To Our Valued Customers,

2009 was a difficult year for many metals markets and suppliers to metals markets. During this period, cost control and production volume reduction were for many the priorities. Many new projects, particularly expansions plans were set aside. Although metals markets are healthier, there are still some residual concerns over the robustness of the global economy and where metals prices are headed.

Throughout the downturn and into 2010, we have retained our commitment to helping you optimize your operations through the delivery of innovative chemical technologies. Our dedication to working collaboratively to develop solutions that meet your specific needs remains at the core of all we do. We believe that this, customer-focused approach combined with our long standing capability to innovate have been and will remain vital to Cytec continuing as the leading provider of reagents to the mining industry. Our 100 year history is a testament of our commitment to the metals mining industry…and to you.

I am thrilled to be joining the Mining Chemicals team at this exciting time. My background in Research and Development serves to reinforce my conviction that the development of technology solutions is a key enabler in sustaining the success of the mining industry. I look forward to learning more about you, your business, and how Cytec can best serve your needs.

Particularly I hope that you enjoy this latest edition of Cytec Solutions and find it to be just one of many valuable resources you can receive from Cytec. We are excited to be sharing technology and ideas that can give you options to improve the performance of your processes. You can be assured that you will see more from us as we bring ever improving and new technologies to you in 2011.

Thank you for your valued business,

Martin Court
Vice President, In Process Separation Technologies
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Introduction

A new series of reagents (The ACORGA NR Series) has been developed by Cytec Industries Inc. for operations with concerns over nitration. These new reagents can be formulated at varying strengths to maximize copper transfer (based on the PLS copper and acid) while maintaining chemical stability under nitrating conditions. Historically plants with high nitrate in their PLS solutions (20 – 40 g/L) or concerns over nitration risk, have elected to use ketoxime (due to its high hydrolytic stability). The use of ketoxime has the disadvantages of reduced copper transfer, copper: iron selectivity, and low copper recovery when the PLS copper grade is high or the pH is low. Often the ketoxime is too weak of a formulation for efficient SX operation.

The new formulations are modified oxime extractants, which demonstrate higher stability than ketoxime extractants under nitrating conditions. These formulations maintain the chemical and physical performance characteristics of the traditional ACORGA products, while allowing customization of the formulation for the specific feed conditions to maximize metallurgical performance.

Chemical Route to Nitration

Oxime nitration is an aromatic electrophilic substitution reaction and requires the presence of nitronium ion (NO$_2^+$); which is produced according to equations (i) and (ii)

\[
\text{NO}_3^- + \text{H}_2\text{SO}_4 \leftrightarrow \text{HNO}_3 + \text{HSO}_4^- \quad (i)
\]

\[
\text{HNO}_3 + \text{H}_2\text{SO}_4 \leftrightarrow \text{NO}_2^+ + \text{H}_2\text{O} + \text{HSO}_4^- \quad (ii)
\]

The mechanism proposed for the nitration of oximes is shown below.

\[
\text{R}_1 \text{H or CH}_3 \text{ (aldoxime or ketoxime)}
\]

\[
\text{R}_2 \text{C}_9\text{H}_{19} \text{ or C}_{12}\text{H}_{23} \text{ (nonylaldoxime or dodecylaldoxime)}
\]

This nitration reaction will occur regardless of the oxime used (aldoxime or ketoxime) if the nitronium ion is present.

Effects of Nitration on Solvent Extraction

Under extreme nitrating conditions, the oxime (aldoxime or ketoxime) will be nitrated resulting in a reduction of copper transfer (Nitrated oxime forms such a strong complex with copper it cannot be stripped under standard conditions resulting in a circulating load of copper within the organic phase). The overall copper production will not be achieved without significant reagent addition to make up for the nitrated oxime. Although reagent may be added to maintain copper production for a short time frame, in time, without corrective action the physical properties of the plant organic will suffer due to the buildup of aldehyde/ ketone or their nitrated equivalents in the organic inventory.

As a consequence, entrainment (both A/O and O/A) will tend to increase which then results in higher impurity concentrations in the electrolyte (potentially affecting cathode quality) and higher reagent loss.
In addition:

- Nitrated oxime has a low solubility, and can precipitate increasing crud formation, and contributing to unstable plant operation.
- The presence of nitrated oxime within a circuit will also tend to reduce copper: iron selectivity, increasing iron transfer to the electrolyte (and the associated costs due to higher bleed requirements or lower current efficiency).
- Although the presence of nitrated oxime in organic does not appear to cause catalytic degradation, when nitration of the oxime does occur, a significant amount of oxime is also degraded via hydrolysis.

**ACORGA NR Series of Reagents**

The NR series of reagents are specially formulated to give enhanced protection to the active oxime when or if the nitronium ion is formed under extract or strip conditions.

To evaluate the NR formulations, static tests were run to evaluate nitration of a conventional modified aldoxime formulation, ketoxime, and the new NR formulation simultaneously.

The test conditions were:

- 60 gpl oxime (~12.3 vol%).
- Temperature: 40 °C
- PLS composition – 60 g/L nitrate ion, 10 g/L chloride ion, 6 g/L copper and 3 g/L ferric ion.
- 600 rpm agitation

The aqueous and organic solutions were mixed for 619 h (~26 days). Samples were taken on a routine basis and analyzed for residual copper, maximum copper load, and copper/iron selectivity. The final organic characteristics were compared to fresh.

Due to the difficulty in reproducing nitration under standard operational conditions (nitration can be a slow process) – it was necessary to use aggressive conditions to evaluate the reagents. Conditions used: extremely high nitrate concentrations, high temperatures, and continuous contact time to force the nitration reaction to occur.

As shown the modified aldoxime and the ketoxime formulations both nitrated in time as evident by the increase in the residual copper. The NR formulation showed no signs of nitration during this test period.

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**ACORGA NR Series of Extractants**

continued
Organic characterization before and after the test are showed in Table 1.

Table 1: Organic characterization before and after static test.

<table>
<thead>
<tr>
<th>CONDITIONS</th>
<th>BEFORE</th>
<th>AFTER 619h</th>
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<tr>
<td>Reagent</td>
<td></td>
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<tr>
<td>Extract Kinetics</td>
<td>Time, s</td>
<td>Approach to Eq., (%)</td>
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<tr>
<td>NR 20</td>
<td>15</td>
<td>92.6</td>
</tr>
<tr>
<td>Ketoxime</td>
<td>30</td>
<td>99.6</td>
</tr>
<tr>
<td></td>
<td>900</td>
<td>100.0</td>
</tr>
<tr>
<td>Extract Isotherm Point (Cu g/l)</td>
<td>5.0</td>
<td>4.4</td>
</tr>
<tr>
<td>Cu/Ir Selectivity</td>
<td>3,831</td>
<td>2,237</td>
</tr>
<tr>
<td>Extract Phase Disengagement</td>
<td>Org Continuity</td>
<td>Org Continuity</td>
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<tr>
<td>Time, s</td>
<td>2.7</td>
<td>116</td>
</tr>
<tr>
<td>NR 20</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ketoxime</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As shown under the accelerated test conditions, the NR formulation demonstrated an advantage over straight ketoxime in terms of nitration, Copper: Iron selectivity, and phase disengagement.

In cases where it is not possible to control / minimize oxime nitration through operational means, or where the risk of nitration is deemed high, a new reagent series (the ACORGA NR series of reagents) with higher chemical stability under nitrating conditions is available. These new formulations, maintain the excellent behavior of the traditional ACORGA products. The formulations provide better nitration resistance than ketoxime while enabling the choice of formulation strength to meet the feed (PLS grade and pH) requirements. The NR formulations allow an improvement in copper transfer vs the commonly used ketoxime.
Introduction

A number of copper SX operations have been identified which contain significant molybdenum values within the leach liquor. At some of these operations the molybdenum is present due to the continuous leaching of molybdenum oxides within the ore; while at others, the Mo is present from other sources such as scrub liquors from smelting operations. Due to large solution inventories, the molybdenum value within the solutions can be substantial. The actual Mo concentration within the leach solution can vary greatly dependent on the acidity of the leach liquor (i.e. Mo can precipitate out within the heaps/dumps dependant on the acidity used).

Cytec Industries Inc. has developed a new modified phosphinic acid reagent formulation (CYANEX® 600) for extracting and purifying molybdenum from these leach liquors. A common problem for recovering molybdenum from these streams in the past has been finding a formulation which would have the right selectivity and kinetic properties to allow the low Mo concentration to be efficiently extracted without altering the standard Cu SXEW process. Due to the unique properties of the formulation it is possible to selectively remove molybdenum while leaving the majority of the impurity elements behind in the leach liquor. Ideally the recovery process would take place downstream of the standard copper SX plant, removing the molybdenum from the acidified copper raffinate stream prior to further leaching.

