

Applications of NSC pressure leaching

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ABSTRACT

Industrially proven and applied nitrogen species catalyzed (i.e. NSC) acidic pressure leaching has been utilized to treat a number of ores, concentrates and recycled materials. This versatile technology offers many advantages including low temperatures, low pressures, low capital costs, low operating costs, faster throughput and less costly materials of construction than conventional pressure oxidation. This paper will elucidate the application of this technology for production of gold, silver, palladium, rhodium, zinc, germanium, gallium, cobalt, copper and nickel. Recycled material applications will also be elucidated. In addition, details of an application for treatment of a refractory chalcopyrite copper and gold bearing ore will be presented and pertinent economics discussed.

INTRODUCTION

Hydrometallurgical treatment of concentrates, ores and materials is a growing global technology. Yet, there is still trepidation in the use of nitric acid in hydrometallurgy. Ironically, Habashi (1) reports that nitric acid has been used for over 50 years in the uranium industry and in phosphate fertilizer manufacture and his annotated comments follow. Its application in the treatment of sulphide concentrates is relatively recent. A review of recent developments is given. Among the well known applications are:

- Dissolution of ammonium or sodium uranate (yellow cake) for purification by organic solvents to obtain high purity uranyl nitrate hexahydrate from which pure UO_2 is obtained;
- Dissolution of spent uranium fuel element for reprocessing and recovery of plutonium;
- As a stripping agent in some ion exchange and solvent extraction processes;
- As a pickling agent for stainless steel;
- Leaching of phosphate rock to produce nitrophosphate fertilizers; and
- Uranium concentrate leaching.

Also, nitric acid has been proposed for leaching of aluminum from clay since aluminum nitrate can be decomposed at a low temperature.

It is further stated that the use of nitric acid in extracting metal values from sulphide ores was proposed as early as 1909 by Kingsley (2). Rankin (3), and later Westby, Joseph and Weber (4-7) used concentrated acid at 100°C . Kingsley in a later patent attempted to recover sulphur in the elemental form by using more mild conditions, namely 5% HNO_3 and 80°C . Pauling (8) found that when mild leaching conditions were used, the yield of sulphur was high (about 80%) for FeS , PbS , and ZnS , was negligible (about 10%) for pyrite and marcasite, and was intermediate (about 50%) for chalcopyrite. Bardt (9) and later Bjorling et al. (10-16) used air oxidation in H_2SO_4 , to which a small amount of HNO_3 was added, to solubilize chalcopyrite with the simultaneous recovery of elemental sulphur. The nitric acid played the role of a catalyst since during leaching, nitric oxide gas is liberated which, in the presence of oxygen and water, is converted back to HNO_3 . Researchers at Kennecott Copper in Utah (17) built a pilot plant for treating chalcopyrite concentrate with HNO_3 - H_2SO_4 mixture. Similar work but on a small scale was also conducted independently at Anaconda (18) and other laboratories (19-22). Habashi (23) studied the nitric acid treatment of a low-grade nickel-copper sulphide concentrate composed of pyrrhotite, pentlandite, and chalcopyrite. It was found that with 15-30% HNO_3 , all the nickel and copper went into solution, while elemental sulphur and iron oxide remained in the residue after 8 hours. Similar work was also conducted elsewhere by Ouellet and Shukla (24,25). The reaction of nitric acid with molybdenite concentrates has also been extensively studied under autoclave conditions

(26-33). In this case molybdic and sulphuric acids are formed. The Nitrox, Arseno, and Redox processes were developed to a pilot plant scale to treat pyrite and arsenopyrite containing refractory gold that was not amenable to direct cyanidation (34-40). After decomposing the minerals with HNO_3 , gold was liberated and became accessible to cyanidation. Industrial research has shown that it was possible to treat matte produced from copper-nickel sulphide concentrates which are rich in platinum metals with nitric acid to solubilize copper and nickel for recovery. The residue obtained can be rich in platinum metals if the conditions are correct (41-44).

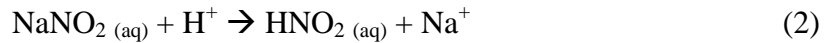
As seen in the above annotated discourse, the use of nitric acid in sulphide oxidation is not new. However, only the nitrogen species catalyzed (i.e. NSC) sulphuric acid oxidative pressure leach has ever been built and operated successfully on a long term industrial scale (45). Moreover, it has been thoroughly studied and it has found successful application in oxidation of other feedstocks as well (46-56). It offers several definitive advantages. First of all it is the only proven industrial process over the long term for pressure leaching of copper sulphides and direct recovery of precious metals without the use of cyanide. Second, the rate of reaction is much faster and subsequent required reactor volume is thus smaller. Third, the process does not require excessively high temperatures or pressures. Fourth, the oxidation reduction solution potential (i.e. ORP) can be adjusted and controlled to be extremely high so it oxidizes almost any sulphide at low oxygen overpressures. Fifth, the materials of construction are readily available stainless steels so there is no need for titanium cladding or brick with lead liners. Thus, the capital and maintenance costs are less. Also, because of the simpler internal design, direct heat exchange can be utilized in-situ for optimal temperature control. Further, in a manner analogous to existing Ni/Co laterite HPAL systems, the energy from the in-situ heat exchanger can be readily utilized for optimizing the plant heat balance or co-generating electrical power resulting in significant process operating cost savings. Sixth, there is no need for a dip tube or special design radial agitators with cowlings and the like. Oxygen transfer is innate with the enhanced nitrogen species chemistry. So, without titanium, or a titanium dip tube in particular, there is much less oxygen fire danger. Seventh, the design of the feed pump system is far less of a challenge as is the flash system and the choke system. Eighth, like a smelter, precious metals recovery can be high and direct. Ninth, there is no sophisticated chloride chemistry or resultant corrosion issues to deal with. Tenth, by-product elemental sulphur formation, handling and treatment for contained gold recovery is proven and readily accomplished and value added by-products such as sodium sulphate, sodium hydroxide sulfuric acid and gypsum can be produced from the waste streams. Finally, there is minor amount of nitrogen species utilized which reports almost entirely to the gas phase upon autoclave flashing. This is readily destroyed and scrubbed using commercially available equipment. So, there are no major economic or environmental issues with the use of the small amount of nitrogen species utilized.

NSC PRESSURE LEACHING FUNDAMENTALS

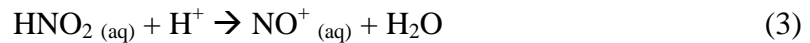
The commonly reported leach reaction of a sulphide mineral with nitric acid in conjunction with sulphuric acid is shown below.



