

Investigation of the Humidity Cell Testing Procedure for Prediction of Acid Rock Drainage

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Abstract

Standard kinetic testing using humidity cells is frequently used to determine the potential for mining rock material to form acid rock drainage. This paper describes an investigation into that procedure that may help explain some of the variation in the results. Additional instrumentation was used that provided continuous monitoring of the interior temperature of the waste rock sample in order to measure thermal effects and water evaporation. Several humidity cells were operated under different operating conditions such as temperature, particle size distribution, and geochemical composition. The temperature of the sample during the drying phase is not constant but decreases as much as 12°C, depending on the air flow rate, the water saturation and the particle size distribution of the sample. Samples with more fines took much longer to dry. Duplicate cells showed that, for the most part, the data were reproducible.

Key words: pyrite oxidation, evaporation, water quality, geochemistry, air-drying of porous media, acidic drainage

Introduction

Prediction of ARD is difficult because it requires knowledge of many rock pile properties such as its acid producing potential, geochemical reactions and kinetics, permeability characteristics, specific reactive surface areas, etc. Usually most of these rock properties are unknown for any given mining rock pile but an estimation of these properties can be obtained by running some standard tests. Humidity cell testing is one of the best methods to measure not only the potential to generate acid but the geochemical kinetics of the rock material as well. An ASTM standard humidity cell testing procedure was published in 1996 and then updated several times (ASTM, 2001; ASTM 2007). There are a number of papers on humidity cell testing for the prediction of acid rock drainage (White and Lapakko, 2000; Lapakko, 1994; Lapakko, 2000; Lapakko, 2003; Mendez-Ortiz, 2007; Morin, 1999; Morin, 1996; Morin, 2000; Howell, 2006; Frostad, 2002; Price, 1997) and, while many of these follow the ASTM procedure, several have developed variations of the method to accommodate their own particular objective.

The main advantages of humidity cell testing are: 1) testing can be conducted using actual rock material under controlled environments over long periods of time; 2) reaction kinetics can be obtained under those same environments; and 3) leaching of metals can be evaluated (ASTM, 1996). The drawbacks are that the testing time is lengthy, expensive and, the extrapolation to predict ARD in the field is uncertain. It is usual for humidity cell tests to continue for several months or even more than one or two years until stabilization is reached. The current ASTM procedure does not prescribe a specific test-duration period, however Price *et al.* (1997) recommends a minimum of 40 weeks. Morin and Hutt (1998) presented compelling evidence showing that in many cases even 40 weeks is insufficient and that the test should be run until stabilized rates are obtained.

Gonzalez-Sandoval *et al.* (2009) studied the effect of air flow and cycle duration on humidity cell testing leachate quality by using a factorial experimental design. They found that cycle duration did not significantly affect pH but higher air flow rates delayed acidification due to water loss. Ardaue *et al.* (2009) studied the limitation of short leaching time in which leach water does not attain equilibrium with the solid phase. But when it comes to field leach water there is usually enough time to come to equilibrium with the solid phases. The time period and number of cells are important factors in humidity cell testing to get statistically meaningful results.

Lapakko and Berndt (2009) studied the effect of differences in test design on drainage quality especially the effect of evaporation and tailings depth. They conducted experiments at three different conditions: 1) uncovered small humidity cell; 2) covered small humidity cell; and 3) covered large humidity cell. They found higher oxidation in the covered small humidity cell compared to the uncovered small cell and the covered large cell due to wetting conditions and higher oxygen diffusion. Dissolution rates are usually measured on individual and pure minerals and reported in terms of unit surface area. The challenge with this approach is that rock pile material is composed of many different minerals in a complex matrix and determining the reactive surface area of each mineral is very difficult.

Lapakko and Antonson (2003) discuss the differences in pyrite oxidation rates determined using humidity cell testing from those measured in the field and in the literature using pure pyrite crystals. Pyrite reaction rates in humidity cells were determined using exposed surface area and sulfate released in drainage. Pyrite oxidation rates are in agreement with literature pure pyrite values when pH is >6 but if pH is around 3-5 then the oxidation rate is 2-4 times greater than the literature value. They also suggest that other than abiotic oxidation by oxygen the other oxidation reaction with Fe^{+3} may be more important when it comes to lowering pH.

