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Technical Article

Evaluation of Laboratory Kinetic Test Methods for Measuring Rates of Weathering

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Abstract. A study was conducted to compare laboratory kinetic test methods for predicting acid rock drainage rates of weathering. Five laboratory kinetic test protocols (standard humidity cells, non-aerated cells, tall cells, shaken cells and NP depletion columns) were evaluated by comparing sulfate release and NP depletion rates, and predicted time to acidity (defined as pH 6). Our tests indicate that the standard humidity cell creates an unnatural oxidizing environment due to its extreme wetting and drying cycles, and therefore produces erratic results. The non-aerated test cells likely create an oxidizing environment that more closely represents the natural conditions, producing more consistent results and therefore a better estimate of the sulfide oxidation rate. The practice of shaking cells to promote efficient rinsing of weathering products disturbs the oxidizing environment and may retard the oxidation rate. Accelerating NP depletion by the high addition of acidic water creates an unnatural leaching environment, producing results that are not consistent with those obtained from other testing protocols having more natural leaching environments.

Key words: Acid rock drainage; kinetic testing; humidity cell; time to acidity

Introduction

Planning and management is essential to minimize the environmental impacts associated with the deposition of mine and milling wastes, particularly with respect to acid rock drainage (ARD). Kinetic humidity cell tests are widely used to estimate the rates of weathering in order to predict the rate of acid generation, the rate of neutralization potential (NP) depletion, and the time until ARD is generated (Day et al. 1997; Li 1997; Morin and Hutt 1997; Price 1997; White and Sorini 1997).

The humidity cell test is designed to accelerate the weathering rate of a mine waste and to allow the weathering products to be collected and analyzed. A standard humidity cell is operated on a weekly cycle that is comprised of three days of dry air, three days of moist air, and a rinse with distilled water on the seventh day. The pumping of air through the sample is conducted to ensure sulfide oxidation reactions are not limited by low oxygen concentrations. The average weekly release rate of sulfate from a humidity cell (measured as mg SO_4/kg/wk) represents the sulfide oxidation rate and the average weekly release rate of calcium and magnesium determine the rate of NP depletion (measured as mg $CaCO_3$/kg/wk).

Prediction of the time before a waste pile produces ARD (lag time) is typically determined by the time required to deplete the available NP and the time required to completely oxidize the sulfide. The years of accelerated weathering to deplete a sample's NP is calculated from the release rates of calcium and magnesium. However, the total NP is not available for neutralizing acidity since a portion of the neutralizing minerals is encapsulated within rock particles. Available NP has been tied to specific percentages of total NP (Ferguson and Morin 1991; Rescan 1992), or quantities, such as the 10-15 kg of unavailable $CaCO_3$/tonne advocated by Morin and Hutt (1997), and by applying mineral reactivity factors to the contained neutralizing minerals (Li 1997).

The humidity cell apparatus and protocol developed by Caruccio (1968) has been modified by researchers, industry, and government. Current leaching protocols include: 1) trickle-leaching the sample; 2) flooding and then draining the sample; and 3) flooding, stirring/shaking the sample to loosen weathering products, and then draining the sample (Morin and Hutt 1997; Price 1997; White and Sorini 1997). Other conditions of laboratory kinetic tests that have been varied include volume of flush water, frequency of flushes, rate of supplied airflow, amount of material used, and particle size distribution (Brodie et al. 1991; Day et al. 1997; Pool and Balderrama 1994; Soregaroli and Lawrence 1998). The objectives of this study were to examine the effects of humidity cell protocol variables on rates of weathering to determine their strengths and weaknesses.

Materials and Methods

Sample Preparation and Characterization

The samples selected for this study represent the two main waste types for the proposed Red Mountain mine in northwestern British Columbia: feldspar porphyritic intrusive rock (samples HC-1 and ABA-2) and bedded tuffaceous sedimentary rock (sample HC-2). The HC-1 and HC-2 samples were obtained from underground slashes. The ABA-2 sample was comprised from samples collected during the development of 25 m of exploration drift. Total weight of rock collected and initial particle size are presented in Table 1.

Representative minus 0.64-cm (¼-inch) sub-samples were obtained using a series of crushing, screening and splitting stages. Mineralogy of the samples was determined from hand specimen examinations and petrographic microscopic analyses. Acid-Base Accounting (ABA) tests were conducted using the "modified" procedure as described by Lawrence (1990). Mineral compositions and results of ABA results are summarized in Table 2.

The feldspar porphyry samples (HC-1 and ABA-2), in hand specimen, show 40-50% white phenocrysts (1-2 mm) within a pale green-grey, aphanitic groundmass. The main sulfide minerals are pyrite and pyrrhotite that are mostly finely disseminated, although a portion occurs as fracture fills. The ABA-2 sample is pyrrhotite rich (5-7%). Minor wispy sphalerite (0.5%) occurs in both samples. The HC-2 sedimentary sample was pale green to light grey, medium to fine grained and locally banded. It contains disseminated pyrite and pyrrhotite and wispy sphalerite.

