



*Presented at Hydroprocess 2008, Tucson, Arizona, 14-16 May 2008*

## **Laboratory and Demonstration-Scale Operation of the Caraiba Heap Leach using GEOCOAT<sup>®</sup>**

Michael Gunn, Pamela Tittes, Paul Harvey and Enrique Carretero, *GeoBiotics, LLC, USA*  
Pablo Medeiros da Silva and Jair Paula de Souza, *Mineração Caraiba, S.A. BRAZIL*

### **ABSTRACT**

Located in Jaguarari, Bahia, Mineração Caraíba S.A. (Caraíba) was, until recently, the only producer of copper concentrate in Brazil. Caraíba is currently heap leaching its oxide ores, and is shipping its sulphide concentrates to a smelter approximately 400km from its mine. Caraiba has unused SX/EW capacity in its oxide circuit, and would like to treat the sulphide concentrates on-site to take advantage of this unused capacity and to avoid high shipping charges. Caraiba would also like to use the additional acid produced from filling its SX/EW circuit for its oxide leach operation. With these goals in mind, GeoBiotics' GEOCOAT<sup>®</sup> technology is being evaluated for application at the Caraiba property to biooxidize the primary and secondary sulphide concentrates on an engineered, re-usable heap leach pad, allowing for the successful leaching of the contained copper. Initially GEOCOAT<sup>®</sup> was evaluated in the laboratory through batch amenability testing followed by column testing. Based on the results of the laboratory tests, a 1600 tonne, 50m x 40m, GEOCOAT<sup>®</sup> demonstration heap has been built at Caraiba. The objective of the demonstration heap test is to define how closely the full-scale heap emulates the laboratory test performance. The challenge is to conduct the demonstration test within the material preparation, handling and pad systems in use at the Caraiba operation, and to achieve high operating temperatures for the autothermal leaching of the primary copper sulphides. Because of heat losses through the heap side walls, autothermal operation is not possible with column tests performed in the laboratory. This paper briefly describes the Caraiba GEOCOAT<sup>®</sup> demonstration heap and discusses the laboratory test results.

The GEOCOAT<sup>®</sup> testwork programs conducted on the Caraiba ore, rougher and final concentrate indicate that high copper extractions can be achieved from these materials. The results of batch amenability stirred tank bio-leaching tests (BAT) showed significantly more Cu was extracted by bio-leaching compared to the baseline copper extraction determined by diagnostic leaching. In the GEOCOAT<sup>®</sup> column tests, when using the low grade ore support rock, Cu extraction was 65% for final concentrate at 30°C. At 70° Cu extraction was 96% from final concentrate. The two methods for calculating Cu extraction, based on solid residue and based on silica analysis, are averaged, and show 90% copper extraction is achieved at 61°C.

GeoBiotics, LLC  
12345 West Alameda Parkway, Suite 310  
Lakewood, Colorado 80228 USA  
1-303-277-0300  
1-303-277-1772 (fax)



The results of these tests were very encouraging. The ability to reproduce these results at the ambient temperatures prevailing on site will only be confirmed by full scale trial in the demonstration heap.

## **BACKGROUND**

The Caraiba operation of Mineraçao Caraiba S/A is located approximately 135 km south east of Petrolina/Juazeiro in Bahia province of northern Brazil. The mine is accessed from Petrolina along routes BR407 and then BA314. The mine workforce is largely accommodated in the township of Pilar, approximately 14km west of the minesite. The copper operation was originally constructed by Governo Brasileiro in the 1970's, was privatized in 1994, and is robust, well designed, and operated by a competent and professional staff and workforce to world class standards.

The concentrator currently mills 3.3 million tonnes per annum ore grading ~0.8% Cu from a mix of open pit and underground mining, and produces approximately 70,000 tonnes per year of ~32% Cu concentrate. All ore is anticipated to be from underground sources by 2012 with head grade then averaging 2.2% Cu. The mineralization is predominantly a coarse grained mix of bornite and chalcopyrite, with copper distributed in an approximately 1:3 mass ratio between the two minerals respectively. A minor amount of copper occurs in cuprite, chalcocite, and covellite and in chloritic material.

The Caraiba operation also operates a copper oxide ore acid heap leach that, until recently, was able to run economically on low cost acid. The solvent extraction/electrowinning plant is rated at around 5000 tonnes per annum (tpa) copper cathode expandable to 7000 tpa.