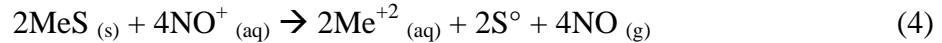
However, it is postulated that the actual reaction species is NO^+ and not NO_3^- (47,50). The addition of or presence of NO_2^- instead of NO_3^- accelerates the formation of NO^+ . As shown in Table I, the NO^+/NO couple is capable of an extremely high redox potential (57). So, NO^+ is readily formed from nitrous rather than nitric acid. For example, a convenient source of nitrous acid can be sodium nitrite (47,50). When it is added to an acidic solution, nitrous acid is readily formed.



Nitrous acid further reacts to form NO^+ .



The NO^+ then reacts with the mineral and oxidizes the sulphide to sulphur.



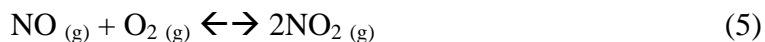
Of course, at higher temperatures and/or nitrogen species concentrations the sulphide can be fully oxidized to sulphate.

Table I - Relative Potentials of Hydrometallurgical Oxidizers

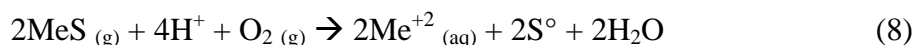
Oxidant	Redox Equation	E°_h (pH = 0, H_2 ref.)
Fe^{+3}	$\text{Fe}^{+3} + \text{e}^- \rightarrow \text{Fe}^{+2}$	0.770 V
HNO_3	$\text{NO}_3^- + 4\text{H}^+ + 3\text{e}^- \rightarrow \text{NO} + 2\text{H}_2\text{O}$	0.957 V
HNO_2	$\text{NO}_2^- + 2\text{H}^+ + \text{e}^- \rightarrow \text{NO} + \text{H}_2\text{O}$	1.202 V
$\text{O}_2_{(g)}$	$\text{O}_2 + 4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2\text{O}$	1.230 V
$\text{Cl}_2_{(g)}$	$\text{Cl}_2_{(g)} + 2\text{e}^- \rightarrow 2\text{Cl}^-$	1.358 V
NO^+	$\text{NO}^+ + \text{e}^- \rightarrow \text{NO}$	1.450 V

As can be seen, nitric oxide gas, NO , is produced from the oxidation of sulphides. As this gas has a limited solubility in aqueous solutions, it tends to transfer out of solution. In the pressure leach system, a closed vessel with an oxygen overpressure is used. The nitric oxide gas emanating from the leach slurry accumulates in the headspace

of the reactor where it reacts with the supplied oxygen to form nitrogen dioxide gas. The NO is then regenerated to NO⁺. Overall this can be viewed as:



Since the nitrogen species is continuously regenerated, its role in the overall reaction as the actual oxidizer is not obvious. The net overall reaction has the sulphide mineral reacting with the acid solution and oxygen to solubilize the metal value into the sulphate solution and form some elemental sulphur. Of course, at higher temperatures and/or nitrous acid concentrations the sulphide would be fully oxidized to sulphate.



Overall, the nitrogen intermediates serve as an expedient means to transport oxygen to the surface of the solid particle and allow the resulting reaction to take place at a heightened redox potential. This inherent asset of the unique system precludes the use of high temperatures and high pressures, which lead to higher costs in other pressure leach processes. For example, commonly available stainless steel can be used for the reactor vessel. And, complete oxidation of sulphide to sulphate can be achieved without the excessive conditions found in other pressure leach systems. Thus, the rapid kinetics of the system leads to smaller reactor volumes and higher unit throughputs. Finally, 99.9% of the nitrogen species utilized in the leach system report to the gas phase when the pressure vessel is flashed and they are readily destroyed and contained by commercially available scrubber systems. So, environmental impacts are minimized and the NSC leach plant solutions contain little or no nitrogen species.

NSC APPLICATIONS

Industrial Partial Nitrogen Species Catalyzed Pressure Oxidation of a Silver Bearing Copper Concentrate

In this original industrial application which operated from 1984 until 1995 (45), the purpose of the NSC plant leach process was to solubilize the silver and copper values from a copper concentrate so they are selectively recovered downstream. The plant flowsheet is shown in Figure 1. The typical plant feed is shown in Table II; operating conditions are provided in Table III.

