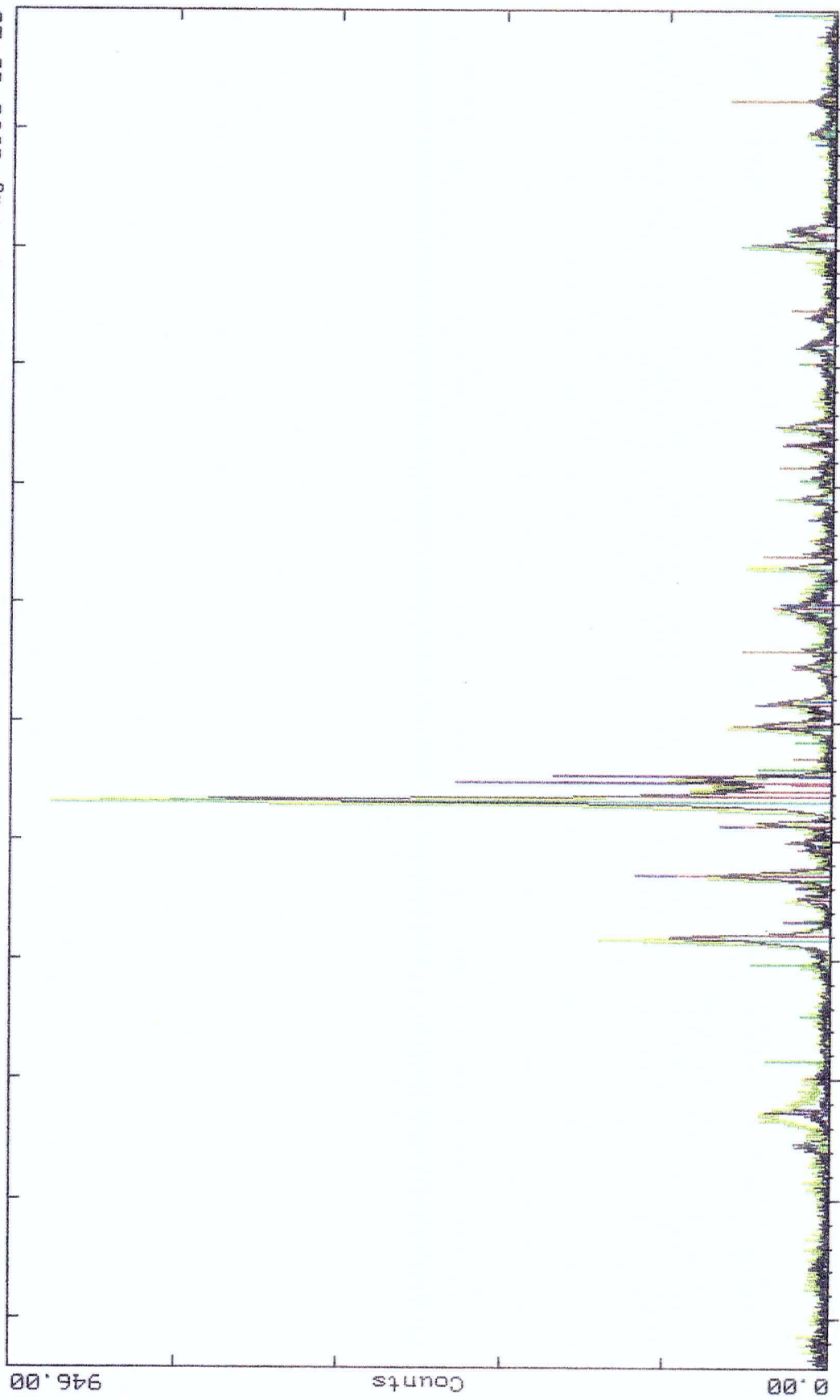UNIVERSITY OF BRITISH COLUMBIA 18-Oct-1993 12:55

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