GeoBiotics, LLC (GeoBiotics) is a U.S. based, minerals biotechnology company which has developed and owns a number of proprietary technologies and processes for the biooxidation and bioleaching in heaps of sulphide ores, concentrates and tailings, together with patents, patent applications and trademarks evidencing such technologies and processes, and trade secrets and know-how related to such technologies and processes. GeoBiotics has developed, patented and owns the GEOCOAT<sup>®</sup> technology which facilitate the recovery of gold and base metals from sulphide refractory ores and concentrates by bacterial oxidation in an engineered heap. GeoBiotics owns a license to use, with the right to sublicense to third parties, the BIOPRO<sup>™</sup> technology which is a proprietary heap bio-oxidation technology for pre-inoculation of heaps with bacteria. GeoBiotics has acquired the HotHeap<sup>™</sup> technology, which is a proprietary heap bio-leaching technology that incorporates a control and operations strategy designed to maximize heat conservation within heap bio-leaching environments through the control of irrigation and aeration rates.

Caraiba, GeoBiotics and the Centre for Mineral Technology (CETEM) have worked closely together to test the use of GeoBiotics' GEOCOAT<sup>®</sup> biooxidation technology for the treatment of Caraiba copper concentrates. Laboratory testwork has demonstrated that this process can provide both acid and supplementary copper to the hydrometallurgical operation. Potential also exists for

increasing overall concentrator recovery of copper, since the GEOCOAT<sup>®</sup> biooxidation process allows for the treatment of lower grade concentrates in the heap.

To confirm the results of the laboratory test program, Caraiba has committed considerable resources and time to the construction of a demonstration heap facility. The testwork reported in this paper was largely the basis for the design of this facility, which is shown under construction in Figure 1.



**Figure 1: Caraiba Demonstration-Scale GEOCOAT<sup>®</sup> Biooxidation Plant  
(Under construction – September 2008)**

The demonstration heap has been designed so that the pregnant leach solution (PLS) from the bioleaching of concentrates is bled back into the existing Caraiba process as required. A total of approximately 1600 tonne of concentrate and 14,400 tonnes of crushed support rock were required to construct the 50m x 40m, GEOCOAT<sup>®</sup> demonstration heap at Caraiba. The size of the heap has been selected on the basis of heat generation and retention, as determined by computer modeling using GeoBiotics's proprietary HotHeap<sup>®</sup> modeling software. Performance of the GEOCOAT<sup>®</sup> demonstration heap is monitored by sampling solutions and solids and by measuring heap temperatures throughout the test.

### **General Testwork Program Sequence**

GeoBiotics (GBL) has developed laboratory test procedures to simulate heap conditions in a full-scale GEOCOAT<sup>®</sup> heap.

Scoping levels tests are initially carried out in stirred reactors to:

- define basic amenability of the material to biooxidation,
- define which bacterial regime gives the best kinetics and greatest extent of oxidation,



- determine whether the mineral suite contains any toxic species harmful to bacteria that may require more elaborate adaptation procedures, and
- define the degree of oxidation required to achieve target levels of metal extraction.

Scoping level tests consist of batch amenability testing in stirred, temperature-controlled aerated reactors. These tests define the balance between copper extraction and the degree of oxidation on concentrates or pulverized ore samples.

The next stage of testing, in 2m kinetic columns, maintains test conditions at optimum temperatures for bacterial activity, and usually continuously irrigates the column with acidic solution in open circuit. The kinetic columns define the oxidation response at three increasing temperature regimes, with ore at a range of crush sizes, using the GEOCOAT<sup>®</sup> process with concentrate coated onto support rock.

Depending on the success of the scoping levels tests, detailed tests are then conducted in 6m high columns with larger sample mass. The height of the column is selected to match the proposed height of a full-scale heap. The full height columns are operated with controlled temperature and with time ramp-up that is defined by the modeling of the kinetic response profiles to simulate the temperature anticipated at full scale operation. All of the column tests are carried out in the bacterial regime that is identified as being most effective, and at all of the temperature/bacterial regimes the ore will need to move through to achieve the optimum operating conditions. The final phase of testing provided is a large scale field demonstration that is typically a minimum of a 40m x 40m heap footprint.

## **Caraiba Testwork Program**

Caraiba had several general objectives in undertaking the biooxidation testwork, followed by the demonstration scale GEOCOAT<sup>®</sup> heap:

- To quantify the amount of acid that can be generated from this process for use in its existing acid consuming oxide copper heap leach.
- To establish that the bornite-chalcopyrite dominant concentrate produced from its conventional concentrator leaches efficiently in a thermophile biooxidation regime to produce copper sulphate solutions.

The successful bio-leaching of copper from concentrates will potentially enable Caraiba to:

- maintain SX-EW cathode production currently supplied from the existing oxide ore acid leach as the oxide ore supply reduces,
- and/or allow expansion of Caraiba's cathode copper production or diversification into copper sulphate production,
- and reduce the cost of acid for oxide heap leaching by using the substantial acid credit provided by the biooxidation reactions.