Acidification of the leach liquor prior to Mo SX allows the majority of other elements typically present in leach liquor (Fe, Cu, Zn, Mg, Si, etc…) to remain in the aqueous while the molybdenum is extracted due to the strong complex formed with the extractant.

Once the molybdenum has been extracted from the aqueous leach solution it is possible to strip and concentrate the Mo using base. In order to avoid or minimize 3rd phase formation, the new formulation contains a phase modifier which enhances the phase separation characteristics. In addition, the stripping solution and stripping conditions need to be closely controlled to maximize Mo stripping while minimizing the formation of the basic salt of the phosphinic acid. The purified and concentrated Mo stream can then be further processed to produce the desired final product.

Molybdenum Extraction

Molybdenum extraction is believed to proceed according to the equation below:

\[
\text{MoO}_2^{(+2)} + 2(H_2R_2) \rightarrow \text{MoO}_2(HR)_2 + 2H^+
\]

The reaction is largely non reversible due to the strength of the molybdenum ligand complex.

Since Mo is typically present in these leach liquors at very low concentrations (5 – 100 ppm), it is advantageous to extract the Mo while utilizing a very low flow O/A ratio. Organic recycle may be utilized to maintain proper mixing characteristics. By concentrating Mo in the organic within the extract circuit, the downstream equipment required for organic processing is minimized. Example: If an operation extracts 20 ppm Mo from a 1000m³/hr raffinate stream while loading the organic to 2 gpl Mo; The loaded organic flow rate to scrub and stripping stages would only be 10 m³/hr.
Organic Scrubbing

Depending on the final molybdenum product purity required (and acidity used in extraction), a scrub stage may be necessary. Acidified water may be used to remove any co-extracted impurities and the process may be optimized to achieve the desired metal separations.

Molybdenum Stripping

Due to the very strong Mo extractant complex, Mo can only be partially back extracted with acid. Molybdenum stripping is best achieved using a base. Molybdenum stripping with ammonium hydroxide is believed to involve a speciation change reaction according to the equation below:

Speciation Change:
\[ \text{MoO}_2^{({\text{+2}})} + 2\text{NH}_4\text{OH} \leftrightarrow \text{MoO}_4^{({\text{-2}})} + 2\text{H}^+ + 2\text{NH}_4^+ \]

Stripping Reaction:
\[ \text{MoO}_2^2(\text{HR})_2 + 2\text{NH}_4\text{OH} \rightarrow (\text{NH}_4)_2\text{MoO}_4 + 2\text{H}_2\text{R} \]

Under these conditions, the formation of the ammonium salt of the phosphinic acid formulation is minimized. However the choice of the base and the ratio of base to ligand / metal must be carefully controlled to prevent third phase formation.

In order to produce a concentrated strip liquor it is advantageous to run a high flow O/A ratio within strip. Aqueous recycle may be used to achieve the necessary mixing conditions. Concentrated base should be utilized to achieve the optimum stripping conditions while minimizing dilution effects.

Crystalization

Ammonium molybdate crystals may be produced from the concentrated strip liquor utilizing multiple methods such as distillation, cooling, selective solubility, etc. In the trials run to date, the molybdenum was recovered from the strip liquor by adjusting the pH of the concentrated solution using sulfuric acid until precipitation of the Mo salt. This was done on a batch basis but could also be achieved continuously by taking a small bleed stream from the aqueous strip liquor. Ammonium heptamolybdate can also be produced in situ through adjustment of the strip liquor pH.

Pilot Plant Results

Molybdenum was recovered, concentrated and purified from a real leach liquor in a 3 week long continuous pilot plant run. The configuration utilized a single extract stage and a single strip stage separated by coalescors to minimize impurity transfer.

The raffinate solution obtained from the copper SX operation was adjusted to a total acidity of 40 gpl H_2SO_4 before being used as the feed to the Mo extraction circuit.

Operating conditions:
Configuration: 1E + 1S
Feed Solution: ~75 ppm Mo and 40 gpl H_2SO_4
Aqueous feed flowrate: 100 mls/min
Organic flow rate: 2 mls/min
Aqueous strip flow rate: 0.045 mls/min
Extraction O/A ratio: 0.02:1 (1:1 including organic recycle)
Stripping O/A ratio: 45:1 (1:1 including aqueous recycle)
The extraction portion of the plant was initially started under 100% organic recycle to build the Mo concentration within the organic phase to a predetermined value. Once the organic Mo content had been reached, a bleed of the organic flow was then sent to the stripping circuit (for this piloting the alternative scrub stage was not utilized). The organic was stripped utilizing an ammonia/ammonium carbonate solution.

The piloting was run for three weeks (24hr/day operation) under operating temperatures of 10 – 15 degrees C. Due to the scale of the operation it was difficult to determine entrainment losses however there were no issues with the physical operation of the plant (dispersion bands, phase disengagement times were consistent and crud build up was negligible). Figure 1 shows a picture of the pilot plant equipment. A standard deep cell (500 ml mix box) was utilized for the extraction stage while a mini rig stage (100 ml mix box) was used as the stripping stage. Note in the picture the second mini rig unit was utilized as a coalescor to minimize aqueous in organic transfer to strip.

During the run, Mo was successfully extracted from the PLS, purified and concentrated to produce a Mo rich liquor containing ~50 gpl Mo. The strip liquor was then treated with sulfuric acid to generate high quality (51.7% pure) ammonium heptamolybdate crystals.

Conclusions

CYANEX 600 has now been used in a number of successful internal / small scale pilotings for the recovery of Mo from leach liquors. Based on the promising results to date, future larger scale pilotings (10 lpm flow rates) are planned.

A longer version of this paper was originally presented at Copper 2010 and can be found on the Cytec website www.cytec.com/miningleaders/
Introduction

Cytec Industries Inc. has developed a new range of formulations with enhanced stability to oxidation. The ACORGA OR series of extractants provide excellent chemical stability under high oxidation-reduction potential (ORP) while maintaining the desired physical and metallurgical characteristics.

Extensive test work and pilot plant trials were run under oxidizing conditions to evaluate the performance of one of the formulations, ACORGA OR25, to oxidative degradation.

Background

Iron and manganese are typically found in the PLS and electrolyte of all copper SX plants. The iron present in the PLS can be transferred to the electrolyte both by chemical (organic loading) and physical means (entrainment, crud movement, or plant upsets), while the manganese will only be transferred physically. If these metal concentrations are not properly managed adverse affects are possible in electrowinning and SX.

The presence of Mn in the electrolyte can be oxidized to the permanganate ion (Mn$^{+7}$ or other valence states) at the anode and then subsequently result in the oxidation of the plant organic: consuming reagent and negatively affecting the physical operation of the plant.

In order to prevent this oxidation, a common industry rule of thumb is to maintain a Fe/Mn ratio of 10/1 (with the expectation that at least 50% of the iron is in the ferrous state) in the electrolyte to prevent the formation of the permanganate ion. Iron present in the electrolyte is reduced at the cathode; the resulting ferrous iron is then oxidized back to ferric iron at the anode in a constant cycle. By maintaining a 10/1 ratio of total iron to Mn within the electrolyte improves the likelihood sufficient ferrous iron will be available to reduce Mn. This ratio ultimately will depend on cell dynamics, where the permanganate ion has been formed during the electrowinning process at Fe/Mn ratios greater than 10/1, while other operations have demonstrated the ability to run at much lower Fe/Mn ratios without generating a high redox potential.

If the electrolyte drops below a 5/1 Fe/Mn ratio, it is possible that the permanganate ion will be formed and oxidative degradation will occur. Operations which have a low iron to manganese ratio in the feed/PLS may have periods where unexpectedly high ORP values are encountered in the electrolyte. This is most likely to occur during periods of high entrainment due to crud runs or plant up-sets. It is during high ORP events that the organic will quickly be oxidized leading to a reduction in copper transfer and longer phase disengagement times (possibly leading to further complications due to increased aqueous in organic entrainments). In these cases, either ferrous sulfate is added to the electrolyte or the electrolyte is passed over scrap metal to quickly reduce the Mn valence state.

In addition to the potential oxidation of Mn to higher valence states Mn is also known to have an effect on lead anode quality. Its presence in the electrolyte should therefore be minimized through operational control and design to minimize aqueous in organic entrainment.

The new oxidation resistant formulations (ACORGA OR series) provide an added level of security for these events and/or when other oxidative conditions exist within the system.
Oxidation Test Procedures

Bench scale and large scale pilot plant tests were completed to evaluate oxidative degradation of the organic by permanganate. In both cases potassium permanganate was added to the electrolyte to simulate what may happen with the electrowinning tankhouse. Phase disengagement time and maximum loads were monitored as well as the effects of clay treatment of the oxidized organic.

