

## **A NEW TEST PROCEDURE TO CHARACTERIZE THE REACTIVITY OF MINING WASTE**

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### **Abstract**

Process Research Associates Ltd. is currently developing a new test procedure that can be used to characterize the reactivity of mining waste materials. The test involves adding the sample to sodium hypochlorite solution, and monitoring the hypochlorite consumption. Results from preliminary tests on pyrite and pyrrhotite show that the test predicts that pyrrhotite is more reactive than pyrite which is well known. Results from tests with waste rock and ore samples revealed differences in reactivity that could not be discerned from the available rock classification and ABA information. More research will be conducted to define the test conditions and to evaluate the effects of factors such as mineralogy, oxidant concentration, sample size, particle size and specific surface area. The test will supplement information from ABA tests to more accurately characterize the reactivity of mining waste materials and provide design information for waste disposal.

### **Introduction**

Concern regarding the impact of acid rock drainage (ARD) on the environment has resulted in the need for reliable methods to predict the potential for the generation of acid from mining waste material. Current prediction procedures include static (acid base accounting) and kinetic (humidity cell/column) tests. Acid base accounting (ABA) predicts the theoretical potential to generate acid and can be misleading since the test assumes that all sulphur will produce acid without consideration of the reactivity of the sulphur. Humidity cell and column tests provide some information about reactivity but are not practical for engineering purposes. These kinetic tests do not provide statistically valid information that can be used for design.

A quick and simple test that provides information about reactivity is therefore needed. Process Research Associates Ltd. has been developing such a test. The test involves placing mineral samples

in a strong oxidizing environment and monitoring parameters that indicate reactivity of the samples such as oxidant consumption, sulphide oxidation, redox potential, pH and temperature. The steps to develop the test included:

1. Selection of an appropriate oxidant;
2. Testing various procedures to identify parameters that could be used to characterize the reactivity of the sample; and
3. Evaluation with samples from potential and operating mine sites.

To evaluate the test, the results for each sample are related to available mineralogical, acid base accounting and humidity cell information. The test is at an early stage of development and more work is required to determine its application to ARD prediction.

### **Selection of an Oxidizing Agent**

Various oxidizing agents were considered for the test including sodium hypochlorite, hydrogen peroxide, potassium permanganate and potassium dichromate. The criteria for selecting an appropriate oxidant were:

1. It should produce the same oxidation products that are produced in natural systems;
2. It should not decompose readily;
3. It should be easy and safe to handle; and
4. It should be inexpensive and readily available.

Preliminary tests were performed on samples of sodium sulphide and pyrite using the four oxidizing agents. Details of these tests are presented elsewhere (Klein, 1993); however, the main results are summarized here. The tests involved adding the oxidants to the sulphide suspensions and monitoring redox potential, sulphide ion potential, pH and temperature.

Potassium permanganate and potassium dichromate produced the weakest responses. For example, when adding 25 mL increments of 5% potassium dichromate solution to 100 mL of 2% sodium sulphide solution, the redox potential increased slightly initially, but remained relatively constant with subsequent additions. Cleaning the electrode, which appeared to have a thin coating on its surface,

resulted in a positive shift in the potential. After a short period of time, however, it approached the level recorded prior to cleaning. Tests using sodium hypochlorite and hydrogen peroxide produced much more significant changes to redox potential with no apparent coating of the electrode. The dark colours of the potassium permanganate and potassium dichromate solutions made observation of the sulphide oxidation products difficult to see the types of sulphide oxidation products that were formed.

While tests with hydrogen peroxide showed very distinct and interpretable responses, the oxidant decomposed rapidly as indicated by effervescence. This loss of oxidant eliminated the capability to measure reagent consumption. Tests with sodium hypochlorite also produced interpretable responses, but the reagent did not decompose as rapidly. This was verified using a 100 mL of 5% NaOCl which was agitated in a 250 mL flask for 24 hours. The concentration of sodium hypochlorite decreased by a small amount from 5.28% to 5.13% over this time period. The oxidation of pyrite by hypochlorite resulted in the formation of visible ferric hydroxide coatings on the pyrite surfaces. Also, sodium hypochlorite is readily available, inexpensive, and is safe and easy to handle making it a suitable oxidant for a reactivity test. Sodium hypochlorite was therefore selected for further testing.