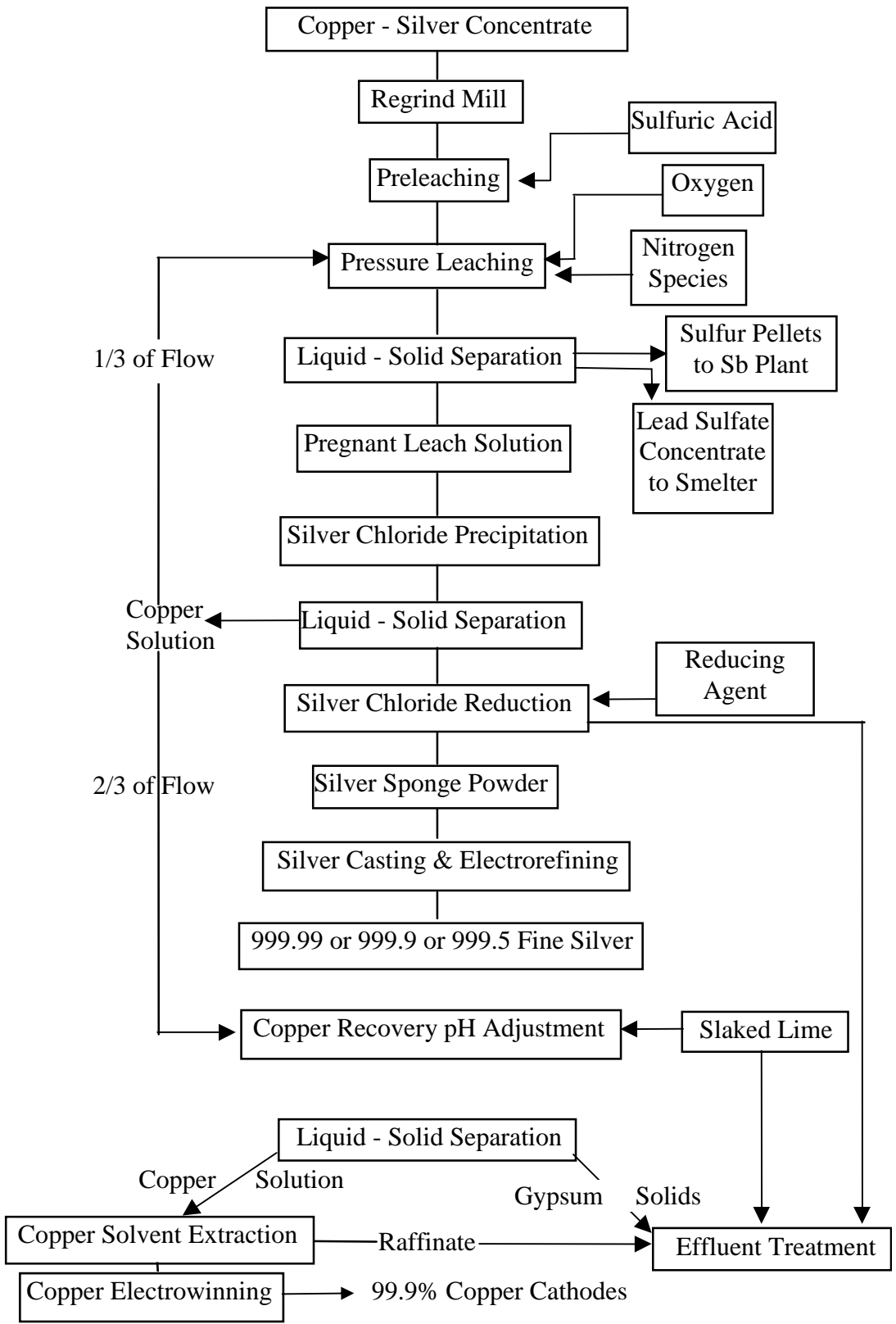


Figure 1 - NSC Leach Industrial Plant Flowsheet

Table II - Typical Analysis of NSC Plant Copper Concentrate Feed

Element	Content, %
Copper	24
Silver	4
Iron	18
Antimony	1
Zinc	2
Arsenic	2
Lead	2-20
Sulphide	35

Table III - Nitrogen Species Catalyzed Leach Plant Conditions

Operating Criteria	Unit
Batch size	17,000 liters
Initial Cupric Ion Concentration	15 g/L
Initial Free Sulphuric Acid Concentration	200 g/L
Preleach temperature	50°C
Preleach Time	1 Hour
Slurry Solids Content	8 - 20% by weight
Reactor Working Pressure	620 kPag maximum (90 psig)
Reactor Working Temperature Range	50° to 155°C
Nitrogen Species Concentration	2.0 g/L
Particle size	80% -10 micron
Reaction Time	1 Hour

Three major products were produced in the NSC plant pressure leach. These were:

1. The NSC leach solution which contained the silver and copper values along with other soluble metals most notably iron.
2. The sulphur pellets which contained most of the oxidized portion of the sulphide mineral.
3. The "fine" leach residue which was the oxidized and finely divided insoluble metallic portion of the feed. In this case both the lead and antimony are insoluble in this leach system and report as lead sulphate and antimony oxide.

Typical plant results are illustrated in Table IV.

The clear emerald green NSC leach solution went on to selective recovery of the silver and copper values. The first step was to recover the silver from the leach solution as the insoluble precipitate of silver chloride. The silver chloride was separated and washed by filtration and was reduced to silver metal. The resulting silver powder (sponge) was cast into a suitable shape for anodes and then placed in an electrolytic refining circuit. The pure silver crystals produced by the refining cells were cast into bars and prepared for market. The details of silver production are beyond the scope of this article but have been published elsewhere (54).

Table IV - Assay and Mass Distribution of NSC Plant Leach Products

Assay	Ag	Cu	Fe	Pb	Sb	As	Zn
Leach Solution	6 g/L	45 g/L	25 g/L	1 mg/L	1 mg/L	2 g/L	2 g/L
Leach Residue	2.7%	1%	3%	35%	4%	1%	0.1%
Sulphur Pellets	nil	0.5%	1%	0.1%	0.1%	0.1%	0.1%
Mass Distribution							
Leach Solution	96%	99%	93%	nil	nil	85%	99%
Leach Residue	4%	0.5%	7%	100%	100%	15%	1%
Sulphur Pellets	nil	0.5%	1%	nil	nil	nil	nil

Meanwhile the silver free copper solution containing the copper along with the other soluble metals went on to the copper section. This was the largest part of the plant and consisted of neutralization of the excess acid needed to leach the silver, followed by solvent extraction and copper electrowinning. As such this operation was the first and longest operating copper concentrate pressure leaching plant in the world (56).

Complete Nitrogen Species Catalyzed Pressure Oxidation of a Chalcopyrite Concentrate

The application of nitrogen species catalyzed oxidative pressure leaching was tested on chalcopyrite concentrate on a lab scale (53,55). In this case, the complete oxidation of sulphide to sulphate was the goal. This facilitated the production of excess acid and ferric iron in-situ, and which could be used to enhance heap leaching of associated copper oxide deposits. Also it provided a means for conventional cyanide recovery of any gold or silver left in the leached solids. Silver could also be recovered via a novel methodology delineated later in the paper. The test conditions and results are illustrated in Tables V, VI, VII and VIII.

Table V - Composition of Chalcopyrite Concentrate Tested

Cu, %	Fe, %	Total S, %
25.1	30.8	36.1

Table VI - Nitrogen Species Catalyzed Complete Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	20 g/L
Reactor Working Pressure	975 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	175°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	30 min

Table VII - Mass Distribution of Nitrogen Species Catalyzed Complete Oxidation Leach Products

	Cu, %	Fe, %
Solution	9.0	98.2
Residue	1.0	1.8

Table VIII - Mass Distribution of Nitrogen Species Catalyzed Complete Oxidation Sulphur Species

	S ⁻² , %	S ⁰ , %	SO ₄ ⁻² , %
Before Complete Oxidation	97.3	0.0	2.7
After Complete Oxidation	0.4	0.0	99.6

As seen, this industrially proven low temperature and low-pressure system was effective in leaching the copper and oxidizing the sulphide to sulphate in a minimum amount of time.

Complete Nitrogen Species Catalyzed Pressure Oxidation of a Chalcopyrite Concentrate With In-situ Iron Precipitation

The application of nitrogen species catalyzed oxidative pressure leaching was tested on chalcopyrite concentrate on a lab scale. In this case, the complete oxidation of sulphide to sulphate was the goal along with in-situ precipitation of most of the iron out of the final leach solution. This can be an added advantage for processing in downstream SX/EW systems. The conditions and results are illustrated in Tables IX, X, XI and XII.