The paste pH for all 3 samples was greater than about 8.3, indicating that the samples had not started generating acid. However, the negative net NP values and NP ratios of less than 0.25 indicate a strong potential for generating acid from these materials at some point in time.

Kinetic Test Cells and Protocols

Five different types of kinetic tests were conducted: standard humidity cells, non-aerated cells, tall cells, shaken cells, and NP columns. The test protocols are summarized in Table 3 and are described below. The tests conducted for each sample are listed in Table 4. All tests were conducted at a room temperature of approximately 20°C.

Table 1. Samples weights and particle sizes

Sample	Weight (kg)	Particle Size (% -5-cm)
HC-1	230	10
ABA-2	91	80
HC-2	110	10

Table 2. Mineralogical and ABA characterization of test samples

Parameter*	HC-1	ABA-2	HC-2
Pyrite	4-5	-	1-2
Pyrrhotite	2-3	5-7	2-3
Sphalerite	0.5		1-2
K-feldspar	30-35	35-40	35-40
Quartz	15-20	15-20	15-20
Chlorite	10-15	15-20	3-5
Sericite	5-10	3-5	20-25
Carbonate	0.5	0.5	tr
Paste pH	8.35	8.22	8.35
Sulfide S	5.82	4.74	3.95
AP	182	148	123
NP	44.6	33.1	11.9
Net NP	-137	-115	-112
NPR	0.25	0.22	0.10

* All values are expressed in percentage with the exception of AP, NP, and Net NP, which have units of kg $CaCO_3$/tonne, NPR, which is a ratio, and paste pH.

The standard humidity cell was 10 cm in diameter by 20 cm tall and held 1 kg of rock that was crushed to minus 0.64-cm (¼-inch) inch. The sample was supported by a perforated acrylic plate, which was covered with 3 layers of fine mesh screen. The air inlet was located below the perforated plate and the outlet was located above the sample. Exiting air passed through water contained in a "bubbler" that allowed for visual assessment of the airflow rate (White and Sorini 1997). An O-ring seal installed in the lid of the cell prevented airflow leakage and thereby ensured good control of airflow.

The first two leaching cycles involved flooding the samples with distilled, deionized water for an hour, and then draining and reflooding them for a day before draining. The weekly cycle consisted of passing dry air through the cell for 3 days, followed by moist air from a humidifier for 3 days and washing with water on the 7th day. The airflow through the cell was maintained at approximately 0.5 L/minute. The water temperature in the humidifier was maintained at 28-30°C for the humid air cycles.

For the wash, 500-mL distilled deionized water was dripped into the cell over a period of 1 hour and the volume of the leachate collected after 1 day was recorded. Cell weights (moisture content) were measured 3 times per week: at the end of the dry air

Table 3. Summary of kinetic test protocols.

Description	Standard	Non-aerated	Tall	Shaken	NP Column
Diameter (cm)	10	10	10	10	5
Height (cm)	20	20	40	20	180
Weight (kg)	1	1	3	1	5
Air flow	0.5 Lpm	0 Lpm	0.5 Lpm	0.5 Lpm	Nitrogen purge
Wash	500 mL – 1 hr drip	500 mL – 1 hr drip	500 mL – 1 hr drip	500 mL – 1 hr soak, swirl 30 sec	3.6 L/wk – pH 3, continuous drip
Cycle (days)	7	7	7	7	NA
Dry Air	3	NA	3	3	NA
Moist Air	3	NA	3	3	NA
Rinse	1	1	1	1	NA

Table 4. Kinetic tests conducted on samples

HC-1	ABA-2	HC-2
Standard	Standard (duplicate)	Standard
Non-aerated	Tall	Tall
Tall	Shaken	Shaken
Shaken	NP Column	NP Column
NP Column		

period, at the end of the moist air period, and at the end of the wash day.

The non-aerated cell setup was the same as the standard cell but the protocol was different. The non-aerated cell was not subjected to weekly cycles of dry and wet air, but was trickle leached over 1 hour with 500-mL of distilled deionized water per week. The cell was not actively aerated but remained open at the top and bottom allowing unrestricted oxygen access.

The tall cell was 10 cm in diameter by 40 cm tall, and held a 3-kg charge of minus 0.64-cm (¼-inch) material. The operating methodology used was similar to that for the standard cell and included the use of a "bubbler" for air-flow control. The wash water volume was the same as for the standard cell (500 mL), resulting in a lower water to solid ratio than for the standard cell.