The materials under investigation were:

- Caraiba ore (Surubim ore)
- Caraiba rougher concentrate
- Caraiba final concentrate

Testwork was conducted at the Little Bear Laboratories in Denver, Colorado (now GeoSynFuels, LLC).

### ***Testwork Results***

The outcome of all work conducted until now is summarized as:

- The chalcopyrite and bornite copper sulphides leach to high copper extraction levels in the thermophile oxidation leach. This has been proven in stirred tank and 2m column level testing, and is strongly indicated in preliminary large scale column tests.
- Using Minerio Marginal (MM) waste rock as the support rock in the GEOCOAT<sup>®</sup> process, the value of the acid consumption by the support rock is similar to the value of the amount of copper released from the support rock during the leach cycle.
- The second cycle of biooxidation testing, using the same support rock and fresh concentrate, indicates very low acid consumptions by the support rock.
- An increased amount of acid can be generated by using the relatively inert quartz available at Caraiba as support rock.
- The gross acid consumption of the concentrate in the small scale testwork is strongly supported by theoretical calculation of gross acid consumption from mineralogical information.
- Any un-reacted copper sulphides and particulate gold in the biooxidation residues can be efficiently recovered to a concentrate by re-flotation. This reduces any potential metal loss to a very low level.
- The biooxidation process, even using the acid consuming MM support rock, generates a net surplus of acid of ~+193 kg/t of concentrate treated.

The results of these tests were very encouraging. However, the ability to reproduce these results at the ambient temperatures prevailing on site can only be confirmed by full scale trial. The results for whole ore testing are not reported in detail in this paper, but for whole ore the response was good. The acid consumptions were high and the copper extractions around 70%. This result did not fit the overall process model required by Caraiba, hence further whole ore testing was not carried out.



## Sample Characterization

### Chemical Analysis

Pulverized samples of Surubim ore and samples of blended rougher concentrate and final flotation concentrate were analyzed by Hazen Research as shown in Table 1.

**Table 1: Composition of Caraiba Rougher and Final Flotation Concentrates**

Material	As mg/kg	CO <sub>3</sub> %	Cu %	Gold mg/kg	Silver mg/kg	Iron %	Si %	S <sub>T</sub> %	S <sup>o</sup> %	SO <sub>4</sub> %	S <sup>2-</sup> -S <sup>1</sup> %
Ore	2.97	0.47	0.888	<0.17	40.8	9.32	24.5	0.61	<0.1	0.02	0.61
Rougher	1.24	0.42	7.99	0.72	19.89	12.6	21.6	5.40	<0.1	0.03	5.39
Final	1.43	0.42	32.2	1.71	13.03	17.9	6.20	22.0	<0.1	1.68	21.4

<sup>1</sup>by calculation: sulphide-s = (total s) – (elemental s) – (sulphate-s)

### Diagnostic Leach

Diagnostic leaching was done on pulverized ore and concentrates following a procedure supplied by GeoBiotics. Copper soluble in 5% acetic acid (heated to 70°C for 15 min), 5% sulfuric acid (shaken for 1 h), and in 5% NaCN (residue from sulfuric acid extraction, shaken for 30 min) was determined as shown in Table 2. Slightly over one half of the copper in the ore and in the rougher concentrate (51% and 59%, respectively) was extracted by sequential leaching with 5% sulfuric acid and 5% sodium cyanide reagents. Slightly over one third of the copper in the final concentrate (35%) was leached by these reagents. Table 2 also indicates the species leaching in the sequential leach regimes.

**Table 2: Diagnostic Leaching of Copper<sup>1</sup>**

Material	Acetic acid soluble, %	H <sub>2</sub> SO <sub>4</sub> soluble, %	NaCN soluble, %	Total Cu, %	Baseline
Ore	0.008 (0.9%)	0.054 (6.1%)	0.400 (45.1%)	0.888	51.2%
Rougher	0.098 (1.2%)	0.261 (3.3%)	4.483 (56.1%)	7.99	59.4%
Final	2.401 (7.5%)	3.018 (9.4%)	8.324 (25.9%)	32.2	35.3%
Minerals leached	Malachite and azurite	Malachite, azurite, tenorite, chrysocolla and 50% of cuprite	Chalcocite, bornite, approximately 8% of chalcopyrite	All copper in ore	

<sup>1</sup>All results are expressed as %Cu in ore as determined by the various extraction techniques, the total Cu determination being the most complete extraction. Values in parentheses are the % of copper in the ore occurring as the given minerals.