Table IX - Composition of Chalcopyrite Concentrate Tested

Cu, %	Fe, %	Total S, %
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25.1	30.8	36.1
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Table X - Nitrogen Species Catalyzed Complete Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	10.0 g/L
Reactor Working Pressure	975 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	180°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	30 min

Table XI - Mass Distribution of Nitrogen Species Catalyzed Complete Oxidation Leach Products

	Cu, %	Fe, %
Solution	99.0	7.3
Residue	1.0	92.7

Table XII - Mass Distribution of Nitrogen Species Catalyzed Complete Oxidation Sulphur Species

	S ⁻² , %	S ⁰ , %	SO ₄ ⁻² , %
Before Complete Oxidation	97.3	0.0	2.7
After Complete Oxidation	0.9	0.0	99.1

As can be seen, this industrially proven low temperature and low pressure system was effective in leaching the copper and oxidizing the sulphide to sulphate in a minimum amount of time while keeping the iron out of solution through in-situ precipitation. This can also be accomplished using partial oxidation of the sulphide.

Partial Nitrogen Species Catalyzed Pressure Oxidation of a Chalcopyrite Concentrate

The application of nitrogen species catalyzed oxidative pressure leaching was tested on chalcopyrite concentrate on a lab scale. In this case, the partial oxidation of sulphide to sulphur was the goal. There were advantages to this operating scenario besides the lower temperatures and pressures. By-product sulphur could be used for sulphidation of associated copper oxide deposits allowing them to undergo conventional flotation, providing further concentrate feedstocks to the process. The test conditions and results are illustrated in Tables XIII, XIV, XV and XVI.

Table XIII - Composition of Chalcopyrite Concentrate Tested

Cu, %	Fe, %	Total S, %
25.1	30.8	36.1

Table XIV - Nitrogen Species Catalyzed Partial Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	50 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	125°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	20 min

Table XV - Mass Distribution of Nitrogen Species Catalyzed Partial Oxidation Leach Products

	Cu, %	Fe, %
Solution	99.5	9.6
Residue	0.5	0.4

Table XVI - Mass Distribution of Nitrogen Species Catalyzed Partial Oxidation Sulphur Species

	S ⁻² , %	S ⁰ , %	SO ₄ ⁻² , %
Before Partial Oxidation	97.3	0.0	2.7
After Partial Oxidation	0.2	84.1	15.7

As can be seen, this industrially proven low temperature and low-pressure system was effective in leaching the copper and oxidizing the sulphide to sulphur in a minimum amount of time.

Partial Nitrogen Species Catalyzed Pressure Oxidation of a Precious Metals Bearing Chalcopyrite Concentrate

The application of nitrogen species catalyzed oxidative pressure leaching was tested on chalcopyrite concentrate bearing precious metals on a lab scale. In this case, the partial oxidation of sulphide to sulphur was the goal. There were several advantages to this operating scenario besides the lower temperatures and pressures. First, any silver would be recovered directly by established industrial practice without the use of

cyanide (21). Also, the by-product sulphur could be used for sulphidation of associated copper oxide deposits allowing them to undergo conventional flotation, providing further concentrate feedstocks to the process. And, since the gold in the chalcopyrite largely reported to the sulphur, it also provided a means for a novel method of non-cyanide gold recovery. These ancillary processes will be delineated in the next section of the paper. The test conditions and results are illustrated in Tables XVII, XVIII, XIV and XX.

Table XVII - Composition of Precious Metals Bearing Chalcopyrite Concentrate Tested

Cu, %	Fe, %	Au, g/t	Ag, g/t	Total S, %
20.1	33.5	16.3	320.2	37.4

Table XVIII Nitrogen Species Catalyzed Partial Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	175 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	125°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	25 min

Table XIX - Mass Distribution of Nitrogen Species Catalyzed Partial Oxidation Leach Products

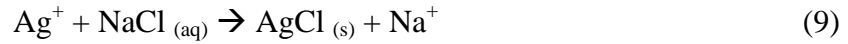
	Ag, %	Cu, %	Fe, %	Au, %
Solution	97.0	99.5	97.0	0.0
Residue	3.0	0.3	2.0	3.9
S° Pellets	0.0	0.2	1.0	96.1

Table XX - Mass Distribution of Sulphur Species with Nitrogen Species Catalyzed Partial Oxidation

	S ⁻² , %	S°, %	SO ₄ ⁻² , %
Before Partial Oxidation	98.4	0.0	1.6
After Partial Oxidation	0.2	83.7	17.1

Nitrogen Species Catalyzed Pressure Oxidation Non-cyanide Gold and Silver Recovery Processes

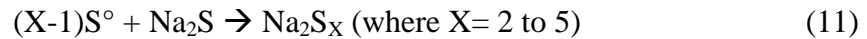
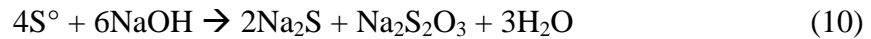
A key element of nitrogen species catalyzed oxidation is the non-cyanide recovery methods available for gold and silver, the use of by-product sulphur and the treatment of any associated copper oxide deposits. First, to directly recover silver, the filtered leach solution from either the partial or complete oxidation process can be stripped efficiently and selectively of its silver using NaCl solution to precipitate AgCl. This is illustrated as:



The solid AgCl precipitate is filtered from the pregnant solution and washed thoroughly. Then, the silver chloride is reduced to elemental silver by established methods. Finally, electrorefining produces silver to a purity of 99.95%, 99.99%, or 99.999% (54).

Next in the partial oxidation process, gold reported primarily to the sulphur product. This is not a novel process phenomena as sulphur collection of gold in pressure leaching has been reported elsewhere (58-60). This prompted use of a non-cyanide method for gold recovery using the sulphur produced. Dissolution of elemental sulphur in sodium hydroxide is used industrially as a lixiviant for alkaline sulphide leaching of antimony (61). To better illustrate this, Figure 2 (62) shows the equilibrium sulphur and gold system while Figure 3 (63) shows the metastable species sulphur system. In reality, the species shown in Figure 3 dominate as the alkaline sulphide system is slow to ever reach equilibrium.