## **Description of the Test Work**

To develop the procedures, a mineral sample was added to a hypochlorite solution and the hypochlorite concentration, redox potential and pH were followed with time. The extent of sulphide oxidation as well as sulphate levels were also monitored. The detailed procedure was as follows:

1. Add 100 ml of 5% NaOCl solution to each of five 250 mL Erlenmeyer flasks.
2. Add 5.00 g of pulverized (-200 mesh) mineral sample to each flask.
3. Place flasks in orbital shaker and then remove one flask after intervals of 1, 2, 4, 8 and 24 hours.
4. Upon the removal of a flask, filter at 0.45  $\mu\text{m}$  and immediately determine the hypochlorite concentration, sulphate concentration, redox potential and pH.
5. Wash the filtered solids and analyze for sulphide sulphur.

The NaOCl solution concentration of 5% was selected since it is the common concentration of commercial bleach. Samples are pulverized in an attempt to equalize the effects of specific surface area and this allows the use of the same samples that are used for acid base accounting tests. The sample size of 5 grams was determined empirically to give an easily measured response. The samples used had sulphide grades ranging from 2.5% to 11.5%. For tests on high grade sulphide mineral samples (e.g. pyrite, pyrrhotite concentrates), 1 g samples were used.

From the data, the sodium hypochlorite consumption per gram of sample can be calculated and plotted as a function of time. The slope of the curve provides a consumption rate for a specific sample. Plotting the other measured parameters; provided additional information about reactivity.

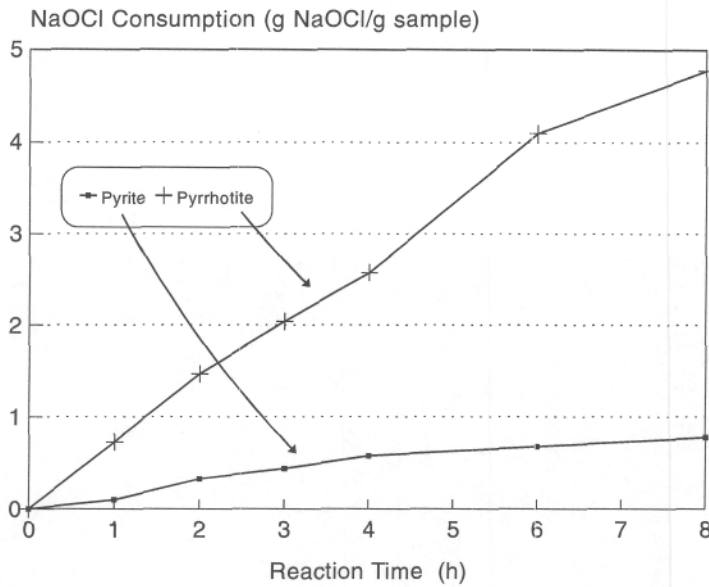
## **Evaluation of Test Results**

Two sets of tests were performed to evaluate the procedure. The first set was performed on concentrates of pyrite and pyrrhotite. The second set of tests were performed on waste rock samples from the QR Project, a gold property near Quesnel, BC. The results of the tests are presented below.

### Tests on Sulphide Mineral Concentrates

Tests were performed on samples of pyrite and pyrrhotite concentrate (greater than 95% pure mineral). The samples were pulverized to -200 mesh. All tests were performed in duplicate. The NaOCl consumptions were calculated using the measured concentrations and the results for the first 8 hours are plotted in Figure 1.

The plots show that the pyrrhotite consumes the hypochlorite faster than the pyrite, indicating that the pyrrhotite is more reactive than the pyrite. The results demonstrate that the test can rank reactivities of these two minerals in conformity with results generally accepted in the biohydrometallurgical literature.