### ***Acid Consumption***

Pulverized ore (250 g) was added to 500 cm<sup>3</sup> deionized water and shaken for 5 min. The natural pH was 8.86. The slurry was brought to pH 1.50 with sulfuric acid. The slurry was agitated for 7 days. Each day the pH was measured and sulfuric acid added to return the pH to 1.5. After 7 days a total of 17.9 g H<sub>2</sub>SO<sub>4</sub> had been added corresponding to 71.6 kg/t.

The copper mass balance from the acid consumption test showed 0.415 g in the solution phase (by ICP at Little Bear Labs) and 1.550 g in the leached residue (241 g dry ore recovered containing 0.643% Cu). The copper content of the starting material was 2.20 g (250 g ore at 0.888% Cu), giving a mass balance of 89.3% (recovered/starting).

### **Batch Amenability Tests**

Batch Amenability Tests (BATs) were conducted to determine maximum copper extraction expected from bioleaching of ores and concentrates with mesophilic, moderately thermophilic and extremely thermophilic iron- and sulfur-oxidizing microorganisms.

Mixed cultures of mesophilic iron and sulfur oxidizing microorganisms were adapted to pulverized Surubim ore and to rougher and final flotation concentrates by growing the organisms in shake flasks on a mineral salts medium amended with Surubim ore or concentrate, pyrite, sulfur and ferrous sulphate. The cultures grew readily in these slurries and were used to inoculate stirred reactors for BAT tests.

Tests were performed in 20 L plastic reactors filled with 10 L of nutrient solution and 500 g of pulverized Surubim ore (minus 150 mesh), rougher concentrate or final concentrate. These materials were inoculated with iron- and sulfur-oxidizing microorganisms adapted to these substrates. Reactors were stirred and aerated at 3.0 L/min and the solutions were monitored for pH, redox potential and dissolved iron and copper.

### ***Mesophiles***

BATs with mesophilic microorganisms were operated at 31°C to 32°C. Mesophiles grew readily on the Caraiba samples; and solution redox potentials were highly oxidizing in all the mesophile BAT tests, exceeding 900 mV (she) from day 1 with ore, and after 3 to 4 days with the concentrates. Copper extractions appeared to be complete after 11 days. Copper extractions and net acid consumptions from Surubim ore, rougher concentrate and final flotation concentrate are shown in Table 3. Copper extractions (determined from residue analyses) ranged from 65% with final concentrate to 72% with Surubim ore to 76% with rougher concentrate.



**Table 3: Summary BAT results with mesophilic microorganisms (31°C to 32°C)**

Material	Test period	%Cu head	Actual % Cu extn <sup>1</sup>	%Sulphide- S content	%Sulphide-S oxidation	Net acid consumption kg/t <sup>2</sup>
Ore	11 d	0.888	71.8	0.61	47.3	131
Rougher	11 d	7.99	75.8	5.39	58.0	144
Final	11 d	32.2	65.0	21.4	43.9	250

<sup>1</sup>bioleached residue versus head analysis

<sup>2</sup>acid added minus free acid in final solution

### ***Moderate Thermophiles***

BATs with moderately thermophilic iron- and sulfur-oxidizing microorganisms were conducted at 48°C to 49°C. Moderate thermophiles also readily leached the Cu from Caraiba samples. Solution redox potentials increased to 847 and to 834 mV after 2 days with ore and rougher concentrate respectively, and stabilized there. Redox potentials with final concentrate were 770 mV after 2 days and slowly increased to 813 mV after 7 days. These high potentials are indicative of vigorous bio-oxidation. Copper extraction after 7 days appeared complete. Residue analyses results in Table 4 shows Cu extractions of 74% with ore, 80% with rougher concentrate and 90% with final concentrate.

**Table 4: Summary BAT results with moderate thermophilic microorganisms (48°C to 49°C)**

Material	Test period	%Cu head	Actual % Cu extn <sup>1</sup>	%Sulphide- S content	%Sulphide-S oxidation	Net acid consumption kg/t <sup>2</sup>
Ore	7 d	0.888	74.4	0.61	64.5	150
Rougher	7 d	7.99	80.1	5.39	62.6	163
Final	7 d	32.2	89.7	21.4	82.1	254

<sup>1</sup>bioleached residue versus head analysis

<sup>2</sup>acid added minus free acid in final solution

### ***Extreme Thermophiles***

Extreme thermophile BAT tests were performed at a temperature of 67°C to 68°C. Redox potentials with ore increased to 834 mV after 2 days and stabilized. Potentials reached 818 mV after 4 days with rougher concentrate and also were relatively stable. Copper extraction from these substrates appeared to be complete after 8 to 9 days and ranged from 85% to 90% by solids analysis as shown in Table 5.