The shaken cell was set up and operated in a similar manner to the standard cell with respect to the weekly cycle of dry/moist air, rate of airflow, and humid air temperatures. For washing, however, the cell was flooded with 500 mL of distilled deionized water for an hour and then gently moved in a circular motion along a horizontal plane to cause swirling of the rinse water. This procedure is similar in principle to the protocols outlined in the BC ARD Guidelines (Price 1997). The swirling was maintained for 30 seconds before draining to promote the removal of all the weathering products. The fines that drained from the cell were returned to the cell at the start of the next leach cycle.

Leach column test work was conducted to determine the available NP. The "NP Columns" accelerated NP depletion by continuously supplying acidified distilled deionized water to the column. The 5-cm diameter by 180 cm tall columns were loaded with 5 kg of minus 0.64-cm (¼-inch) material. After two months, the columns were fitted with a hose to accommodate the hookup of a compressed nitrogen gas cylinder. Approximately 3.6 L of acidified rinse water (pH 3 with H_2SO_4) was applied to the column weekly for 360 days. The column leachate was collected once a week for the first 14 weeks, then once every 2-3 weeks. Nitrogen was routinely blown through the columns to purge the closed system of oxygen.

The leachate pH and conductivity were recorded after each wash cycle. Alkalinity, sulfate, Ca, Mg, K, and Na concentrations were determined after every second cycle using analytical procedures described in Standard Methods for the Examination of Water and Wastewater (1989). Sulfate concentrations were determined by a turbidimetric method using a Perkin-Elmer Lambda 8 UV/VIS Spectrophotometer. Ca, Mg, K and Na concentrations were measured by atomic absorption spectrophotometry.

Results and Discussion

The kinetic test protocols were compared with respect to the sulfate release, NP depletion and estimated time to acidity. Similar operating conditions were deemed necessary to ensure repeatability of results and to permit meaningful comparisons of test protocols. However, difficulties were encountered in maintaining equal and uniform airflow through the standard, tall, and shaken cells.

The moisture content of the non-aerated cell and tall cells remained relatively steady on a week-to-week basis. The moisture content of the standard and shaken cells fluctuated throughout the testing and, on average, these cells contained less moisture prior to leaching than the other cells. Visual observation of the tall cells revealed that only the bottom portion of these cells was drying during the 3-day dry air portion of the cycle.

All tests on the two feldspar porphyry samples, HC-1 and ABA-2, maintained a leachate pH above 7 for the duration of the test period. All tests conducted with the sedimentary sample HC-2 produced acid leachate.

Sulfate Release

Sulfate release profiles from kinetic tests on the samples are shown in Figures 1-3. The horizontal lines represent average sulfate release rates obtained from the last 5 cycles of the standard humidity cell tests. For the HC-2 sample, the horizontal line on Figure 3 represents the average sulfate release rate from the 5 cycles prior to pH dropping to less than 6. The lines are included to assist with comparison of results from the various test protocols.

The patterns of sulfate release as a function of time for the non-aerated and tall cells were less erratic and approached stabilization earlier than the standard cells (Figures 1-3). For sample HC-1, sulfate release stabilized at about 200 and 225 days for the non-aerated and tall cells, respectively, compared to 250 days for the standard cell. After stabilization, the sulfate release for the non-aerated and tall cells varied by less than about 10 mg/kg/wk, compared to as much as 25 mg/kg/wk for the standard cell. The difference in sulfate release patterns is attributed to the relatively consistent moisture content in the tall and non-aerated cells.

Consistent moisture content creates an operating condition that is similar to an actual waste rock environment. Field tests using these samples (Frostad 1999; Frostad et al. 2000b) showed that considerable time was required to attain the field capacity moisture content (e.g., HC-1 required 1.5 years). Once field capacity was attained, the variations in water content resulting from wetting and drying was small. Therefore, conventional humidity cells that have weekly drying and wetting cycles create an unnatural environment that could affect important factors in the acid generation process. The erratic pattern of sulfate release as a function of time for the standard humidity cells is evidence of this unnatural environment. These results suggest that test protocols should be selected based on site-specific factors.

In the small-scale non-aerated cell, the overall oxidation rate was similar to those obtained from the standard humidity cell. Lapakko and White (2000) also generated similar data from duplicate samples tested by non-aerated and humidity cell protocols. Therefore, non-aerating does not seem to limit oxygen diffusion rates. This finding indicates that eliminating the dry and wet air cycles does not affect oxidation rates and may result in more repeatable and representative reaction rates being returned in a shorter period of time.

Final sulfate release rates for samples HC-1 and HC-2 in the tall cells were similar to those obtained with the standard cell (Figures 1 and 3), suggesting that oxygen diffusion is not affected. However, for ABA-2 samples, the tall cell rates were slightly lower than in the standard cell (Figure 2). This sample contained a considerable amount of fine material that likely reduced the permeability and limited oxygen diffusion.