The combination of sodium hydroxide and elemental sulphur results in the formation of species other than just sulphide (S^{2-}). Both sodium polysulphide (Na_2S_x) and sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) are created along with sulphide. This is illustrated simplistically in the following scenario.



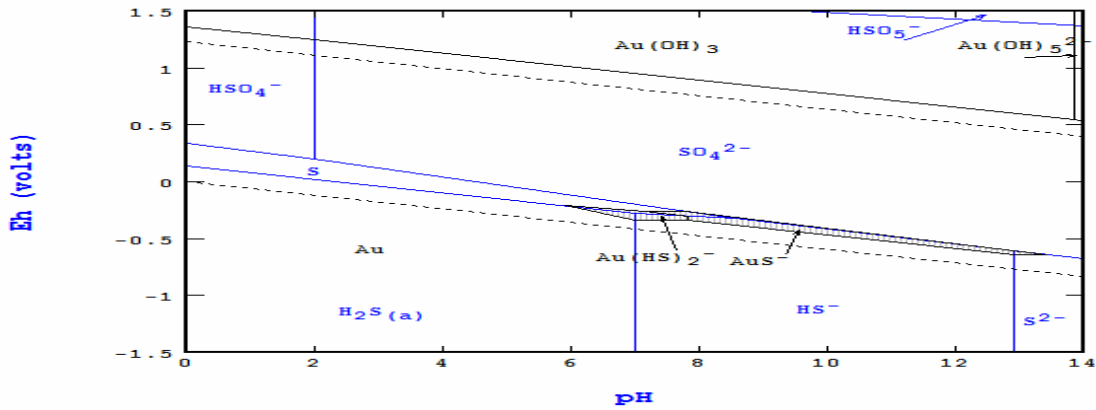


Figure 2 - Equilibrium Species Eh-pH Diagram for Sulphur and Alkaline Sulphide Gold

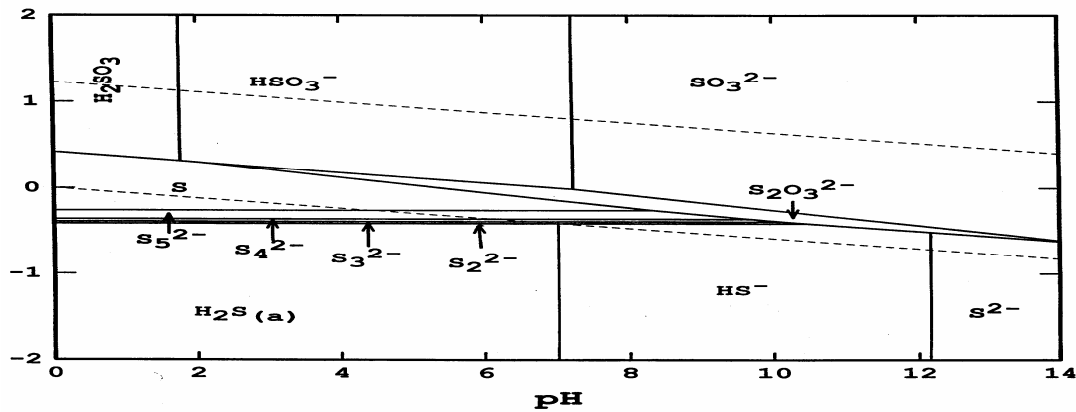
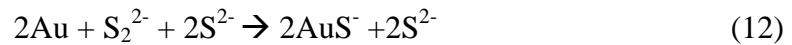


Figure 3 - Meta-Stable Species Eh-pH Diagram for Sulphur

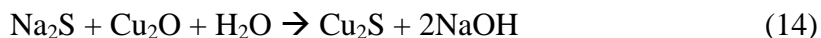
Currently, studies are underway on the actual kinetics and mechanism of the alkaline sulphide system (64,65,66). A rotating electrochemical quartz crystal microbalance (REQCM) is being utilized to study the system. The initial work suggests that complexation of gold may be by sulphide after oxidation by polysulphide. So, gold lixiviation is the result of leaching by polysulphides and sulphides.



Gold leached by the alkaline sulphide system is readily recoverable by several means including electrowinning, gaseous precipitation, chemical precipitation, cementation, solvent extraction and ion exchange.

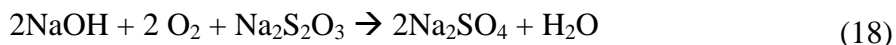
Alkaline sulphide solutions made from by-product sulphur from the partial oxidation process can be used in sulphidation of associated copper oxide ores. This has

been practiced industrially in the African copper belt as well as other global venues (67,68). This is illustrated simplistically by the following scenario.



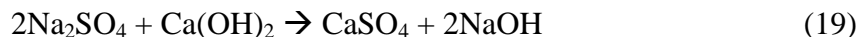
The resultant copper sulphides can then be floated to make concentrate feedstocks for the partial nitrogen species catalyzed oxidation process.

Also the waste alkaline sulphide solutions can be recycled for further gold leaching or further processed with low temperature oxidation to sodium sulphate, Na_2SO_4 . This has been routinely practiced in industry (69). The resultant sodium sulphate is further treated by purification and crystallization to produce high grade, marketable sodium sulphate. This process is illustrated simplistically in the following scenario.

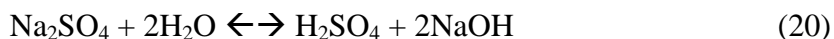


This versatile and environmentally benign chemical is then be sold to and utilized in industries such as pulp and paper, glass, ceramics, detergents, textile dyes, mineral feed supplements, bleach and photography. As such, there is no environmental or toxicological issue in the use of alkaline sulphide gold recovery as the waste products become value added, marketable by-products.

As well the sodium sulphate produced can be used to regenerate the sodium hydroxide needed in the process in a manner analogous to industrial dual alkali scrubbing systems (70). This is as follows:



The clean gypsum product can then be marketed and used in such applications as agricultural soil amendments or as an additive in primary cement manufacture. In addition, initial efforts are underway and have been successful in regenerating the necessary H_2SO_4 and $NaOH$ reagents from the Na_2SO_4 by-product. The details of this process will be given in future publications and may be illustrated as follows;



Partial Nitrogen Species Catalyzed Pressure Oxidation of a Refractory Chalcopyrite Gold Ore

NSC pressure leaching can be utilized for treatment of gold bearing concentrates and ores. It has particular utility when used in conjunction with alkaline sulphide gold recovery from carbonaceous preg-robbing and sulphide ores and concentrates and especially those containing copper and other cyanicides. As previously delineated in the introduction, several other candidates for refractory gold ore and concentrate pretreatment utilizing nitric acid have been applied. These are outlined in Tables XXI and XXII.