**Table 5: Summary BAT results with extreme thermophilic microorganisms (67°C to 68°C)**

Material	Test period	%Cu head	Actual % Cu extrn <sup>1</sup>	%Sulphide- S content	%Sulphide-S oxidation	Net acid consumption kg/t <sup>2</sup>
Ore	8 d	0.888	88.5	0.61	69.4	226
Rougher	9 d	7.99	89.7	5.39	82.1	193
Final	6.25 d	32.2	85.1	21.4	77.8	212

<sup>1</sup>bioleached residue versus head analysis

<sup>2</sup>acid added minus free acid in final solution

### **BAT Results Summary**

The results of BAT tests showed significantly more copper was extracted by bio-leaching compared to the baseline Cu extraction determined by diagnostic leaching. Although bio-leaching of copper generally increased with increasing temperature, sulphide oxidation and bio-leaching of copper from final concentrate at 48°C (after 7 days) was slightly higher than at 67°C (after 6.25 days).

It is worthy to note that the batch test is considerably more difficult to run than are column tests with respect to realistically simulating a full scale heap. This is due to the closed nature of the batch test and the accumulation of product species at high concentrations. In a column test the system bleed allows the control of the concentrations of the various metals leached.

### **GEOCOAT<sup>®</sup> Kinetic Column Tests**

The batch amenability tests were followed by a program of kinetic column tests at 2 m scale. The purpose of this testing is to determine preliminary leaching kinetics for mass balance calculations, preliminary process design and heap sizing. The columns tests were in most cases run for 96 days. GEOLEACH™ column tests were not conducted on the Surubim ore as it was agreed the project could not support the 131 to 226 kg acid / t ore consumption. GEOCOAT<sup>®</sup> column tests were conducted on the rougher concentrate and final concentrate.

The tests were run under standardized conditions, based on GeoBiotics' experience with numerous ores from a wide variety of sources. These conditions usually include:

- Mixture of concentrates with acid and biological culture
- Use of bacteria adapted to the concentrate to be biooxidized
- Simulated starting solution – ferric and metals concentrations, pH, free acid
- Temperatures ranging between 35 °C – 67 °C to determine the effect on kinetics of metal extraction



Although the results of these kinetic tests may not represent the optimum GEOCOAT<sup>®</sup> conditions, they do provide enough data to adequately size the heaps, and the results can be useful as a basis for a scoping level evaluation of process economics.

**Column Tests**

A total of ten GEOCOAT<sup>®</sup> column tests with rougher concentrate and final concentrate were conducted in small columns (10 cm x 0.6 m). An initial group of 6 columns were tested at ca. 30°, 50° and 70° with MM as support rock (+5, -16 mm) and coated with pre-leached concentrate. Following these tests a second set of 4 columns tests was operated similarly at ca. 40°C and 60°C. Column solutions (10 L total volume) were monitored for pH, oxidation/reduction potential, dissolved iron and copper, ferrous iron and free acid. Column tests operated for 95 to 96 days.

**Operation**

An initial leach solution (at pH 1.1 containing microbial nutrients and 2.0 g/L Fe<sup>3+</sup>) was applied at 16 L/h/m<sup>2</sup> until acid consumption was brought under control. Thereafter the solution application rate was decreased to approximately 6 L/h/m<sup>2</sup> with recirculated PLS solution. Columns were aerated from the bottom at 0.5 L/min.

Leach solutions in all 10 column tests within a few weeks became highly oxidizing (high Eh) and there was little or no detectable ferrous iron in solution. This indicated good microbial growth and iron oxidation in all columns. Copper concentrations in leach solutions increased to between 2 g/L to 3 g/L in columns containing rougher concentrate and to between 7 g/L to 11 g/L with final concentrate. Eh curves indicating the highly oxidizing conditions and high bacterial activity during the operation of the rougher concentrate columns are shown in Figure 2.