For the shaken cells, the sulfide oxidation rate slowly decreased, then increased (Figures 1-3). The objective of the shaken cell protocol is to promote rinsing for the removal of sulfate and other reaction products created during a cycle. However, after 200 days, the shaken cells released a cumulative amount of sulfate that was less than or comparable to the standard and tall cells (Table 5). For samples ABA-2 and HC-1, the lower cumulative amount of sulfate released from the shaken cell suggests that the initial rates of sulfide oxidation were lower.

There are several possible explanations for the lower shaken cell oxidation rates. Particle segregation was observed in the shaken cells, which resulted in an accumulation of fine material at the bottom of the cell. This segregation was pronounced during the first two months of testing and may have influenced the diffusion of oxygen. Specifically, the fine material at the bottom of the shaken cells retained water, which may have limited oxygen diffusion to much of the dissolved ferrous iron and exposed sulfide surfaces. The oxidizing environment within the accumulated fine material would be conceivably quite different than in the well-mixed standard cell. Due to the high surface area of the fine fraction, a small change in the oxidation environment can significantly influence the rate of sulfide oxidation. The fine material may also have contained a higher concentration of liberated sulfide minerals (Lapakko 1994; Price and Kwong 1997), since soft sulfide mineral grains break easier during sample preparation than most relatively harder minerals.

The continuous disturbance of particles in the shaken cell may hinder the establishment of micro-environments that favor sulfide oxidation. Micro-environments can form, in part, by the non-uniform distribution of sulfide and neutralizing minerals.

Table 5. Cumulative sulfate released by kinetic cells after 200 days

Kinetic	HC-1		ABA-2		HC-2	
Cell Type	(mg/kg)	(mg/kg/d)	(mg/kg)	(mg/kg/d)	(mg/kg)	(mg/kg/d)
Standard	1951	9.8	3150	15.8	2264	11.3
Standard (dup)			3155	15.8		
Non-aerated	1494	7.5				
Tall	1994	10.0	2708	13.5	2365	11.8
Shaken	1988	9.9	1936	9.7	2197	11.0