Table XXI - Nitrogen Species Pressure Leaching Gold Application
Historical Record

Process Name	Industrial Application	Operation	Other Applications
NSC	Ag, Cu	11 years	PGM's, Au, Ni, Co, Zn
REDOX	Au	Pilot 1 year	None
NITROX	None	None	Au

Table XXII - Comparative Nitrogen Species Catalyzed and Selected Conventional Acid Pressure Leaching Gold Plant Operating Criteria

Name	Process Temp.	Process Pressure	Nitrogen Species Conc.	Nitrogen Species Regen. Process	Residence Time, min	Materials of Const.
NSC	125-170°C	625-975 kPag	2-3 g/L	In-site	15-45	Stainless steel
REDOX	85-95°C	Atmos.	140-180 g/L	External	60	Titanium
REDOX	195-210°C	1950 kPag	70-110 g/L	External	15	Titanium
NITROX	85-95°C	Atmos.	10-150 g/L	External	60	Stainless steel
McGlaughlin	180°C	2200 kPag	O ₂ Only	O ₂ Only	90	Lead/ acid brick
Sao Bento	190°C	1600 kPag	O ₂ Only	O ₂ Only	120	Lead/ acid brick
Goldstrike	225°C	3000 kPag	O ₂ Only	O ₂ Only	75	Lead/ acid brick
Getchell	210°C	3200 kPag	O ₂ Only	O ₂ Only	90	Lead/ acid brick

In the following example, 500 tonnes per day of a gold bearing chalcopyrite ore has been proposed to be treated by NSC partial sulphide oxidation pressure leaching followed by SX-EW production of copper. The gold will be recovered via alkaline sulphide leaching followed by electrowinning. Table XXIII illustrates the plant conditions utilized while Table XXIV shows the result of NSC partial sulphide oxidation on the chalcopyrite concentrate. Table XXV delineates the site-specific economics of the application for treatment of the chalcopyrite ore. Figure 4 illustrates the proposed flowsheet. A key aspect here is to minimize sulphide oxidation to elemental sulphur. This minimizes oxygen consumption, reduces in-situ acid production and the closed circuit process causes in-situ iron precipitation. This limits the amount of iron and acid build up in the process circuit thereby limiting the amount of the circuit impurity bleed stream. As well, the elemental sulphur produced acts as a non-cyanide lixiviant for gold in the alkaline sulphide recovery system. Another key aspect is that the solutions recycled do not build up nitrates or nitrites, which could be damaging to the solvent extraction reagents. Continuous closed circuit testing and analysis of the flowsheet has consistently shown both nitrogen species solution concentrations of less than 0.1 mg/L. Thus, by in-situ methods, the closed circuit NSC system does not allow any buildup of nitrogen species in the recycled solutions, which may be harmful.

Table XXIII - Nitrogen Species Catalyzed Partial Oxidation Leach Conditions with In-Situ Iron Precipitation

Operating Criteria	Unit
Initial Free Sulphuric Acid	15 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	125°C
Nitrogen Species Concentration	2.0 g/L

Table XXIV - Summary of Nitrogen Species Catalyzed Partial Oxidation Leach

Composition of Chalcopyrite Ore				
	Cu, %	Fe, %	Total S, %	Au, g/t
	3.2	4.1	5.7	11.0
NSC Partial Oxidation Leach Elemental Distribution				
	Cu, %	Fe, %	Total S, %	Au, %
Solution	99.2	8.3	3.4	0.0
Residue	0.8	91.7	96.6	100.0

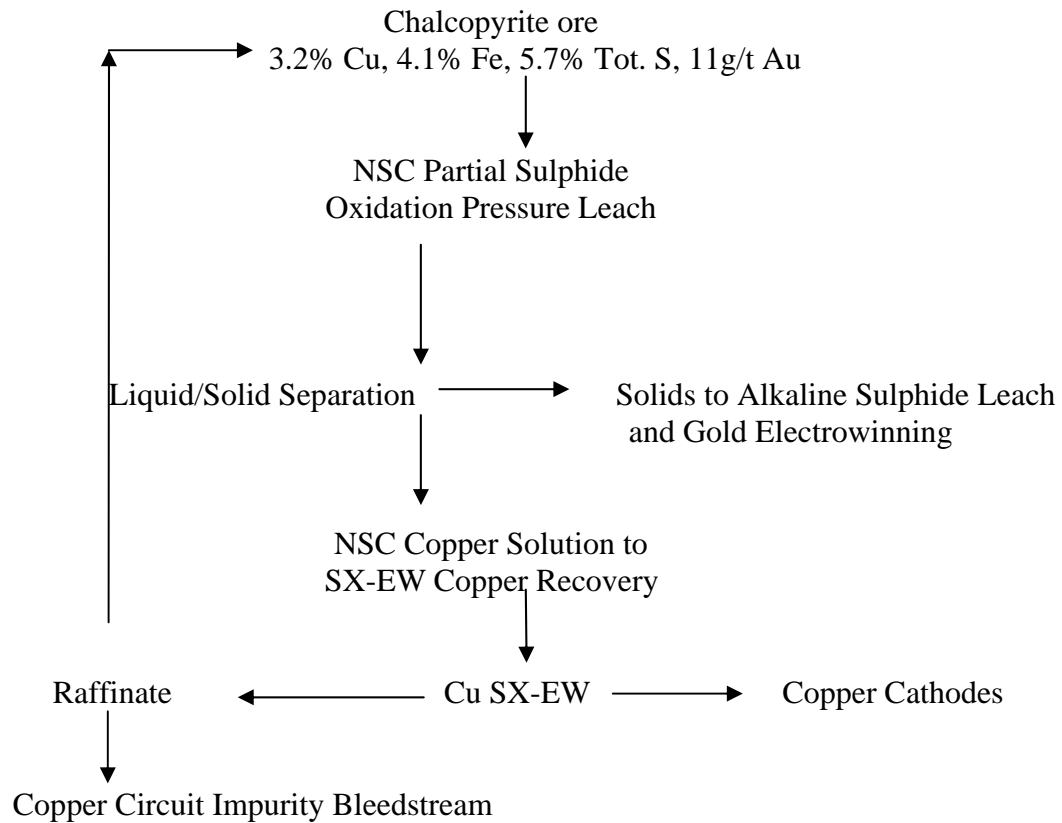


Figure 4 - Flowsheet of NSC Partial Sulphide Oxidation of Gold Bearing Chalcopyrite

In this scenario, both sulphuric acid generation and iron dissolution are minimized along with production of elemental sulphur to be utilized in non-cyanide gold recovery. For gold processing, alkaline sulphide leaching with electrowinning was used and resulted in a gold recovery of 93.1 %.