Oxidation Results

Bench Scale

Figure 1 shows the affect of repeated contact of a modified aldoxime extractant with an electrolyte containing varying concentrations of permanganate.

Figure 1
Organic Continuous Phase Disengagement

The graph shows that the increase in the strip phase disengagement time is dependent on the amount of permanganate present in the electrolyte and the number of contacts with the permanganate ion.

Figure 2 shows the phase disengagement time of a standard oxime formulation (“unprotected oxime”) and the oxidation resistant formulation ACORGA OR25 when contacted multiple times with permanganate containing electrolyte.

Figure 2
Organic Continuous Strip Phase Disengagement

As shown with elevated permanganate, the oxidation resistant formulation had the same increase in phase disengagement time as would be expected with all organics (likely due to diluent oxidation). However, as shown in figure 3, the active oxime (as designated by the maximum Cu loading capacity) remained constant for the OR formulation while it decreased for the unprotected formulation.
Similar testing was completed under more aggressive oxidative conditions, 200 ppm permanganate in the electrolyte. The test results are shown in Figure 4.

As shown, ACORGA OR25 performed well under high ORP conditions. The maximum copper load decreased 8.2% for the standard formulation while the oxidation resistant formulation’s maximum load only decreased 1.9%.

Table 1 shows the impact of oxidation of the organic phase after 10 contacts with the permanganate ion. Following each contact the ORP value of the electrolyte decreased due to oxidation of the organic and reduction of the Mn+7.

Table 1: Electrolyte ORP and Organic Max Load

<table>
<thead>
<tr>
<th>10 Contacts with Electrolyte at 0.5/1 O/A Ratio</th>
<th>Initial ORP (mV)</th>
<th>Mn+7 (gpl)</th>
<th>Final ORP (mV)</th>
<th>Org ML Cu (gpl)</th>
<th>% Oxime Oxidized</th>
</tr>
</thead>
<tbody>
<tr>
<td>615</td>
<td>1258</td>
<td>172</td>
<td>0</td>
<td>705</td>
<td>84.4</td>
</tr>
<tr>
<td>5.8</td>
<td>4.19</td>
<td>n/a</td>
<td>28%</td>
<td>0.172</td>
<td>0.172</td>
</tr>
</tbody>
</table>

Multiple contacts of the organic phase with a 172 ppm permanganate solution will have a detrimental effect on the copper loading. The data shows a decrease in the max load from 5.8 gpl Cu to 4.19 gpl corresponding to 28% of the oxime being oxidized.

Pilot Plant Results

A side by side pilot plant was run using an unprotected oxime and ACORGA OR25. Three conditions were tested during the pilot trial:

1. “standard operating conditions” targeting 90% copper recovery
2. oxidative conditions (permanganate dosed into Lean electrolyte)
3. clay treatment of the organic followed by “standard conditions”
Physical measurements were taken during the pilot plant trial and copper maximum loads were measured pre and post the oxidation step.

The E1 stage organic continuous phase disengagement times are shown in Figure 5 below.

Figure 5
E1 Extract Stage Phase Disengagement Time

The data shows an increase in the organic continuous phase disengagement time under oxidized conditions. This increase was significant for an unprotected oxime, greater than 600 seconds while the phase disengagement time of ACORGA OR25 surprisingly remained under 150 seconds. Due to the increase in phase disengagement time, the total run time under oxidizing conditions was minimized to prevent settler flooding.

Clay treatment of the organic phase did improve the phase disengagement times but not equivalently to the initial organic quality (likely due to insufficient clay treatment).

The strip phase disengagement times for both reagents are shown in Figure 6.

Figure 6
Strip Phase Disengagement Times

The phase disengagement times of both reagents were similar in the strip stage (consistent with the bench test results).

The maximum copper load of each reagent was tested prior, during and after operation under oxidizing conditions. During the test, approximately 8% of the oxime in an unprotected oxime degraded while only 2% of the oxime in the oxidation resistant formulation degraded.
Conclusions

Prior to the development of the ACORGA OR series of reagents – there has been no copper SX formulations designed to protect against oxidative degradation.

To prevent oxidation of the organic phase due to high ORP values, the Fe$^{2+}$/Mn ratio in the electrolyte and ORP should be monitored and controlled. During plant upset conditions, steps should be taken to quickly bring the electrolyte ORP back under control.

The use of Cytec’s oxidation resistant formulations will provide an extra level of security to address temporary permanganate issues or other conditions resulting in oxidation (short term or ongoing).
Introduction

Cytec Industries Inc. has developed a simulation software package to assist operators and engineering companies who wish to optimize and design solvent extraction circuits for CYANEX 272. These new modeling capabilities are expected to significantly reduce the amount of laboratory work required by the designer while increasing confidence in the ability to achieve the desired metal separations. This new in-house capability allows one to evaluate the expected impact of various changes to the PLS metal composition, reagent concentration, O/A ratios, pH profile, and overall circuit configuration/layout.

Historic Methodologies for Circuit Design

Historically, in order to have a first approximation of the extract staging and O/A ratio required for a given Co recovery, a reagent concentration would be chosen, and an isotherm would be generated for a given pH. The cobalt isotherm could then be graphed and the staging estimated via McCabe Thiele analysis. Figures 1 shows a McCabe Thiele diagram for cobalt based on a feed solution initially containing 2.5 g/L cobalt, 0.1 g/L manganese, 5 g/L magnesium, 0.2 g/L calcium and 10.0 g/L nickel. The isotherm was generated using 10% v/v CYANEX 272 at 50°C and an equilibrium pH of 6-6.2.

As shown, the McCabe Thiele analysis indicates cobalt could be efficiently extracted from the PLS using 2 stages in extract and an O/A ratio of 1.00. The diagram does not however give any indication of the performance of the other metals. McCabe Thiele techniques can not be used to accurately assess a circuit when multiple metals are loading. In addition, for a typical application, a different pH would be utilized in each stage (typically higher pH in the raffinate stage, with a decreasing pH profile towards the loaded organic or E1 stage). Since the aqueous feed to each stage is typically at a different pH, separate extract isotherms would need to be generated for each stage to more accurately predict staging requirements.

For these reasons, CYANEX 272 circuits are typically piloted under a number of conditions: pH, O/A ratios, staging (extract, scrub, and strip), scrub solution compositions, etc. to estimate the impurity transfer and expected recovery for given feed conditions.
CYANEX Modeling

Cytec industries Inc.’s new modeling capabilities have been designed to significantly simplify the overall design process. The simulation software does not rely upon McCabe Thiele techniques but the simultaneous solution of multiple equilibrium calculations based on pre-generated equilibrium curves. The pre-generated equilibrium data covers various metals, metal concentrations, acidities, ligand concentrations, and temperatures.

Given a known feed composition, O/A ratio, targeted equilibrium pH, expected stage efficiency, and ligand concentration, the program will calculate the aqueous and organic mixer outlet composition. Based on standard chemical engineering techniques, the equilibrium calculations can be used to obtain an iterative solution to the expected overall plant (extract, scrub, and strip staging) performance.

Figures 2-4 show the simulated output for the extraction, scrubbing and stripping sections of a solvent extraction plant.

Extract Conditions: PLS (g/L): 2.5 Co; 10.0 Mg; 50.0 Ni; 10% v/v CYANEX 272, O/A=1, Temperature=25°C, 3 extraction stages, pH: 5.0 (E1), 5.2 (E2) and 5.4 (E3).

The loaded organic composition from the extraction circuit is then fed to the scrubbing circuit to estimate the scrub organic and scrub aqueous compositions.

Scrub Conditions: Loaded Organic (g/L): 2.498 Co; 0.446 Mg; 0.118 Ni; 10% v/v CYANEX 272, Scrub Liquor (g/L): 40.0 Co; O/A=20; Temperature=25°C; 2 scrub stages, pH: 4.50 (Sc1) and 4.2 (Sc2).

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The scrubbed loaded organic composition is then fed to the stripping section to simulate the barren organic and rich electrolyte compositions.

Strip Conditions: Loaded Organic (g/L): 3.046 (Co) + 0.008 (Mg) + 0.000 (Ni), 10% v/v CYANEX 272, Strip Liquor: Sulfuric acid, O/A=15, Temperature=25°C, 2 strip stages, pH: 2.0 (S1) and 2.0 (S2).

Figure 4
Process Flow Diagram (Stripping Section)

The program allows alteration of the feed, scrub, or electrolyte compositions, O/A ratios, pH profiles, or reagent concentration to allow a quick assessment of the expected circuit performance.

Piloting Results

A piloting was recently completed to allow comparison of expected and measured results under various conditions. In this case the pilot plant utilized 10 vol% CYANEX 272 to treat a feed containing 2.97 gpl cobalt and 4.99 gpl nickel. For pH control, the barren organic from the strip circuit was preneutralized with 10 M NaOH (i.e. 400 g/L) to achieve a preneutralization of 15% of the ligand. The measured and predicted metal concentrations are denoted by “(M)” and “(P)”.