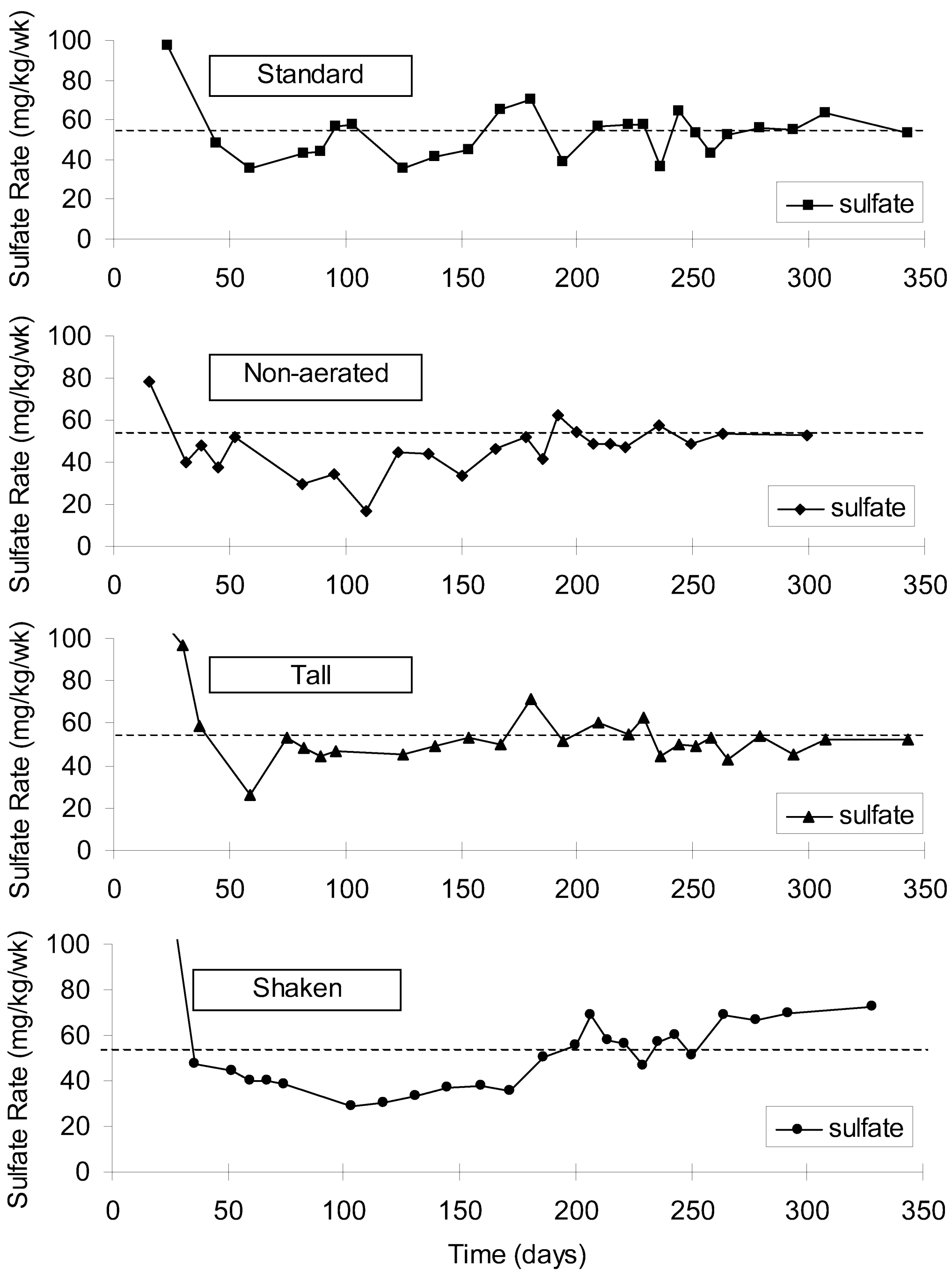

Figure 1. Sulfate release over time for standard, non-aerated, tall, and shaken cells containing HC-1 with average (dashed line) from the last five cycles of the standard cell tests