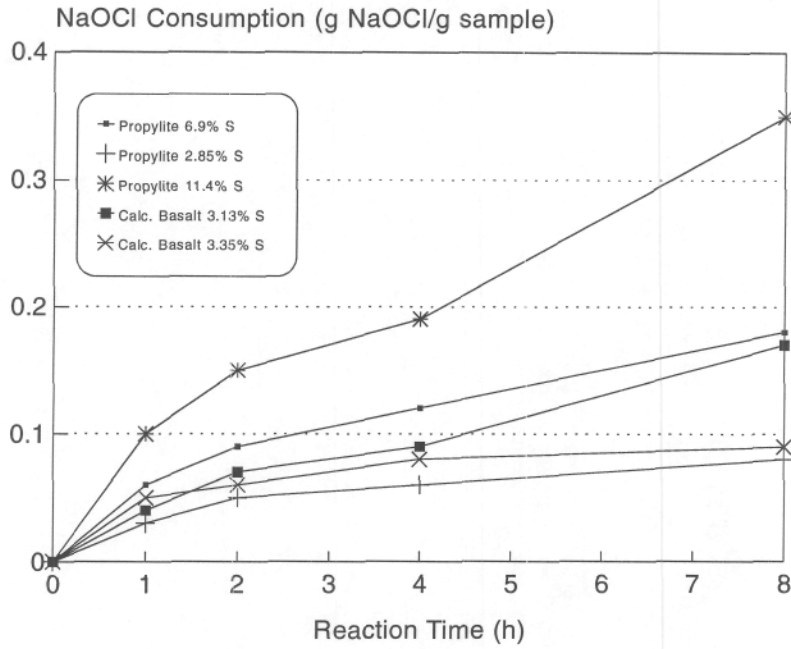


**Figure 1**  
**NaOCl Consumption Rates - Pyrite & Pyrrhotite:**

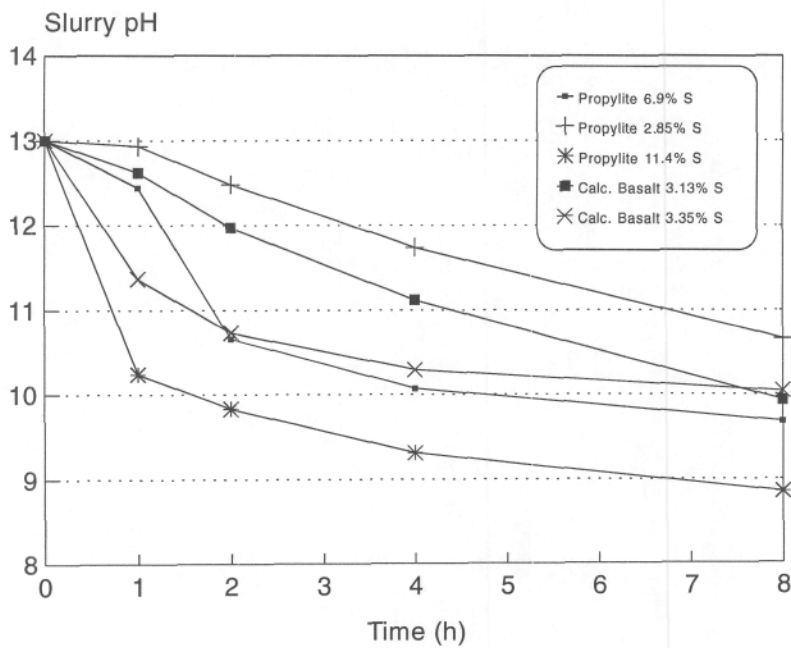
### Tests on Waste Rock Samples

To demonstrate the applicability of the test to waste rock, tests were performed on five samples from the QR Project, a potential gold mining operation in the central interior of British Columbia. Acid base accounting and chemical composition information about the samples is presented in Tables 1 and 2. Sample 1 is described as propylitic basalt waste rock and contains 2.85% sulphur which is mostly in the form of pyrite. Samples 2 and 3 are classified as calcareous basalt. The ABA data for the two samples indicates that they are very similar. The levels of metals, as presented in Table 2, shows that sample 2 has a much higher copper grade (1481 ppm versus 93 ppm). This is attributed to chalcopyrite mineralization accounting for approximately half of the sulphide in the material. Samples 4 and 5 are both classified as low grade ore although sample 4 contains substantially less sulphur than sample 5 (6.92% versus 11.4%, respectively). Sample 5 also contains significant chalcopyrite that is not present in sample 4 (1935 ppm Cu versus 345 ppm Cu, respectively).

All five samples were subjected to the test procedure described above. Figures 2 and 3 are plots of the NaOCl consumption and pH versus time, respectively, for an 8 hour period. The results of the tests are summarized in Table 3.