As shown, the program provides a reasonable prediction. Variations between measured (M) and predicted (P) data are attributed to minor variation in recorded pH, as well as variations in stage efficiency. The accuracy of the model is highly dependent on the solutions reaching equilibrium and accurate pH and flow control.
Summary and Next Steps

Cytec’s modeling and predictive capabilities associated with CYANEX 272 have substantially advanced. The tool and skills that have been developed, will continue to advance as Cytec continues to invest in expanding modeling capabilities. The program has been used successfully to evaluate operational parameters at existing operations and is being used to assist in multiple design scenarios. The software can aid in the design of plants by modeling the performance under multiple staging scenarios to find the optimal configuration and potentially minimizing the overall staging requirements. It also can be used to optimize existing operations by providing quick guidance to reach the optimal O/A ratio, pH profile, or reagent concentration thus maximizing metal transfer while minimizing impurity transfer. Cytec believes there is substantial opportunity to improve the economic performance of both existing and future operations. The modeling capabilities should allow optimization of CYANEX 272 circuits while allowing easy economic assessment of the benefits of Co SX.

A longer version of this paper was originally presented at ALTA 2010 and can be found on the Cytec website www.cytec.com/miningleaders/
Introduction

In the early 1990’s the U.S. Bureau of Mines (USBM) Reno Research Center undertook research to evaluate options for electrowinning copper without acid mist generation. This would require that the conventional anode reaction in copper electrowinning, the decomposition of water to form oxygen gas, be replaced with an alternative anode reaction that was free from gas evolution. In 1995, USBM published a pilot plant study where-in copper electrowinning was carried out using the ferrous/ferric anode reaction in conjunction with reduction of ferric iron external to the cell using sulfur dioxide and an activated carbon catalyst [1].

The Freeport-McMoRan Technology Center became interested in AART in 2002 because of the potential for decreasing energy consumption in copper electrowinning (due to voltage reduction in the tankhouse and the potential to generate electricity from burning sulfur to provide sulfur dioxide for the process). Co-generation of sulfuric acid was also of interest for use in copper leaching. In 2003, the Technology Center’s SXEW Test Facility, a pilot-scale demonstration plant, was converted for continuous demonstration of AART to evaluate these benefits against addition capital and operating costs of the process.

Methods and Procedures

The process flow diagram for AART at the Test Facility matched conventional SXEW flow except that the lean copper electrolyte from electrowinning was diverted through the absorption tower where sulfur dioxide was absorbed into the electrolyte. This mixture was then passed through an activated carbon module for conversion of ferric iron to ferrous iron (and the resultant production of sulfuric acid from sulfur dioxide). A slip stream from the carbon module effluent was sent to the acid recovery unit for removal of sulfuric acid. The carbon module effluent and the acid-depleted electrolyte were returned to the plant recirculation tank where they were mixed with rich electrolyte from solvent extraction.

The economics of AART are dependent on power cost (AART decreases cell voltage and generates power through sulfur burning), acid credit to the project (AART produces sulfuric acid), sulfur cost (from which sulfur dioxide is generated to feed the process) and capital costs of incremental AART equipment. The focus of the demonstration was to operate AART under commercial plant operating conditions and to develop the information needed for an economic analysis.

Results and Discussion

Electrowinning

Electrowinning was carried out with four electrowinning cells in series, each containing six stainless steel cathodes and seven anodes. Active surface area of each cathode was 3.5 by 3.1 feet. Amperage was 3,570 amps, which corresponded to a copper production rate of just over 31 lbs/hr at 84% current efficiency. Current density was 27 amps/ft$^2$ of plating surface. Electrolyte flow was 19 gpm per cell, which corresponded to a specific flow rate of 0.14 gpm/ft$^2$ of plating surface. An electrolyte distribution manifold was utilized in the bottom of each cell. Lean electrolyte (i.e. electrowinning cell discharge) chemistry parameters maintained during operation were 38 g/L Cu, 27 g/L Fe, 3.5 g/L Fe$^{3+}$, 180 g/L sulfuric acid, and 125 °F electrolyte temperature.
Demonstration of voltage savings through continuous operation of AART using projected operating conditions was one of the goals of this study. Table 1 displays the average cell voltage measured during continuous operation with the anode types shown. With implementation of ferrous/ferric electrowinning, oxygen gas generation ceased at the anode and acid misting in the tankhouse was completely eliminated.

Each anode design in Table 1 was a flow-through anode that allowed electrolyte to move through the screens. This was shown to be beneficial for AART voltage reduction [2]. The stainless steel anodes were not superior in voltage performance to the titanium anodes but the cost differential made the stainless steel anodes attractive in order to decrease capital cost. Still, the lower voltage achieved with the coated titanium anodes may make them attractive for a particular project depending on power costs.

Table 1: Average cell voltage results versus type of anode.

<table>
<thead>
<tr>
<th>Anode</th>
<th>Cell Voltage, Volts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium mesh with metal oxide coating</td>
<td>1.00</td>
</tr>
<tr>
<td>316 stainless steel woven wire screen (1 screen)</td>
<td>1.13</td>
</tr>
<tr>
<td>316 stainless steel woven wire screen (2 screens)</td>
<td>1.07</td>
</tr>
<tr>
<td>Titanium mesh with metal oxide coating</td>
<td>0.86</td>
</tr>
</tbody>
</table>

As shown in Table 1, making the stainless steel anode with two screens on each side decreased voltage by 60 mV. More importantly, addition of the second screen helped protect the anode from corrosion. The 1.07 volts measured with the two-screen stainless steel anodes represents a 46% reduction in cell voltage versus conventional electrowinning with Pb-Ca-Sn anodes (which typically operate at 2.0 volts).

Copper cathode produced during the AART demonstration measured less than 10 ppm sulfur and less than 3 ppm iron, which met Freeport-McMoRan’s standards for “AA” cathode. No lead was present in the circuit because of the anodes used. Current efficiency of the AART process averaged 84%. Total iron concentration in electrolyte averaged 29.9 g/L. Cell inlet ferric concentration averaged 1.1 g/L. Cell overflow ferric concentration averaged 3.4 g/L.

Absorption of Sulfur Dioxide

The AART demonstration at the Test Facility was successful in continuously absorbing sulfur dioxide into copper electrolyte. The tower was operated with 2-inch tri-packs in the 0 to 12.5-foot zone, 1.5-inch Pall rings in the 12.5 to 25.4-foot zone, and 2-inch tri-packs in the 25.4 to 31.1-foot zone. With 6 feet of additional tower height, an exit gas of 182 ppm was achieved versus the predicted 75 ppm. Although sulfur dioxide solubility was somewhat less than predicted, the Technology Center determined that absorption of sulfur dioxide into copper electrolyte was technically and economically feasible.

Ferric Reduction with Sulfur Dioxide

Electrolyte containing sulfur dioxide from the absorption tower was pumped through three activated carbon modules of varying sizes in order to test residence time versus specific flow rate through the carbon bed. In the Test Facility demonstration, a residence time of 2 minutes was required (at a specific flow rate of 24 gpm/ft²) in a carbon module to fully react the sulfur dioxide and a 1 g/L ferric concentration was maintained in the carbon module discharge to insure complete reaction. Control of this process was accomplished using Ag/AgCl ORP sensors. The activated carbon used in the AART demonstration did
not lose its efficacy in the demonstration period. A mechanism that degrades the activated carbon so as to disproportionately reduce its efficiency during the AART process was not observed, suggesting a long lifetime for activated carbon use in the process.

**Sulfuric Acid Recovery**

The acid recovery unit utilized a fixed bed of resin that retarded the flow of sulfuric acid through the bed. The acid recovery unit performed consistently and for the most part operator-free. A peak acid concentration of 140 g/L was achieved in the by-product acid with a feed containing 180 g/L acid. The unit was cyclic in nature. Plant water was used to rinse the sulfuric acid from the resin bed. Both copper and iron from the feed solution were entrained in the by-product acid in concentrations of 5 to 10 g/L. This entrainment could be decreased by decreasing the concentration of the by-product acid.

**Solvent Extraction**

The solvent extraction process for AART would have been the same as conventional SXEW circuits except for the requirement to provide both copper and iron make-up to off-set losses in the by-product acid stream. The needed iron make-up required by AART can be addressed through solvent extraction if the extractant utilized extracts iron in sufficient quantities from the pregnant leach solution (PLS). With this approach the Cu/Fe transfer ratio in AART solvent extraction needs to be low, in the 15/1 to 20/1 range. This is a reversal of direction in terms of modern extractant development which has sought to maximize Cu/Fe selectivity, achieving well over 1000/1 in conventional SXEW plants. Of course other approaches are possible to make up iron in the AART circuit. A smaller-scale SX circuit could be implemented to extract only iron from the PLS; or iron as iron sulfate could be added directly to the circuit if economically feasible.