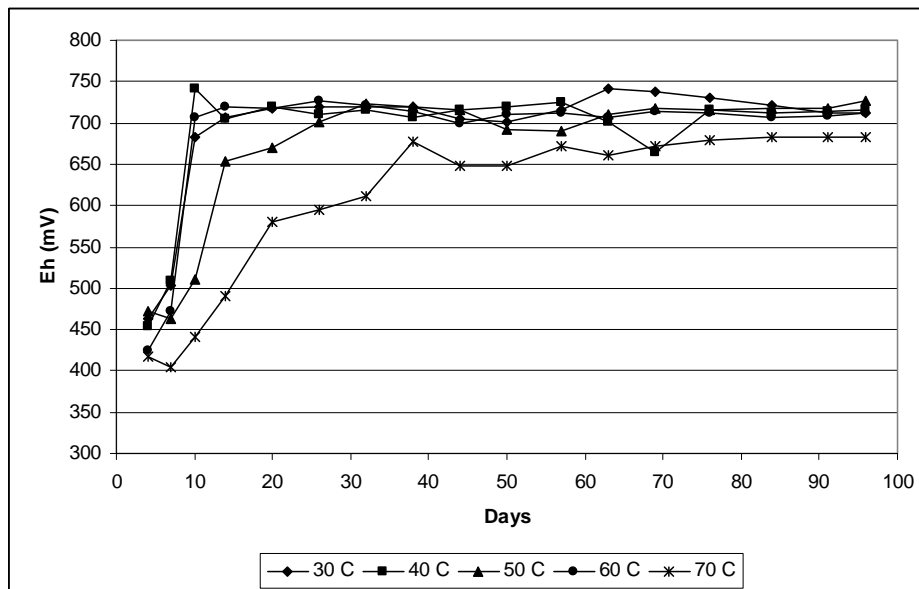


Figure 2: Eh curves during the operation of the columns



**Results**

The rate of copper extraction slowed after 3 months and tests were concluded. Support rock and remaining concentrate residues were recovered, rinsed with water and dried. The solid residue analyses from the 30°C, 40°C, 50°C, 60°C and 70°C GEOCOAT® Column Test are presented in Tables 6 and 7. Sulphide oxidation and copper extraction increased at higher temperatures. This was expected based on:

- diagnostic leaching of the concentrates which showed a significant portion of copper as “refractory” (Table 2) and,
- batch amenability tests (BATs) which showed increasing copper extraction with increasing temperature of bioleaching (Tables 3, 4, 5).

**Table 6: Analyses of ferric pre-leached Caraiba rougher concentrate before and after biooxidation in GEOCOAT® columns for 95-96 days at various temperatures**

	Au g/t	Ag g/t	As g/t	Fe %	Cu %	S %	S <sup>-</sup> %	S <sup>o</sup> %	SO <sub>4</sub> %	Si %	%Cu extn <sup>1</sup>	% S <sup>-</sup> oxidn <sup>1</sup>
<b>Head-1</b>	0.96	13.70	4.60	13.3	6.10	5.33	5.16	<0.1	0.51	24.6	--	--
<b>Head-2</b>	0.72	3.43	1.58	12.3	6.02	5.26	5.12	<0.1	0.43	24.2	--	--
<b>30°C</b>	0.55	25.71	5.31	12.6	1.58	2.98	1.90	0.2	2.63	22.5	75.5	65.4
<b>39°C</b>	0.69	69.9	1.91	14.5	1.26	3.47	1.71	<0.1	5.27	28.6	78.5	65.7
<b>49°C</b>	0.55	24.34	3.38	13.6	0.888	3.30	0.89	0.6	5.43	20.8	85.7	83.2
<b>60°C</b>	0.34	29.49	1.08	15.3	0.460	3.10	0.41	<0.1	8.07	23.2	90.8	90.3
<b>68°C</b>	0.48	16.46	1.67	13.6	0.416	3.26	0.40	0.2	7.99	20.1	90.9	89.8

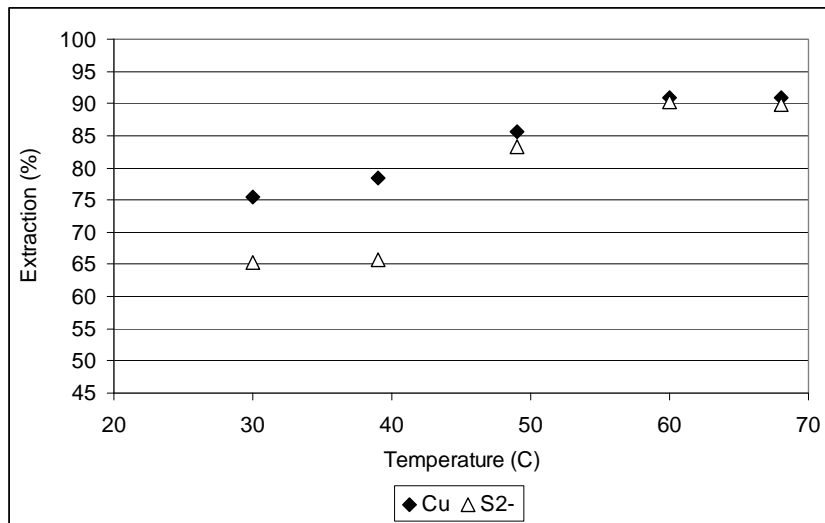


**Table 7: Analyses of ferric pre-leached Caraiba final concentrate before and after biooxidation in GEOCOAT® columns for 95-96 days at various temperatures**