Table XXV - Summary of Site Specific Economics for NSC Partial Oxidation of a Gold Bearing Chalcopyrite Ore

Economic Criteria	Cost
NSC Chalcopyrite Ore Partial Oxidation CAPEX	\$ 3,500,000.00 USD
NSC Chalcopyrite Partial Oxidation OPEX	\$ 0.056 USD/lb Cu

The relative economics of the NSC pressure oxidation methodologies are better than that of conventional pressure leaching. This is illustrated by an order of magnitude economic analysis of various pressure oxidation pretreatment options subject to:

- The plant is located in the Western United States treating 250 tonnes per day of gold bearing pyrite ore with gold = 35 g/t, Fe = 25.0%, As = 6.31 % and Total Sulphur = 24.0%; and
- The costs include all normal unit operations involved in pressure leaching.

Operating costs are expressed as dollars per % contained sulphide in the concentrate being oxidized. Based on this, the order of magnitude capital and operating costs for NSC compared to conventional pressure leaching are summarized in Tables XXVI and XXVII.

Table XXVI - Comparative Order of Magnitude Capital Cost Estimate

Type of Process	USD
Conventional Complete Sulphide Pressure Oxidation	\$12,500,000
Complete NSC Sulphide Oxidation Process	\$10,000,000
Partial NSC Sulphide Oxidation Process	\$ 7,500,000

Table XXVII - Comparative Order of Magnitude Operating Cost Estimate

Type of Process	USD/% Contained Sulphide Oxidized
Conventional Pressure Oxidation	\$ 2.75
Complete NSC Sulphide Oxidation Process	\$ 2.00
Partial NSC Sulphide Oxidation Process	\$ 1.50

Finally, the relative reagent costs of sodium cyanide versus alkaline sulphide produced from sulphur are illustrated in Table XXVIII. This does not include the by-product revenues available when using the alkaline sulphide system or the costs associated with destruction of waste cyanide. Thus, as seen, there are significant economic incentives with the use of alkaline sulphide leaching of gold instead of cyanide. This advantage was illustrated before in the treatment of gold bearing chalcopyrite where conventional pressure leaching followed by cyanidation is far more expensive.

Table XXVIII - Comparative Cost of Gold Leaching Reagents

Gold Reagent Type	Cost
Sodium cyanide	\$ 1.00 - \$ 2.25 USD/kg
Alkaline sulphide	\$ 0.05 - \$ 0.30 USD/kg

Partial Nitrogen Species Catalyzed Pressure Oxidation of a PGM Concentrate

The application of NSC oxidative pressure leaching is illustrated by testing a PGM smelting concentrate and a chalcopyrite, pentlandite, palladium bearing ore on a lab scale. In this case, the partial oxidation of sulphide to sulphur and the lixiviation of palladium and rhodium and recover them selectively was the goal (52). Alternatively, PGM's can also be retained in the solid phase by controlling operating parameters (44). The test conditions and results are illustrated in Tables XXIX, through XXXIV.

Table XXIX - Composition of PGM Smelting Concentrate Tested

Pt g/t	Pd, g/t	Rh, g/t	Ni, %	Cu, %	Fe, %	Total S, %
2004	5967	538	19.5	12.3	34.2	25.5

Table XXX - Nitrogen Species Catalyzed Partial Sulphide Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	50 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -40 micron
Maximum Temperature	140°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	45 min

Table XXXI - Mass Distribution of Nitrogen Species Catalyzed Partial Sulphide Oxidation Leach of PGM Smelting Concentrate Products

	Pt %	Pd %	Rh, %	Ni %	Cu %	Fe %	Total S, %
Solution	0.0	99.7	99.6	99.5	99.6	93.8	78.3
Residue	100.0	0.3	0.4	0.5	0.4	6.2	11.7

Table XXXII - Composition of Chalcopyrite, Pentlandite Palladium Bearing Ore Tested

Cu, %	Co, %	Ni, %	Fe, %	Pd, g/t	Total S, %
0.85	0.10	0.74	0.91	6.2	2.36

Table XXXIII - Nitrogen Species Catalyzed Partial Sulphide Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	50 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% - 40 micron
Maximum Temperature	140°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	45 min

Table XXXIV - Mass Distribution of Nitrogen Species Catalyzed Partial Sulphide Oxidation Leach of a Chalcopyrite, Pentlandite Palladium Bearing Ore

	Cu, %	Co, %	Ni, %	Pd, %	Fe, %	Total S, %
Solution	97.3	99.4	99.6	>99.0	95.3	1.5
Residue	2.7	0.6	0.4	ND	4.7	98.5

As can be seen in both cases, this industrially proven low temperature and low pressure system was effective in leaching the palladium, rhodium copper cobalt nickel and iron. Platinum is also successfully solubilized in the NSC system with the addition of chloride for complexation (71). The applicability has also been confirmed on testing of other PGM bearing materials such as tailings, pyrometallurgical mattes, secondary recycled materials and concentrates.

Partial Nitrogen Species Catalyzed Pressure Oxidation of a Cobaltite Chalcopyrite Concentrate

It is now proposed that the NSC pressure leach facility be used to treat a gold bearing chalcopyrite and cobaltite concentrate (72). NSC concentrate leach testing has been conducted at CAMP and the results were as follows.

Table XXXV - Composition of Concentrate Tested

Cu, %	Co, %	Fe, %	As, %	Au, %	Total S, %
7.2	14.4	14.4	20.0	0.42	19.0

Table XXXVI - Nitrogen Species Catalyzed Partial Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	100 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	125°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	20 min

Table XXXVII - Mass Distribution of Nitrogen Species Catalyzed Pressure Leaching of Cobaltite, Chalcopyrite Gold Concentrate

	Cu, %	Co, %	Fe, %	As %	Au, %
Solution	97.3	97.7	97.7	93.4	0.0
Solid	2.7	2.3	2.3	6.6	100.0

As seen the copper, cobalt and arsenic are solubilized and can be separated by conventional means with the sulphide oxidized to sulphur in a minimum amount of time. The gold is concentrated in the solids for recovery by alkaline sulphide leaching solids.