Table 2: Average ACORGA M6000 solvent extraction results.

<table>
<thead>
<tr>
<th></th>
<th>Cu, g/L</th>
<th>Fe, g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Aqueous Assays</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PLS</td>
<td>1.6</td>
<td>2.3</td>
</tr>
<tr>
<td>Raffinate</td>
<td>0.04</td>
<td>---</td>
</tr>
<tr>
<td>Lean Electrolyte</td>
<td>37.7</td>
<td>27.6</td>
</tr>
<tr>
<td>Rich Electrolyte</td>
<td>43.4</td>
<td>28.7</td>
</tr>
<tr>
<td><strong>Organic Assays</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Loaded Organic</td>
<td>7.0</td>
<td>0.111</td>
</tr>
<tr>
<td>Barren Organic</td>
<td>4.2</td>
<td>0.048</td>
</tr>
<tr>
<td>Net Transfer</td>
<td>2.8</td>
<td>0.063</td>
</tr>
</tbody>
</table>

The Test Facility utilized a new extractant, ACORGA M6000 from Cytec Industries Inc., at a concentration of 18% by volume in Orform® SX-80 diluent for the AART demonstration. The SX circuit was operated in a S1-E1-E2 configuration. Solvent extraction results are shown in Table 2. Copper recovery averaged 97%. The Cu/Fe ratio averaged 44 based on the organic assays. The aqueous assays (rich versus lean) showed a lower Cu/Fe ratio, but this was due to the sensitivity of the iron assay (by atomic absorption) at these high iron levels.

The ACORGA M6000 extractant performed well but did not quite reach the Cu/Fe selectivity goal of 15 to 20. Additional work by Cytec resulted in the development of ACORGA M6001 which was shown on a bench-scale to achieve the desired selectivity target.
AART Economics

A prefeasibility study was conducted based on the results of the AART demonstration at the Test Facility and applied to an operating commercial plant scenario. A copper production rate of 200,000 tons copper annually was utilized in the study. Key assumptions of the analysis are shown in Table 3. The burning of diesel to heat electrolyte would be replaced by steam heating from the sulfur burner.

Table 3: Key assumptions utilized in AART economic analysis.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper production rate</td>
<td>200,000 tons/year</td>
</tr>
<tr>
<td>Electrical power cost</td>
<td>$0.0848/kWh</td>
</tr>
<tr>
<td>Sulfur cost</td>
<td>$76.79/ton</td>
</tr>
<tr>
<td>Acid credit to project</td>
<td>$80.60/ton</td>
</tr>
<tr>
<td>Diesel cost (averted) for electrolyte heating</td>
<td>$1.38/gallon</td>
</tr>
</tbody>
</table>

Results of the economic analysis are presented in Table 4. The AART operating savings was estimated to be $0.0914/lb copper (approximately $37 million operating savings annually) as shown in Table 4. Capital cost was estimated to be approximately $150 million.

It is interesting to note in Table 4 that the largest operating savings was the acid credit for the by-product acid. This acid would replace a portion of the forecasted acid purchases for the leaching process. After the acid credit the power savings from reducing cell voltage in the tankhouse was the next largest savings. The largest operating cost for AART was the purchase of sulfur. Replacing conventional Pb-Ca-Sn anodes with stainless steel anodes eliminated the need for cobalt additions to electrolyte and also cleaning of lead sludge from electrowinning cells. Complete elimination of acid misting in the tankhouse eliminated the need to add a mist suppressant to electrolyte and the need for cell cover blankets.

Table 4: Prefeasibility study results for AART operating savings.

<table>
<thead>
<tr>
<th>Savings</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>0.0296 $/lb Cu</td>
</tr>
<tr>
<td>Diesel for electrolyte heating</td>
<td>0.0114</td>
</tr>
<tr>
<td>Labor for cell cleaning</td>
<td>0.0004</td>
</tr>
<tr>
<td>Cobalt</td>
<td>0.0071</td>
</tr>
<tr>
<td>Mist suppressant</td>
<td>0.0019</td>
</tr>
<tr>
<td>Cell cover blankets &amp; maintenance</td>
<td>0.0006</td>
</tr>
<tr>
<td>No lead anode replacement cost</td>
<td>0.0034</td>
</tr>
<tr>
<td>Acid credit for forecast demand</td>
<td>0.0622</td>
</tr>
<tr>
<td><strong>Total Savings</strong></td>
<td><strong>0.1166 $/lb Cu</strong></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Costs</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>SO2 (incl. sulfur, labor, consumables)</td>
<td>0.0227 $/lb Cu</td>
</tr>
<tr>
<td>Power for AART increment</td>
<td>(0.0018)</td>
</tr>
<tr>
<td>Carbon make up</td>
<td>0.0006</td>
</tr>
<tr>
<td>Resin make up (acid recovery)</td>
<td>0.0002</td>
</tr>
<tr>
<td>Fe SX Extractant</td>
<td>0.0002</td>
</tr>
<tr>
<td>Other operating supplies</td>
<td>0.0005</td>
</tr>
<tr>
<td>Repair supplies</td>
<td>0.0020</td>
</tr>
<tr>
<td>AART anodes maintenance</td>
<td>0.0008</td>
</tr>
<tr>
<td><strong>Total Costs</strong></td>
<td><strong>0.0252 $/lb Cu</strong></td>
</tr>
</tbody>
</table>

| Net AART Savings                | 0.0914 $/lb Cu |
Summary

Test Facility operation was successful in demonstrating the benefits of AART, which included operating at a cell voltage of approximately 1 volt, eliminating acid mist in the tankhouse, continuously absorbing sulfur dioxide into copper electrolyte, and converting sulfur dioxide into a 140 g/L sulfuric acid for use in leaching. Unit operations were demonstrated, including absorption of sulfur dioxide into copper electrolyte, conversion of sulfur dioxide to sulfuric acid in activated carbon modules, and recovery of the sulfuric acid using an acid recovery unit.

Results of a prefeasibility study for implementation of AART in an SXEW plant with 200,000 tons/year copper production estimated an operating savings of $0.0914/lb copper (approximately $37 million annually) at a capital cost of approximately $150 million. Commercialization of AART is viable but will depend on the relative costs of power, sulfur, sulfuric acid and availability of capital funds.

This article is based on paper presented at Copper 2010 in Hamburg, Germany. See conference proceedings for full paper details.

References


2. SANDOVAL, S., COOK, P., HOFFMAN, W., and ROBINSON, T: Method and apparatus for Electrowinning Copper using the Ferrous/Ferric Anode Reaction and a Flow-through Anode, United States Patent 7368049.
Traditional Reductionist Process

In the 1920s and 1930s, the number of commercially used flotation reagents was relatively small, and reagent selection for a given application was relatively straightforward. As the number and diversity of commercially available flotation reagents steadily increased to meet the ever-increasing technical and economic challenges, reagent selection became gradually more complicated. Four other factors have made the situation more acute:

- The obsessive research focus on “old” reagents (e.g., xanthate, fatty acid, cyanide) meant that the chemistry and applications knowledge for all other reagents remained woefully inadequate in the published literature.
- Science did not keep pace with technology; the increasing emphasis on reductionistic research over the decades meant a widening gap between research and practice.
- Application knowledge developed by in-house research in the chemical companies remained largely inaccessible in the public domain.
- There has been a steady decline of expertise in flotation reagents in the industry over the decades.

Consequently, at present, the process of reagent selection and optimization can be characterized as rather informal, reductionistic, and frequently based on extension of personal experience from one mineral system or plant to another, gut feeling, anecdotes, and myths (Nagaraj and Bruery 2003; Nagaraj 2005). There is no recognized “standard” practice, and the informal process is fraught with pitfalls.

For a given separation, at first glance the pool of reagent classes—and homologues within each class—available for selection appears to be large and bewildering. This is partly the result of the evolutionary nature of reagent development over the decades. But more importantly, given the complexity of the flotation system, a particular reagent class or a specific homologue just happens to produce the optimum results for the separation and ore type in question. General guidelines are available for reagent selection in the form of an accumulated knowledge base in reagent developers’ handbooks and product literature (e.g., the Mining Chemicals Handbook [Cytec 2002]). This serves to narrow the selection for a given application; however, the metallurgist must still use additional filters to refine the selection to a manageable level in order to move to the laboratory testing phase. Historically, this stage of reagent selection has tended to be reductionistic and informal. Problems in complex systems invariably have multiple solutions, each with its degree of desirability. Even an arbitrary process of reagent selection will provide a solution to a plant’s problem. However, such a solution may not be sufficiently robust, that is, the selected reagent will have a narrow, unoptimized window of performance—and may only be a temporary fix—and its nonrobustness may not even be discovered for a long time, given the significant routine variability in plant performance. This approach is very costly in the long run and adds little to our knowledge base.