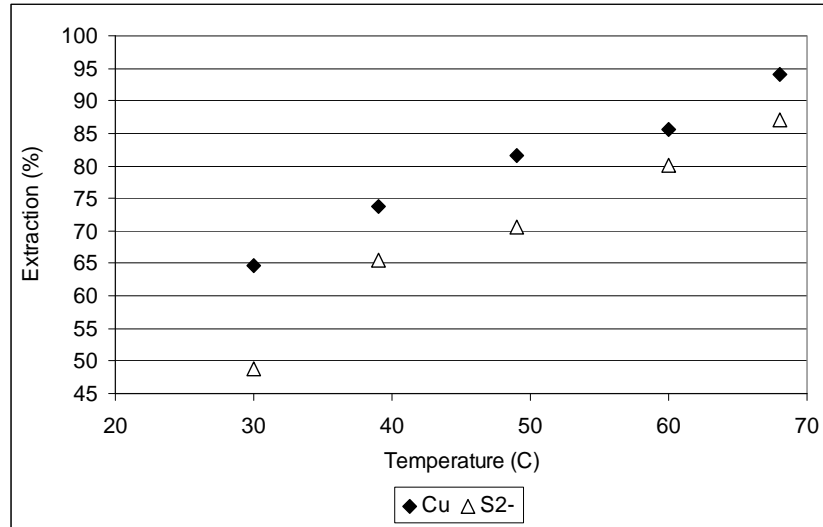
	Au g/t	Ag g/t	As g/t	Fe %	Cu %	S %	S <sup>=</sup> %	S <sup>o</sup> %	SO <sub>4</sub> %	Si %	%Cu extn <sup>1</sup>	% S <sup>=</sup> oxidn <sup>1</sup>
Head-1	2.54	34.30	14.2	19.8	29.4	23.1	22.6	<0.1	1.62	7.00	--	--
Head-2	1.51	23.31	2.71	20.3	27.0	23.0	21.9	0.2	2.71	6.42	--	--
31°C	1.99	56.23	2.54	19.8	11.8	16.6	13.1	2.0	4.51	11.3	64.6	48.9
39°C	1.37	65.14	1.32	19.2	6.58	12.8	6.98	3.5	6.95	14.9	73.7	65.6
49°C	1.17	37.71	3.06	16.6	4.27	11.1	5.21	3.7	6.58	16.7	81.5	70.7
61°C	1.58	41.49	1.62	18.5	3.65	10.5	4.09	3.4	9.03	16.1	85.6	80.1
69°C	0.75	19.89	1.40	14.8	1.00	4.78	1.66	0.8	6.97	18.8	94.1	87.2

<sup>1</sup>% extraction/oxidation based on solids analyses. "Head-1" are the analyses associated with the first set of tests (30°C, 50°C and 70°C) and "Head-2" data corresponds to the second set of tests (40°C and 60°C).

Summary copper extraction and sulphide oxidation after 95 to 96 days at the various column operating temperatures are shown in Figure 3 (rougher concentrate) and Figure 4 (final concentrate).



**Figure 3: Copper extraction and sulphide oxidation (based on analysis of solids) from Caraiba rougher concentrate--5 kg column tests operated at various temperatures for 95-96 days**



**Figure 4: Copper extraction and sulphide oxidation (based on analysis of solids) from Caraiba final concentrate--5 kg column tests operated at various temperatures for 95-96 days**

The recovered concentrate appears to have been diluted with fines from MM support rock decomposition as the gold content of biooxidized material was lower than the un-oxidized head sample, with one exception (60°C final concentrate). Gold dilution was higher at higher temperatures, 60°C final concentrate excepted. Masses of recovered concentrate residue exceeded the initial concentrate mass with all other tests.

MM support rock had some effect on column test results. Unlike inert support rock it:

- contained copper,
- consumed acid and,
- decomposed into smaller particles, especially at elevated temperatures.

Although MM support rock contains only a small concentration of copper (0.295%) it was agglomerated at a 11:1 ratio with concentrate. Consequently, MM support rock contributed a significant mass of copper to columns, ranging from 11% of the total copper in columns containing final concentrate to 37% of the mass of copper in columns containing rougher concentrate.

Analysis of the recovered MM support rock showed mass losses ranging from 81 g to 453 g, amounting to 1.62% to 9.06% of the 5000 g starting mass. Copper extraction from the support rock ranged from 0% to 31.7%, based on solids analyses.



## Large Column Testing at CETEM

CETEM is the Centre for Mineral Technology, under the Ministry of Science and Technology of Brazil. The Metallurgical and Biotechnological Processes Division in Rio de Janeiro was engaged by Mineracao Caraiba S/A to act as a third party laboratory for the large column testwork phase of the project.