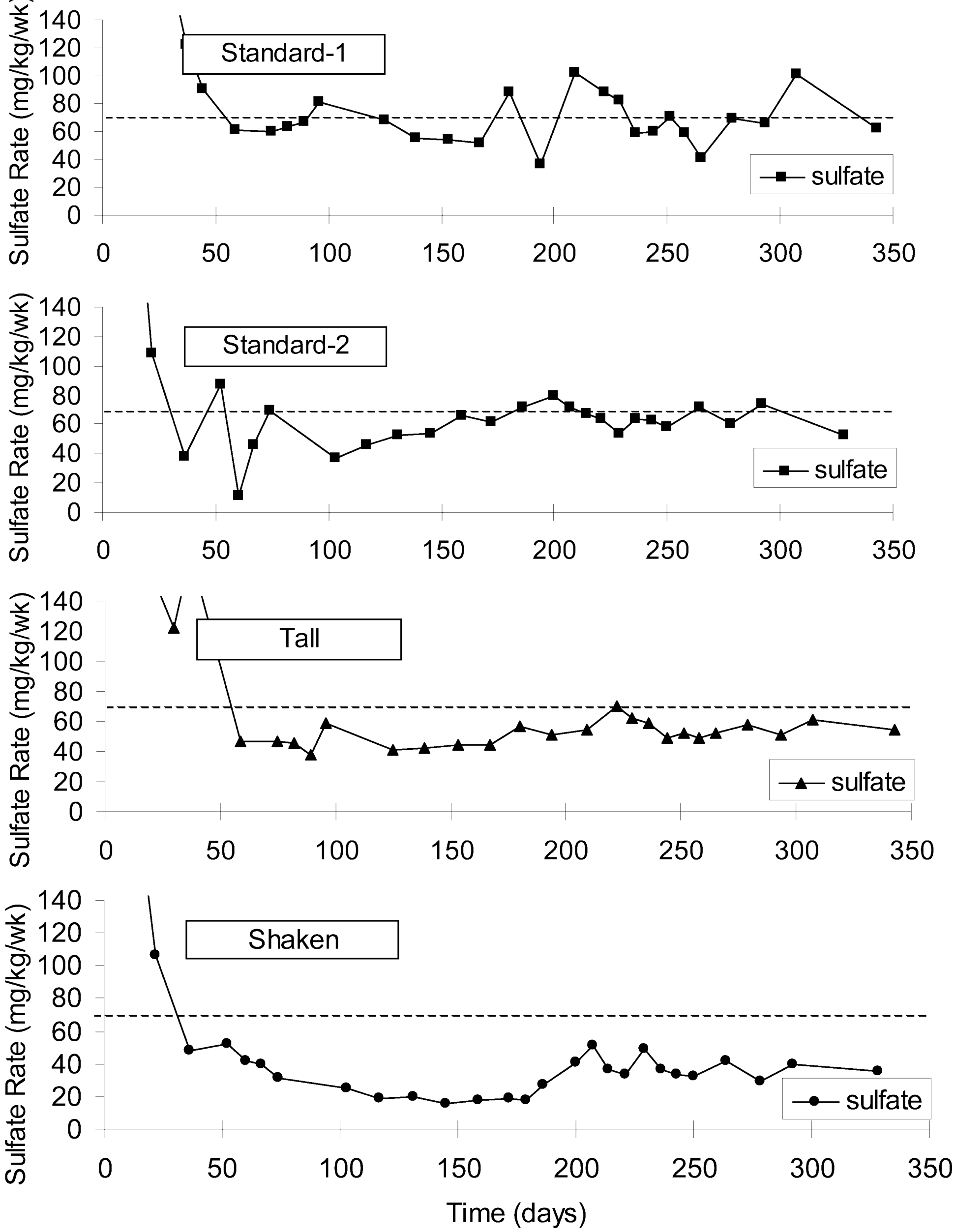

Figure 2. Sulfate release over time for standard, tall, and shaken cells containing ABA-2 with average (dashed line) from the last five cycles of the standard cell tests

Locations with high concentrations of sulfide minerals and low concentrations of neutralizing minerals, referred to as "hot spots", will generate acid that is not readily neutralized.

For sample HC-1, after 250 days of weathering, the shaken cell had a higher sulfate release rate than the standard, tall, and non-aerated cells. The higher rate may be explained by the formation of favorable oxidation conditions in the accumulated fine material. For the pyrrhotite-rich sample, the final sulfate release rate for the shaken cell remained low compared to the standard and tall cells. There is no obvious interpretation for this, emphasizing the point that the shaken cell protocol can drastically influence the processes that are being monitored and is therefore not a good procedure for obtaining sulfide oxidation rates.

NP Depletion

The amount of calcium and magnesium in solution represents the amount of carbonate minerals dissolved. For the standard, tall, and shaken cells, the patterns of calcium release rates were very similar to those patterns of sulfate release. This correlation indicates that the dissolution of carbonate minerals is directly influenced by the rate of acid generation.

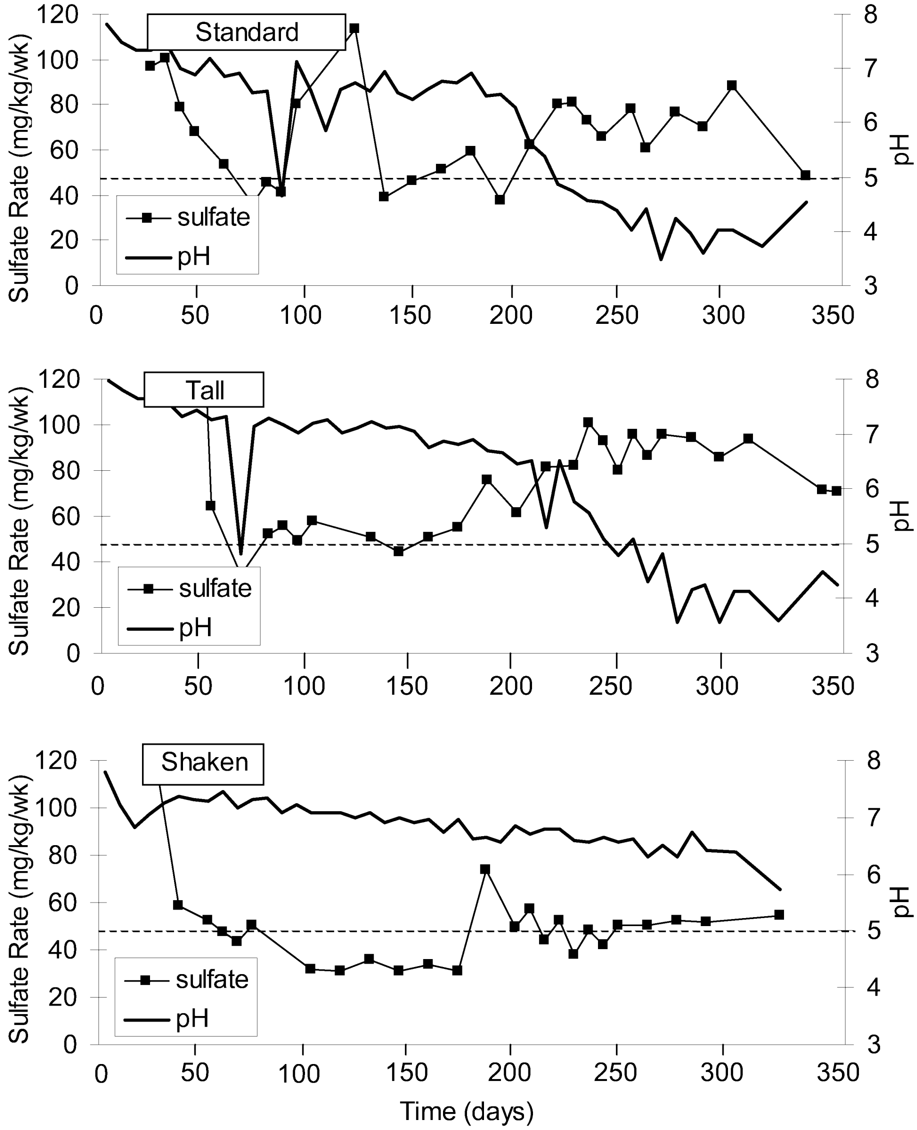

Figure 3. Sulfate release over time for standard, tall, and shaken cells containing HC-2 with average (dashed line) from the last five cycles of the standard cell tests