An Expanded Holistic Process – Cytec’s FLOTATION MATRIX 100®

To achieve an optimized and sustainable process, the mining companies have three major factors to work with: the mechanical, operational and chemical aspects of flotation. Typically the optimization of mechanical and operational factors is subjected to significant capital investments; however the chemicals can frequently have greater leverage in optimizing the process with little or none investment.
Cytec has developed a novel holistic approach to optimize the flotation process called FLOTATION MATRIX 100®, that encompass all the synergistic effects of the chemicals used in the process: collector, frother and modifiers; while incorporating all the factors to make the optimization practical and economical.

The process comprises of four critical phases schematically represented in the Figure 1 below. Although these four phases proceed in a logical sequence, they are indeed highly iterative. The discovery/definition phase provides all the necessary information for reagent selection and sets the objectives, goals and success criteria. The reagent selection phase, then, begins with preliminary screening of available reagents using knowledge base and expertise of the reagent developer to arrive at a subset of reagents that meet the requirements established in the discovery/definition phase. The selected reagents can then be screened in the laboratory by an iterative process. Historically, the preliminary screening phase has been rather simplistic, as it involved merely selecting representative candidates from several different families, and essentially ignoring other important reagents in the circuit and operational variables. Thus, if the objective is to screen collectors for a given separation, the candidate products might be a set of representative collectors from several families, keeping all other reagents and conditions constant. The assumption in this one-reagent-at-a-time approach is that reagents perform independently of one another and independent of the operating conditions.

This holistic approach for reagent selection is based on the consideration of more than just the chemistry of one reagent in isolation or more than just one type of reagent for a given mineralogy. In fact, all the chemical and operational factors are considered simultaneously.

**Case studies- Application of FLOTATION MATRIX 100**

**Collector – Xanthate replacement with newer chemistries in sulfide Cu flotation**

**Case Study:** Using its proprietary FLOTATION MATRIX 100 process for mineral flotation, Cytec has developed a number of new reagents that are delivering measurable improvements over existing technology. One such reagent is AERO® MX-5128 promoter, an AERO XD-5002 promoter formulation, evaluated in copper / Mo operation in the European region. AERO MX-5128 promoter was tested in conjunction with OREPREP®
F-549 frother to improve the copper and molybdenum metallurgy over the current reagent suite of Potassium Isobutyl Xanthate (KBX), diesel oil and OPSB frother.

The recovery data pre-trial was statistically treated to determine plant variability and the trial results are presented in the Figures 2, 3 and 4:

Figure 2
Copper Recovery data

Figure 3
Copper Concentrate Grade data
The period around the trial appears to be an optimum in terms of recovery of both copper and molybdenum in the operation; therefore, to have a better interpretation of the results the data was normalized based in both the head grades and grinding. The normalized results are shown in the table 1.

**Table 1: Normalized recoveries:**

<table>
<thead>
<tr>
<th>Period</th>
<th>Mo Recovery (%)</th>
<th>Cu Recovery (%)</th>
<th>Normalized for Head Grade</th>
<th>Normalized for Grind</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>5 Months Pretrial</strong></td>
<td>69.72</td>
<td>67.38</td>
<td>69.72</td>
<td>67.38</td>
</tr>
<tr>
<td><strong>4 Weeks Pre, 1 Week Post Trial</strong></td>
<td>76.63</td>
<td>70.99</td>
<td>75.30</td>
<td>70.91</td>
</tr>
<tr>
<td><strong>Overall Trial</strong></td>
<td>80.84</td>
<td>72.29</td>
<td>77.80</td>
<td>71.95</td>
</tr>
<tr>
<td><strong>MX-5128 + OP F-549</strong></td>
<td>80.74</td>
<td>73.03</td>
<td>78.45</td>
<td>72.59</td>
</tr>
</tbody>
</table>

Based on this normalized comparison, the increase in copper recovery of about 2% and in molybdenum of 3.7% is statistically significant with 90% confidence.

**Case Study:** In this example, AEROPHINE® 3418A promoter has demonstrated benefits in replacing xanthates by providing significant metallurgical improvements and other benefits like significantly lower dosage and optimization of circuit performance, due to its unusually rapid flotation kinetics and operational stability.

In this example, a Cu-Zn ore floated at a rate of 10,000 t/d capacity using a conventional Cu flotation followed by copper sulfide activation and zinc flotation. After successful laboratory tests, AEROPHINE 3418A promoter was introduced for a full-scale, ten-day, plant trial and the performance of AEROPHINE 3418A promoter was compared to the plant’s standard PAX (potassium amyl xanthate).
As can be seen from the Table 2 below and Figure 5, AEROPHINE 3418A promoter provided copper recovery increase of about 8%, while reducing the reagent consumption by 30% with no change in frother consumption. AEROPHINE 3418A promoter also provided minor improvements in zinc grade and recoveries. Zinc recovery in the copper concentrate and copper recovery in the zinc concentrate were similar for both reagents. The improvement obtained in the copper metallurgy translates into substantial, about 8%, copper recovery increase.

Table 2: The results of a typical ten-day period

<table>
<thead>
<tr>
<th>Parameters</th>
<th>AEROPHINE 3418A</th>
<th>Sodium Amyl Xanthate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heads % Cu</td>
<td>0.97</td>
<td>0.84</td>
</tr>
<tr>
<td>% Zn</td>
<td>1.21</td>
<td>1.59</td>
</tr>
<tr>
<td>Cu Conc. % Cu</td>
<td>28.3</td>
<td>28.4</td>
</tr>
<tr>
<td>Cu Tails % Cu</td>
<td>0.10</td>
<td>0.17</td>
</tr>
<tr>
<td>Cu Recovery %</td>
<td>88.6</td>
<td>80.7</td>
</tr>
<tr>
<td>Zn Conc. % Zn</td>
<td>51.0</td>
<td>48.3</td>
</tr>
<tr>
<td>Zn Tails % Zn</td>
<td>0.24</td>
<td>0.32</td>
</tr>
<tr>
<td>Zn Recovery % Zn</td>
<td>80.7</td>
<td>80.6</td>
</tr>
<tr>
<td>Reagent Consumption (g/t)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu Rougher Float</td>
<td>17</td>
<td>24</td>
</tr>
<tr>
<td>Zn Rougher Float</td>
<td>0*</td>
<td>10</td>
</tr>
</tbody>
</table>

With AEROPHINE 3418A promoter being fed on the Cu rougher float, no additional collector was required in the Zn float.
MINERAL: Southwestern U.S. Copper / Molybdenum Ore

**Operational parameters:**
- **Feed Grade:**
  - Cu: 0.35%
  - Mo: 0.037%
- **Tonnage:** 115,000 tpd
- **Flotation pH:** 11.6
- **Grind:** 35% +65 Tyler mesh

**Reagent Scheme:**
- **Collector:** Thionocarbamate and Aromatic fuel oil
- **Fothing:** Blend of 85% MIBC / 15%
- **OREPREP® X-133 Fothing** @ 25 gpt
- **Frother tested:** OREPREP F-603 @ 25gpt

**Plant Trial:**
One grinding and flotation test section was compared to the remaining standard seven sections on its side for a period of 4 months. During the 4 month trial, 214 test comparisons were made from 255 test shifts and compared to 237 standard comparisons that were made during a 6 month pre and during trial period from 365 standard shifts. Results are presented in the Table 3.

### Table 3: Rougher Flotation Recovery Results

<table>
<thead>
<tr>
<th></th>
<th>% Cu Recovery</th>
<th>% Mo Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Standard</strong></td>
<td>84.1</td>
<td>78.1</td>
</tr>
<tr>
<td><strong>OREPREP F-603 frother</strong></td>
<td>85.8</td>
<td>79.3</td>
</tr>
</tbody>
</table>

The trialed frother OREPREP F-603 frother improved the test section results by an average of 1.72% in copper recovery and an average of 1.23% in Mo recovery.

Statistical analysis (t-test for samples with unequal variances) shows that this difference is significant at the 95% confidence level; proving the metallurgical enhancement in the process when using the frother OREPREP F-603 frother as a replacement of the standard MIBC blend.

**MODIFIER – Enabling the processing of challenging base metal ores**

The mining industry reached a point in which the “good” mineral reserves were depleted and the more challenging ores cannot be treated with the standard technologies and processes. Therefore, new technologies need to be developed to convert these resources into profitable reserves. In mining reagents, collectors and frothers are considered as major players to achieve successful flotation; however, modifiers are the unsung reagents that help optimize a plant’s processes. Unfortunately, modifiers are often avoided because, although they can help to amplify gains, they can equally hinder gains when not properly understood and dosed without statistical analysis, as is also the case with collectors and frothers.