**Figure 2**  
NaOCl Consumption Rates - QR Waste Rock & Ore



**Figure 3**  
Slurry pH - QR Waste Rock & Ore

Table 1  
QR Waste Rock & Ore Reactivity Test - ABA Results

| Sample # | Rock Type Grouping                | S (tot) % | S(SO4) % | NP  | MPA | Net NP | NP/MPA |
|----------|-----------------------------------|-----------|----------|-----|-----|--------|--------|
| 1        | Propylitic Basalt                 | 2.85      | <0.01    | 115 | 89  | 26     | 1.3    |
| 2        | Calcareous Basalt                 | 3.13      | <0.01    | 226 | 98  | 128    | 2.3    |
| 3        | Calcareous Basalt                 | 3.35      | 0.01     | 243 | 105 | 138    | 2.3    |
| 4        | Propylitic Basalt - Low Grade Ore | 6.92      | 0.01     | 94  | 216 | -122   | 0.4    |
| 5        | Propylitic Basalt - Low Grade Ore | 11.4      | 0.01     | 112 | 356 | -244   | 0.3    |

Table 2  
Metals and Whole Rock Results

| Sample # | Rock Type Grouping                | Ag ppm | Au g/tonne | Cu ppm | Fe % | Ni ppm | Zn ppm | Whole Rock Analyses |       |       |
|----------|-----------------------------------|--------|------------|--------|------|--------|--------|---------------------|-------|-------|
|          |                                   |        |            |        |      |        |        | SiO2 %              | MgO % | CaO % |
| 1        | Propylitic Basalt                 | 0.3    | 0.2        | 132    | 3.85 | 37     | 35     | 39.34               | 4.79  | 17.82 |
| 2        | Calcareous Basalt                 | 4.4    | nil        | 1481   | 4.94 | 60     | 68     | 35.69               | 5.55  | 21.79 |
| 3        | Calcareous Basalt                 | 0.3    | nil        | 93     | 3.83 | 38     | 29     | 36.26               | 3.92  | 24.39 |
| 4        | Propylitic Basalt - Low Grade Ore | 0.8    | 7.0        | 345    | 7.19 | 34     | 49     | 37.08               | 4.20  | 13.07 |
| 5        | Propylitic Basalt - Low Grade Ore | 4.0    | 5.9        | 1935   | 9.54 | 35     | 43     | 30.40               | 3.04  | 15.36 |

Table 3  
NaOCl Test Summary at 8 Hours

| Sample # | Rock Type Grouping                | Slurry pH | Eh  | NaOCl Consumption  |                    | % S Oxidized after 24 h |
|----------|-----------------------------------|-----------|-----|--------------------|--------------------|-------------------------|
|          |                                   |           |     | g NaOCl/g sample/h | g NaOCl/g Totals/h |                         |
| 1        | Propylitic Basalt                 | 10.66     | 731 | 0.64               | 0.33               | 33.3                    |
| 2        | Calcareous Basalt                 | 9.93      | 847 | 1.36               | 0.66               | 38.0                    |
| 3        | Calcareous Basalt                 | 10.04     | 856 | 0.72               | 0.34               | 35.2                    |
| 4        | Propylitic Basalt - Low Grade Ore | 9.68      | 842 | 1.44               | 0.32               | 31.5                    |
| 5        | Propylitic Basalt - Low Grade Ore | 8.95      | 887 | 2.8                | 0.38               | 24.9                    |

Examination of Figures 2 and 3 and Table 1 reveals that, in general, NaOCl consumption increases and pH levels decrease with increasing sulphur grade.

The responses for samples 2 and 3 indicate that sample 2 is more reactive than sample 3. The rock type classifications and ABA test data are practically the same for the two samples and provide no explanation for the difference in reactivities. As described above and as indicated by copper contents presented in Table 2, sample 2 has significant amounts of chalcopyrite. The electrochemical series presented by Yakhontova (1985, presented in Rossi, 1990) indicates that chalcopyrite is more easily oxidized than pyrite and would therefore be expected to consume oxidant more rapidly. Therefore, it is apparent that the oxidant consumption test provided important information about the samples that could not be obtained from the ABA data.

## **Summary and Conclusions**

Research to develop a test that characterizes the reactivity of mining waste materials is underway at Process Research Associates Ltd. In the test procedure, a pulverized sample is added to a strong oxidizing solution and parameters that indicate its oxidation are monitored (oxidant consumption, redox potential and pH). From preliminary testwork with various oxidants, sodium hypochlorite was selected for the test. Comparing the hypochlorite consumption rates for pyrite and pyrrhotite revealed that pyrite is less reactive than pyrrhotite which is known to be true. Tests were also performed on two waste rock samples that had the same rock classification and practically the same ABA characteristics. Despite this, the samples had quite different reactivities as indicated by hypochlorite consumptions which could be related due to their different mineralogical characteristics.

## **Acknowledgements**

The authors would like to thank the Science Council of British Columbia, Homestake Canada Inc., and Process Research Associates Ltd. for their financial support of this research project.



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