The molar ratio of calcium and magnesium to sulfate is referred to as the "carbonate molar ratio" and is an indicator of the amount of excess neutralizing capacity and thus the effectiveness of the carbonate minerals (Morin and Hutt 1997). A pH of 6 was used to signify the onset of acidic drainage for this study and is referred to as the point of acidity. The carbonate molar ratio for all the standard, tall, and shaken cells decreased during the initial test period. The ratio for the feldspar porphyry samples stabilized near unity, while the sedimentary sample reached unity at the point of acidity, and continued to decrease.

NP Column tests were conducted to estimate the amount of NP available to neutralize acidic drainage in mining waste. Figure 4 shows the NP depletion rate, sulfate release rate, and pH profiles for the NP column test on sample HC-2. During testing, an orange-brown precipitation front was observed in the column that progressed steadily from the top to the bottom. The sulfate release rate began increasing after 80 days, the NP depletion rate began decreasing after 200 days and the two rates were equal (molar ratio of 1.0) at 227 days. Reaching a molar ratio of 1.0 corresponded with a dramatic pH drop to below 6 and the precipitation front reaching the bottom of the column.

The high volume of acidified rinse water applied to the NP column increased the rate of NP depletion relative to the other kinetic tests. For sample HC-2, the average NP depletion rate prior to acidity in the NP column was 244 mg $CaCO_3$/kg/wk, whereas the NP depletion rates from the other kinetic tests were significantly lower, ranging from 85-121 mg $CaCO_3$/kg/wk. The NP columns containing samples HC-1 and ABA-2 did not produce acidic drainage during the test period but the rate of NP depletion was similarly greater than from the other kinetic tests.

Applying acidified rinse water to the NP column apparently overestimated the available NP relative to the other protocols. For the NP column containing sample HC-2, 67% of the total NP was depleted from the sample at the point of acidity. For the standard, tall, and shaken cells, the NP depletion at the point of acidity were only 26-33% (Table 6). The available NP from the NP column test was therefore twice the amount determined from the standard, tall and shaken cells. The results show that estimates of available NP will depend on test conditions, and that acidified rinse water should not be used to obtain a quick estimate of available NP. The results raise issues related to the use of NP depletion from laboratory testing for the prediction of time to acidity in the field.

Time to Acidity

Kinetic tests have been used to estimate the time to acidity for reactive waste rock to determine how long material can be stored prior to disposal under water. After sufficient time, all kinetic tests conducted on the HC-2 sample produced acidic leachate (pH less than 6.0). The respective times to acidity estimates are presented in Table 6 along with the cumulative amount of NP depleted and the cumulative amount of sulfate produced at the point of acidity.

The shaken cell required the longest time to become acidic and produced the greatest amount of sulfate (acidity). The NP column released the greatest amount of NP prior to acidity, but this protocol did not decrease the time to acidity. These results demonstrate that increasing the rate of NP depletion of a sample does not necessarily shorten the time to acidity. Therefore, the rate of NP depletion may not always be a good indicator for estimating time to acidity.

The results in Table 6 reveal a relationship between time to acidity and cumulative sulfate release; with an increase in the time to acidity, there is a corresponding increase in the cumulative sulfate produced. The cumulative sulfate release at the time of acidity is related to the amount of sulfide oxidation that has occurred and, therefore, the amount of acidity effectively neutralized.

We believe that the shaken cell protocol prolonged the time to acidity relative to the other kinetic tests by decreasing the rate of sulfide oxidation. As indicated by the results in Table 6, the slow oxidation rate of the shaken cell protocol also corresponded with a greater amount of sulfate being produced prior to reaching acidity (which is equivalent to a greater amount of acidity being neutralized). These results may be explained by the more effective washing in the shaken cell, compared to conventional cells. Readily dissolved and dissolved species, such as iron, may play a role by promoting oxidation within microenvironments even if the bulk pH is neutral.