Cytec has been working to better understand the interactions and effects of modifiers. Current efforts are converting untreatable resources into profitable reserves as slimes are controlled with new modifiers. Cytec has been working on a nickel ore that has demonstrated untreatable slime characteristics.
Below, in Figure 6, are demonstrations of the test results showing the marked improvement when using a Cytec chemical system of frother, collector, and modifier when compared to the standard reagent suite: xanthates and MIBC.

Figure 6
Concentrate grade and recovery results

The metallurgical benefits are a significant increase in both nickel and sulfur recoveries from 75% to higher than 90%, while keeping the MgO content in the concentrate in specification. Other than that, the new chemical system composed of collector, frother and modifier; allow the elimination of the desliming circuit and the possibility of processing at higher solids what enables significant savings in energy and costs.

Conclusion

The mining industry’s major need is to improve the efficiency of plant operation with a special emphasis on value recovery in the most profitable manner. Solutions to this overarching need lie in the total system or holistic approach, first for the whole operation comprising geology, mine, comminution, concentrating units, and downstream operations. The focus next would be within an individual unit operation, without losing sight of interlinks between unit operations, and incorporating the physical, chemical, and operating factors simultaneously rather than optimizing one factor at a time. Furthermore, proper reagent optimization has the unique ability to add value outside the processing realm by expanding the utilizable reserves of mines, as it converts previously untreatable assayed resources into profitable reserves.

As presented, Cytec Industries Inc. continues to collaborate with mines to develop solutions that deliver the highest value, by conservation of raw materials and operational variations.
Bibliography


Introduction

Rising demand and high gold price along with dwindling reserves of easy to beneficiate ore bodies have created the necessity for the development of novel mineral processing reagents capable of enhancing precious metal recoveries. This need is more prevalent in the beneficiation of these ores by froth flotation. An internal market study demonstrated the need for new, stronger, more versatile flotation collectors.

Froth flotation, a process used since the early 1900's, consist of introducing various chemicals into an agitated pulp or suspension of finely ground ore and water, followed by the introduction of air to generate a mineral laden froth which is subsequently removed from the agitated vessel (float cell) to generate a concentrate of the wanted mineral entity.

One of the critical reagents is the collector whose role is to adsorb on to targeted mineral and metal surfaces, making these hydrophobic. Chemically speaking, collectors are surfactants, which consist of a hydrophobic tail and hydrophilic, ionic head. The ionic head attaches to the mineral particle, while the tail end provides hydrophobicity to the mineral particle-collector aggregate. These hydrophobic particles then attach to air bubbles and float. A short chain alcohol frother is also used to provide a froth to capture create a froth that will support the collected minerals.

Product Development

Collector Design and Implementation Strategy

Mineral collectors have a hydrocarbon tail, (hydrophobic) and a functional group (hydrophyllic). Collector structure (Chain length, doner atom(s), alkyl type), affects the properties the products properties (pKa, hydrophobicity, toxicity, selectivity).

![Collector Design Diagram](image-url)
The following parameters were to drive the development process:

- Use Knowledge-base of successful chemistries developed over 70 years in base metal flotation.
- Combine expertise and proprietary position in phosphorous chemistry (Welland & Mt. Pleasant) with expertise in non-phosphorous chemistry (Stamford, Blakeley & Atequiza)
- Use Model compounds as templates
- Modify Functional Groups and Substituents
- Develop Structure-Activity Relationships
- Screen Reagents for ALL Technical and Commercial Drivers

After months of fundamental studies (microflotation, contact angle, and Cyclic voltammetry), and laboratory ore flotation testing using a variety of experimental designs and using the FLOTATION MATRIX® 100 approach, several new chemistries were then evaluated systematically on a variety of ores using multivariate designs to identify performance attributes and to test robustness. The short list of collectors was field testing under multivariate testing (varying granulometry of ground ore, collector dose, collector addition point, pH, etc) using experimental designs. This was imperative to the success of the project. One product, AERO MAXGOLD® 900 Promoter was selected as the final commercially available product.

AERO MAXGOLD 900, is manufactured through a proprietary two step process, which is classified as a Trade Secret.

**Industrial Product Testing**

After laboratory testing, the product entered the plant testing phase. The main problem with testing precious metal collectors is that there is a high degree of variability in the feed grade, ore type, and the process itself. Below is data from a customer’s processing plant that treats ore containing between 0.3ppm to 0.6ppm gold. This plot represents gold in the tails (waste stream) over a 4 month period.

The statistical mean for this period is 0.0073 opt (ounces per short ton) or 0.25 ppm. The MAXGOLD product was used at 10ppm in the process along with an auxiliary collector Aero MX6205 Promoter. Assuming a consistent gold head grade, a decrease of 0.002 opt (0.03-0.06 ppm) in the tails is economically significant as the plant processes 11,000 short tons per day. A 0.002 opt increase translates out to USD$8.6-9.2 million/year extra revenue at today’s gold price (USD1190/ounce).
Industrial Trial Results

The mean for the flotation tails using the plant standard, potassium amyl xanthate is 0.0073 opt. When Cytec AERO® MX6205 was used on its own for one week, the following results were obtained:

<table>
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<th>Transfer Function Analysis</th>
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<td>Time Series Tails, opt</td>
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<td>Mean</td>
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<tr>
<td>Std</td>
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<td>N</td>
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<tr>
<td>Zero Mean ADF</td>
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<td>Single Mean ADF</td>
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<td>Trend ADF</td>
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The mean for this run was 0.0073 opt, which is equivalent to xanthate. When 10ppm of MAXGOLD was added in conjunction with 30ppm of AERO MX6205, the following results were generated:

<table>
<thead>
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<th>Time Series Tails, opt Au</th>
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<tr>
<td>Mean</td>
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<td>Std</td>
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<td>Single Mean ADF</td>
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<td>Trend ADF</td>
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In all of the trial dates, the mean for the tails decreased by 0.001 opt to 0.002 opt.

**Conclusions**

Field trials of MAXGOLD, and MAXGOLD containing products continue. In most cases, improvements in gold recovery have been demonstrated. We have also expanding the testing scope to field testing of platinum group metals, and nickel ores.
A Robust Chemically Enhanced Electrostatic Separation Technology

S.A. Ravishankar, H. Kolla and R. Raitani

Introduction

Heavy minerals operations predominantly use the physical separation processes exploiting the differences within gravity, electrostatic and magnetic properties for separation. While the perpetual challenges focus on selective and efficient separation technologies particularly for difficult ore bodies and finer particles sizes, the underpinning sustenance of these new developments depends mainly on their ability to conserve energy and minimize waste. Many incremental solutions have been shown to marginally improve recovery and yield, although it is clearly envisaged that a step change is needed to provide robustness to process, beneficiable hard to process ores, minimize carbon footprints and simplify process flow to result overall cost-benefit effectiveness.

Electrostatic Separation Technology

The electrostatic separation, a dry separation technique, is an integral part of heavy minerals separation as illustrated in many references (Dascalescu, 1998; Venkatraman, 1998; Kelly, 1989). It fits in the heavy minerals processing where mineral mixtures cannot be separated any further by either gravity or magnetic separations. It exploits the differences in the surface conductivities of two minerals and thus enabling conducting minerals such as rutile and ilmenite to be separated from the non-conductors such as zircon, quartz and monazite. A simplified flow diagram (Figure 1) of heavy minerals processing involves a series of pre-concentration steps using gravity and magnetic separations. Gravity separation is used to separate the heavy minerals such rutile, ilmenite, zircon and leucoxene from low density minerals such as quartz, clay etc. This is followed by the magnetic separation to remove all the strongly magnetic iron bearing magnetic minerals from Rutile and zircon which then subsequently dried, heated to 120-180°C range to be separated by electrostatic separator based on surface conductivity differences.

Figure 1
Simplified process flow diagram of heavy minerals separation

Separation Efficiency and Challenges

The electrostatic separation is an effective process; however, it is not very efficient. The reasons for poor separation has been explained by several studies as the sensitive response of the technology to the fluctuations in process conditions such as relative humidity and temperature of the feed (Venter, 2008; Dance, 1992; Lindley, 1997). Higher humidity typically reduces the separation efficiency owing to indiscriminate condensation of conducting water layer on the minerals resulting misreport of nonconductors on to the conductors.

One of the major issues related to electrostatic separation, besides being too sensitive to the process conditions, is the surface contaminations. The loss in separation efficiency due to misreporting of the minerals such as non-conducting zircon with titanium species coating to conducting fraction resulting in poor grade quality of final product.
The other types of contamination include the indiscriminate coating of clay or other species such as aluminum, titanium and iron, once again, resulting in poor separation. Any indiscriminate alumino-silicate coating is also, once again, responsible for poor separation. Normally, the efficiencies with electrostatic separation in the first pass averages about 70 percent and it results in substantial recirculation load. And, there is considerable advantage in reducing the recycle load. For example, in a 50 t/Hr feed rate plant, even a 1 percent improvement in efficiency would improve 3% production rate of the fresh feed.