Partial Nitrogen Species Catalyzed Pressure Oxidation of a Zinc Concentrate with Gallium and Germanium

The application of nitrogen species catalyzed oxidative pressure leaching was tested on a high grade North American gallium and germanium bearing zinc concentrate on a lab scale. In this case, the partial oxidation of sulphide to sulphur was the goal while solubilizing zinc, germanium and gallium for selective recovery (73). The test conditions and results are illustrated in Tables XXXVIII, XXXIX and XXXX.

Table XXXVIII - Composition of Sphalerite Concentrate

Zn, %	Fe, %	Ga, g/t	Ge, g/t	Total S, %
64.8	0.4	997	513	31.7

Table XXXIX - Nitrogen Species Catalyzed Partial Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	50 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	125°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	20 min

Table XXXX - Mass Distribution of Nitrogen Species Catalyzed Partial Oxidation Leach Products

	Zn, %	Ga, %	Ge, %	Fe, %
Solution	97.3	98.2	96.3	95.6
Residue	2.7	1.8	3.7	4.4

As can be seen, this industrially proven low temperature and low-pressure system was effective in leaching the zinc, germanium and gallium and oxidizing the sulphide to sulphur in a minimum amount of time. The zinc, gallium and germanium can then be separated and recovered using conventional means.

Partial Nitrogen Species Catalyzed Pressure Oxidation of a Pentlandite Concentrate With In-situ Iron Precipitation

The application of nitrogen species catalyzed oxidative pressure leaching was tested on pentlandite concentrate on a lab scale. In this case, the partial oxidation of sulphide to sulphur was the goal along with in-situ precipitation of iron from pyrrhotite. There were advantages to this operating scenario besides the lower temperatures and pressures. The test conditions and results are illustrated in Tables XXXXI to XXXXIII.

Table XXXXI - Composition of Pentlandite Concentrate

Ni, %	Cu, %	Co, %	Fe, %	Total S, %
16.3	4.1	0.2	30.3	28.9

Table XXXXII - Nitrogen Species Catalyzed Partial Oxidation Leach Conditions

Operating Criteria	Unit
Initial Free Sulphuric Acid	10 g/L
Reactor Working Pressure	620 kPag
Slurry Solids Content	100 g/L
Solids Size	80% -10 micron
Maximum Temperature	125°C
Nitrogen Species Concentration	2.0 g/L
Reaction Time	20 min

Table XXXXIII - Mass Distribution of Nitrogen Species Catalyzed Partial Oxidation Leach Products

	Ni, %	Cu, %	Co, %	Fe, %	Total S, %
Solution	99.5	9.6	97.8	0.8	10.3
Residue	0.5	0.4	2.2	99.2	89.7

As can be seen, this industrially proven low temperature and low-pressure system was effective in leaching the nickel and copper, precipitating the iron in-situ and oxidizing the sulphide to sulphur in a minimum amount of time.

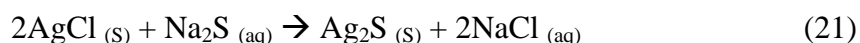
Industrial NSC Plant Recycled Material Application

In the photographic industry, silver and silver halides play a major technological role. Most of the silver utilized is recycled in some fashion. This requires burning of the film to concentrate the precious metals. Then it is generally sent to a pyrometallurgical smelter for recovery. Using NSC pressure leaching, this silver bearing ash was blended with silver bearing copper sulphide concentrates and leached for recovery in the industrial NSC process plant (74). The experimental results are presented in Tables XXXXIV and XXXXV.

Table XXXXIV - Silver Film Ash Assay

Ag	AgCl
7.9%	1.3%

AgCl content is first removed by reacting it with sodium sulphide as:



The resultant silver sulphide was then feed for the nitrogen species catalyzed pressure leach system and it was easily recycled back through the existing plant.

Table XXXXV - NSC Industrial Plant Pressure Oxidation
Recovery of Combined Film Ash and Copper Concentrate

Assay	Ag	Cu	Fe
Solution	96.5%	99.90%	99.28%
Residue	3.4%	0.09%	0.67%
S ^o Pellets	0.1%	0.01%	0.05%

The percentage of silver that reported to the leach solution was 96.5%. Using the existing non-cyanide plant scale technology outlined before, the silver was processed from this solution to a final product of up to 99.999% purity.

DISCUSSION

This paper has outlined the various applications of industrially proven nitrogen species catalyzed pressure (i.e. NSC) oxidation to concentrates, ores and recycled materials. Both the complete and partial oxidation version were illustrated. As well the versatility of the application to some chalcopyrite and gold ore and concentrate flowsheets has been highlighted. The process offers several definitive advantages. First of all it is the only proven industrial process over the long term for pressure leaching of copper sulphides and direct recovery of precious metals without the use of cyanide. Second, the rate of reaction is much faster and subsequent required reactor volume is thus smaller. Third, the process does not require excessively high temperatures or pressures. Fourth, the oxidation reduction solution potential (i.e. ORP) can be adjusted and controlled to be extremely high so it oxidizes almost any sulphide at low oxygen overpressures. Fifth, the materials of construction are readily available stainless steels so there is no need for titanium cladding or brick with lead liners. Thus, the capital and maintenance costs are less. Also, because of the simpler internal design, direct heat exchange can be utilized in-situ for optimal temperature control. Further, in a manner analogous to existing Ni/Co laterite HPAAL systems, the energy from the in-situ heat exchanger can be readily utilized for optimizing the plant heat balance or co-generating electrical power resulting in significant process operating cost savings. Sixth, there is no need for a dip tube or special design radial agitators with cowlings and the like. Oxygen transfer is innate with the enhanced nitrogen species chemistry. So, without titanium, or a titanium dip tube in particular, there is much less oxygen fire danger. Seventh, the design of the feed pump system is far less of a challenge as is the flash system and the choke system. Eighth, like a smelter, precious metals recovery can be high and direct. Ninth, there is no sophisticated chloride chemistry or resultant corrosion issues to deal

with. Tenth, by-product elemental sulphur formation, handling and treatment for contained gold recovery is proven and readily accomplished and value added by-products such as sodium sulphate, sodium hydroxide, sulfuric acid and gypsum can be produced from the waste streams. Finally, there is minor amount of nitrogen species utilized which reports almost entirely to the gas phase upon autoclave flashing. This is readily destroyed and scrubbed using commercially available equipment. So, there are no major economic or environmental issues with the use of the small amount of nitrogen species utilized.

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