The testwork currently underway at CETEM was undertaken in two phases, with the first phase returning results somewhat inconsistent with the earlier, smaller scale Little Bear testwork. This is in part attributed to the development of new equipment in the laboratory and technology transfer issues. The second phase of testing has been initiated using recycled support rock and is proceeding well. Acid consumptions are low and the kinetics of copper extraction are rapid.

## Residue Flotation Test

The biooxidation testwork was complicated by the breakdown of support rock, which makes the calculation of the biooxidation residue mass loss very difficult. This in turn creates some doubt as to the exact mass of copper remaining in the biooxidation residue – calculated to be around 6% of the copper in feed. This small percentage of the copper, along with and all of the gold associated with the copper minerals, will remain in the biooxidation residue, and is of value.

The flotation testwork on biooxidation residue shows that a concentrate of 19 – 23 %Cu grade, containing 99% of the biooxidation residue copper and 94% of the residue gold, can be produced as shown in Table 8. This would appear to be suitable for blending back into concentrate shipments to the smelter with no significant commercial disadvantage.

**Table 8. Flotation Test Balance for Biooxidation Residue**

Product	wt%	Cum wt%	%Cu	Units	Cum Units	Recovery %	Cum Recovery %	Cum Cu%
Head Assay			5.40					
Head Calculated	100.01		5.55	554.8				
Concentrate A	22.87	22.87	22.68	518.7	519	93.49%	93.5%	22.68
Concentrate B	6.35	29.22	4.54	28.8	548	5.20%	98.7%	18.74
Concentrate C	6.25	35.47	0.44	2.8	550	0.50%	99.2%	15.51
Tail	64.54	100.01	0.07	4.5	555	0.81%	100.0%	5.547



Product	wt%	Cum wt%	Au g/t	Units	Cum Units	Recovery %	Cum Recovery %	Cum Au g/t
Head Assay			1.25					
Head Calculated	100.01		1.54	154.1				
Concentrate A	22.87	22.87	5.6	128.1	128	83.13%	83.1%	5.60
Concentrate B	6.35	29.22	2.6	16.5	145	10.72%	93.9%	4.95
Concentrate C	6.25	35.47	0.38	2.4	147	1.54%	95.4%	4.14
Tail	64.54	100.01	0.11	7.1	154	4.61%	100.0%	1.540

## CONCLUSIONS

The GEOCOAT<sup>®</sup> testwork programs conducted on the Caraiba ore, rougher and final concentrate indicate that high copper extractions can be achieved from these materials. Baseline copper extraction achieved from Caraiba final concentrate by diagnostic chemical leaching (sulfuric acid- and NaCN-soluble Cu) was 35%. The results of batch amenability stirred tank bio-leaching tests (BAT) showed significantly more Cu was extracted by bio-leaching compared to the baseline copper extraction determined by diagnostic leaching. Although batch bio-leaching of copper generally increased with temperature, sulphide oxidation and bio-leaching of copper from the final concentrate at 48°C (after 7 days) was slightly higher than at 67°C (after 6.25 days).

Bio-leaching kinetics were also determined in GEOCOAT<sup>®</sup> column tests (2m high, 5.5 kg concentrate) for both Caraiba rougher concentrate and final concentrate coated onto MM support rock. These tests were operated for 95 to 96 days at ca. 30°C, 40°C, 50°C, 60° and 70°. Copper extraction increased with increasing temperature.

In the GEOCOAT<sup>®</sup> column tests, when using the MM support rock, Cu extraction was 65% for final concentrate at 30oC. At 70° Cu extraction was 96% from final concentrate. The two methods for calculating Cu extraction, based on solid residue and based on silica analysis, are averaged, and show 90% copper extraction is achieved at 61°C.

Additional flotation testwork has been completed on the biooxidation residues. This work has established that the residual copper sulphides and gold in the biooxidation residues can be readily recovered to a high grade concentrate suited to blending back into the main plant concentrate production.

The results of these tests were very encouraging. The ability to reproduce these results at the ambient temperatures prevailing on site will only be confirmed by full scale trial in the demonstration heap.

GeoBiotics, LLC  
12345 West Alameda Parkway, Suite 310  
Lakewood, Colorado 80228 USA  
1-303-277-0300  
1-303-277-1772 (fax)



## **ACKNOWLEDGEMENTS**

The authors wish to thank the General Management of Mineração Caraíba S.A. for permission to publish this paper. The technical contributions to the work made by the plant personal at Caraiba and staff at Little Bear Laboratories in Denver and GEOMET S.A. in Santiago are also acknowledged.