The more thorough washing of dissolved species could also influence the availability of NP by preventing the formation of metal coatings on carbonates. In the case of the NP column tests, washing with acidic water would have reduced the amount of iron precipitate formed and allowed for a much greater depletion of NP prior to the leachate becoming acid. Once sufficient reaction product precipitated to limit neutralization, the leachate could become acidic. The HC-2 test column, which saw the precipitation front reach the bottom of the column at the same time as the leachate became acidic, supports this explanation. Therefore, protocols that use excessive washing conditions to remove dissolved

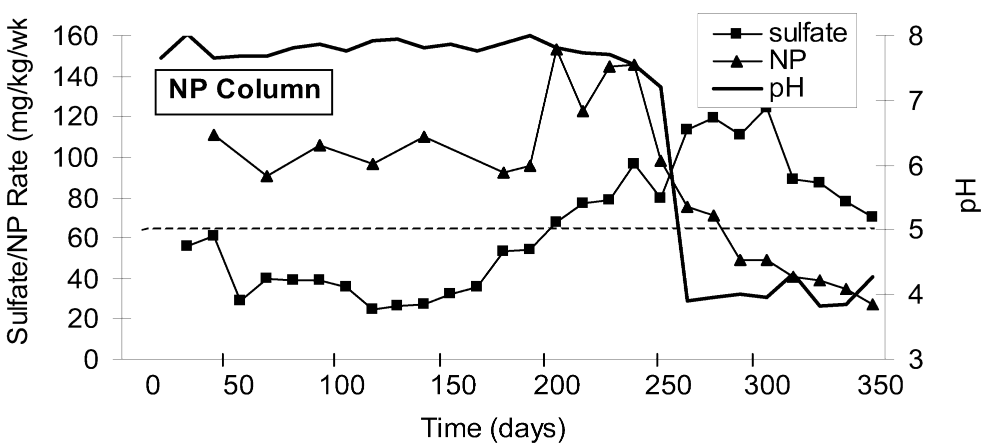

Figure 4. Sulfate release, NP depletion, and pH over time for the NP column containing HC-2 with average sulfate release rate (dashed line) from the five cycles prior to pH dropping to less than 6 in the standard cell test

Table 6. NP depletion and sulfate release when leachate became acidic for sample HC-2

Kinetic Test Type	Time to pH < 6.0 (days)	Cumulative NP Depletion (mg/kg)	(mg/kg/d)	NP Depleted (%)	Cumulative Sulfate Released (mg/kg)	(mg/kg/d)	Sulfur Depleted (%)
Standard	209	3057	14.6	25.7	2336	11.2	1.96
Tall	209	3609	17.3	30.3	2458	11.8	2.06
Shaken	328	3971	12.1	33.4	3161	9.6	2.65
NP Column	227	7919	34.9	66.5	2796	12.3	2.35

species can cause a reduction in the estimate of the sulfide oxidation rate and can overestimate the available NP.

Conclusions and Recommendations

Kinetic tests were conducted on 3 different samples to examine the effects of humidity cell protocol variables on rates of weathering. ABA results indicated that all samples had a strong potential to generate ARD although only one sample became acidic during the study. Five different test protocols (standard humidity cells, non-aerated cells, tall cells, shaken cells, and NP columns) were evaluated.

With the non-aerated cell protocol, sulfate release over time was less erratic and approached stabilization earlier than the standard cells. Air is not actively pumped through the cell for this protocol but oxygen is permitted access to the top and bottom of the sample. Reaction rates from the non-aerated cell were found to be similar to those obtained from the standard humidity cell. An advantage of non-aeration is that the operating condition is easily reproduced. The relatively constant moisture content is also considered to represent natural conditions more closely than the conditions created by dry/wet cycles. Therefore, a non-aerated cell protocol is considered to provide more repeatable and representative reaction rates than the standard humidity cell.

Trickle or flood leaching of kinetic tests is recommended since these protocols are considered to be the least disruptive to the oxidizing environment. White and Lapakko (2000) found that flood leaching produces comparable results to trickle leaching. Although the shaken cell protocol removes weathering products more effectively, the weathering rates appear to be affected by particle segregation and by changes to the oxidizing micro-environment. Test results also demonstrate that the volume of rinse water used during leaching must be sufficient to remove the weathering products, but must not be excessive. Excessive rinsing can influence the dissolution rate of the carbonate minerals (Day et al. 1997; Frostad et al. 2000a; Li 2000) and may overestimate the neutralization depletion rate that would be realized under field conditions.

Increasing the rate of NP depletion is not recommended as a method to calculate time to acidity. NP column tests showed that a high addition of acidic water did not decrease the time to acidity yet succeeded in increasing the rate of NP depletion relative to acid generation. At the point of acidity, the NP column indicated that 67% of the NP was available for neutralization, while other tests indicated that only 26-33% was available. Consequently, estimates of available NP obtained from laboratory tests that have used acidified water to accelerate the NP depletion rate may not be good indicators of time to acidity in the field.

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