Chemically Enhanced Electrostatic Separation Technology

Specific reagent design for improving in-process separation efficiencies has remained one of Cytec’s core strengths for several decades. The Chemical enhancement of electrostatic separation technology is developed by embracing reagent design attributes of selective functionalities to the mineral surface and ability to impart reagent chemistries to enhance the surface conductivity and/or non-conductivities of the targeted particles considered for separation. The robustness to endure the thermal cycle and the immediate response to induced surface charging are key attributes. The readily applicable nature to the existing electrostatic equipment allows minimal capital investment. The enhancer technology is applied under wet condition before drying the feed (as described in the experimental section). In short, the Chemical enhancement of electrostatic separation technology is a “bolt-on” technology for enhanced electrostatic separation using tailor-made functionalized reagents that selectively increases the conductivity or non-conductivity of the rutile or zircon particles, respectively.

Experimental

A bulk quantity of the feed (25 -30Kg) was passed through a riffle splitter to ensure a good representative feed sample. With continuous splitting procedure, the sample size was reduced to ~ 500g. Each of the 500 grams representative sample batches were separately packed and stored. In each test about 166.0g of water was added to an octagonal shaped tall tubular steel container and was mixed and conditioned using an impeller design that provided countercurrent slurry push. An appropriate amount of electrostatic enhancer reagents was added to this and homogenized for 1 minute. The feed (500 g) was then added to this mixture and conditioned at natural pH for 5-10 minutes. The treated slurry was transferred to a tray and the solution decanted. The tray was placed in an oven 140 °C for ~ 3 hours and the dried sample was mixed well to remove the agglomerates, if any. The sample was reheated to 140 °C by replacing it in the oven. Then the sample was quickly removed from the oven and transferred in to the feeder to electrostatic separator (model HTP (25)111-15). The electrostatic separator was operated at a roll speed of 260 RPM, an applied voltage of 23 kV and a feed rate of 50 Kg.hr/in. The humidity was noted and a control (without reagent) test was conducted with every set of experiments. The separated feed was captured in a continuous train of 18 trays where trays 1-9 were designated as conducting portion (C), trays 10-12 as middling-1 portion (M1), trays 13-15 as middling-2 portion (M2), trays 16&17 as middling-3 portion (M3) and tray 18 (NC) as the non-conducting portion. The weights in the above trays were recorded. XRF analysis was then performed on each group (conducting, middlings-1,2,3 and non-conducting portion). The mass recovery (weight of each portion) and grades (XRF analysis) were plotted to evaluate the efficiency curves.
The efficiency improvement is calculated from the cumulative recoveries of individual fractions such as C, M1—NC. Maximum Efficiency (ME) is highest value between cumulative efficiencies CE (C)...CE (M2)...CE (NC).

\[ E = \frac{(C_{RTi} + (100 - C_{RZr}))}{2} \]

As mentioned before, if the reagent improves the separation then the Maximum efficiency (ME) of the separation with the reagent will have to be higher than the control (no reagent) and the difference (DE) of 3 to 5% is considered significant in a laboratory operation.

**Results and Discussion**

**Chemical Enhancement at High Temperatures**

An extensive pilot plant optimization study was conducted using the chemical enhancer technology under plant operating conditions. The feed sample was obtained from a heavy minerals plant in South Africa. The separation conditions were 260 RPM on the roll speed, 26kV on the applied voltage, 50-60% relative humidity, and electrode distances were kept as per plant conditions. An improvement of 8-11% in the separation efficiency was observed after the first pass on the rutile and zircon separation. The performance efficiency improvements are represented by efficiency plots as shown in Figure 2. The increase in peak height is resulted from the increase in separation efficiency.

Another way of representing the improvement will be through cumulative grade-recovery plot. Figure 3 shows the cumulative mass yield vs cumulative recovery of both rutile and zircon portion for the conductive, middling 1-3, non conductive fractions. The outer envelope of solids red line indicates the ideal separation and red and diagonal solid line indicates no separation between rutile and zircon. The recovery-yield separation curves above the red diagonal (no separation) line denote the data for rutile and the ones below correspond to cumulative recovery of zircon. Closer the recovery-yield separation curves to the ideal separation, more efficient the separation is between rutile and zircon minerals. Figure 3 shows that the chemically enhancement exhibits better separation (solid green lines) than the control (dotted red line) without reagents.

Figure 2
Representative efficiency improvement plot
Further, the reagent adsorption may be mitigating the finicky nature of electrostatic separation owing to changes in process parameters such as feed temperature, humidity, applied voltage and allows the process to be more forgiving to mineralogy. An understanding of the mechanism will help to expand this technology into other minerals systems such as: a) Ilmenite / Staurolite, b) Ilmenite / Monazite, c) Zircon / Leucoxene, d) Iron ore – silicate removal, e) Hard rock ilmenite or rutile, f) metal-plastics recycling, and g) kyanite/zircon.

Electrostatic Separation at Room Temperature

It is well known that for effective separation by high tension or electrostatic separation, substantial energy is spent at the feed preparation stage for drying the feed from ambient wet condition to above 120 °C. Also, during the separation stage, the feed is kept at a high temperature in the range of 120-180 °C to avoid loss in separation. The hot temperature enables to maintain low humidity in the vicinity of the roll which is a major factor in the separation as mentioned before. Therefore, substantial energy savings could be conceived if the separation could be achieved at ambient temperature and at high humidity conditions. The chemical enhancement technology is capable of separation at room temperature and at high humidity conditions after drying. Figure 6 and 2 compared the separation at room temperature and high temperature. At room temperature, there was no separation (Figure 6, dotted line) in the absence of chemical enhancement, however, with our chemical enhancement technology there is substantial the improvement (Figure 6, solid line) which was not only better than the control at high temperature (Figure 2, dotted line) but also as good as the chemical enhancement technology at high temperature (Figure 2, solid line).

Effect of Reagent Dosage

The chemical enhancement using AERO® EZ-2100/AERO EZ-1000 reagents appears to increase linearly with respect to dosage within 0-1Kg/T level range. Figure 4 shows the efficiency plots at two different dosage levels. Even at lower dosages of 0.5 Kg/T level, the separation efficiency improvement is significantly better than the control. It is anticipated that operating conditions such as humidity, temperature, applied voltage, polarity and most importantly the mineralogy will have a bearing on the dosage level. The efficiency plots are also represented as cumulative recovery and Yield plots for comparison. The higher dosages provide better separation as shown in Figure 5.

The elucidation of the mechanism of interaction of these reagents to improve the efficiency of separation is ongoing, however, it can be surmised that the enhancer reagent may specifically adsorb onto or selectively desorb species from the surfaces of rutile and zircon to render them more conductive and non-conductive, respectively.
A Robust Chemically Enhanced Electrostatic Separation Technology
continued

Figure 4
Efficiency plot illustrating improvements in rutile-zircon separation with AERO EZ-2100/AERO EZ-1000.

Figure 5
Cumulative Recovery-yield plot showing dosage dependence on separation: both the dosages are significantly better than the control without chemicals.

Figure 6
Separation enhancement due to chemical treatment from no separation (dotted line) to 75% efficiency (solid line) at room temperature.
Conclusions

Chemical enhancement of electrostatic separation can be achieved by judicious combination of surface specific reagents that selectively modify the surface conductivities for improved separation. An improvement of 8-11% in separation efficiency has been achieved at high temperature (140 °C). But, most importantly, the separation can be effected at room temperature between rutile and zircon. We believe that the chemical enhancement to electrostatic separation can provide the many benefits:

1. conduct electrostatic separation at room temperatures
2. improve the separation efficiency at high temperatures
3. reduce the recirculation load
4. translate into energy savings by the elimination of a certain number of cleaning stages
5. a “bolt-on” technology to the existing electrostatic separation technology with minimal capital investment
6. provide robust performance mitigating adverse process variations in terms of temperature and humidity conditions

References

## Upcoming Tradeshows

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<th>Location</th>
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<td>January 18 - 20, 2011</td>
<td>Ottawa, Canada</td>
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<tr>
<td>SME Annual Meeting and Exhibit 2011</td>
<td>February 27 - March 2, 2011</td>
<td>Colorado Convention Center Denver, CO</td>
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<tr>
<td>Expomin</td>
<td>April 2011</td>
<td>Santiago, Chile</td>
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<td>ALTA 2011 Nickel - Cobalt - Copper, Uranium &amp; Gold Conference</td>
<td>May 23 - 28, 2011</td>
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