Oxygen transport is very important and thus it is necessary to make sure the cells are air-saturated during testing. Pretreatment of the rock sample will also avoid some of the problems related to passivation of mineral surfaces and secondary minerals interference. But in the field, passivation of minerals from secondary minerals is present. So by pretreatment, laboratory weathering rates will tend to be higher than field rates. Hakkouet *et al.* (2008) compared the results from the humidity cell and the weathering cell (uses a thin layer of sample and more flushing-drying cycles compared to a humidity cell) and found that the two cell methods are relatively close. They also studied the effect of particle size distribution (PSD) on leachate data. Fine tailings produce higher concentrations of sulfate and acidity compared to coarse tailings but the pollution potential is higher in coarse tailings due to higher transport of air and water.

Objectives

The goal of this project was to investigate the humidity cell procedure further by collecting additional data during the weekly cycle process and for three types of media –glass beads, ground quartz and a field rock sample. Specific objectives were to (1) measure temperature changes during the drying cycle, (2) determine the effect of fines (PSD) on leachate quality over time, (3) run tests at elevated temperatures, and (4) run tests using pH 3.0 synthetic water as the leaching liquid. This additional information would be helpful in understanding humidity cell results, their variability and their application towards predicting leachate quality in the field.

Experimental Equipment and Methods

The humidity cell experimental procedure in this study generally followed the ASTM procedure (2007) except that additional instrumentation was added to determine more detailed information during the weekly cycles and over the testing period. Basic humidity-cell components are described in D5744-07 (ASTM, 2007). Our modifications included adding the following:

- Temperature probes (Omega PR-17 RTD) at specific heights within the humidity cell
- Pressure transducers (Omega RX-170) to record the pressure drop across the cell during the drying phase and as a function of air flow rate
- Heaters to conduct humidity cell tests at elevated temperatures. The heaters were a combination of custom-made band heaters for each cell and a filament heater to heat the incoming air flow. The heaters were controlled by a Chromalox™ 2110 controller interfaced with a computer using Specview™ software
- Mass flow controllers (Omega FMA-3300) to control the volumetric air flow to each cell.

Calibrations were conducted according to established procedures for the temperature probes, pressure transducers and mass flow controllers. All data were continuously recorded using an OPTO-22™ data acquisition system. Humidity cell testing was done for one year. Approximately one kilogram of sample was charged to each cell. The modified testing procedure was divided into three phases: 1) leach phase; 2) drying phase; and 3) stationary phase. In the leach phase, 500 ml of deionized water (or synthetic water) was trickled from a separatory funnel to the top of the cell with the cell's bottom valve closed. The rock sample was soaked for 1 hour after which the leachate was collected in an Erlenmeyer flask for 2 hours or more. The drying phase started on the second day by pumping dry air (1 L/min) from the bottom for 3 days. After drying, the humidity cell was left alone for the remaining 3 days.

Leachate collected every week was divided into four subvolumes in which one of the four subvolumes was unfiltered. Filtered subvolumes were filtered through a 0.2 micron filter using a vacuum pump. The unfiltered subvolume was used to analyze temperature, pH, Eh, and conductivity. One filtered subvolume was measured for calcium, magnesium, sodium, aluminum, manganese, potassium, total iron and silica and was preserved using very pure nitric acid. A second filtered subvolume was measured for fluoride and sulfate and not preserved. The final filtered subvolume was used to measure Fe^{+2} immediately after collection and was also not preserved with acid. Cell weights before and after the drip-trickle leach and cell weights before the dry-air portion of each weekly cycle were taken.

A Beckman pH meter (300 series) was used to measure pH with an accuracy of ± 0.05 at 25°C . Conductivity was measured using an Orion conductivity meter (Model 150) with a microsiemens scale. The oxidation-reduction potential was measured using an Eh probe and a Chemcadet controller. Metal ions were measured by outside laboratories using an inductively coupled plasma spectrometer (ICP-MS). Fe^{+2} was measured using a flow injection instrument (FIA lab Inc, model 2500). Fe^{+3} was determined as the difference between the total iron measurement and the Fe^{+2} measurement. Sulfate and fluoride ions were determined using ion chromatography. Leachate was filtered and the filter paper weighed in order to measure the amount of fines produced each week by those cells. This was done for the other cells as well. Particle size distributions were measured for the field sample with and without fines. Particle size distributions were measured of the air-dried samples using 12 different-sized meshes at the beginning and end of the testing period using a dry-sieving process.

Sample description and preparation

Three types of materials were used in these experiments – uniform glass beads, a pure quartz sample and a field sample. The uniform glass beads were from Fisher-Scientific (Catalog No. 11312C) and were 5 mm in diameter. Properties of the duplicate glass bead cells are given in Table 1. Note the difference in initial water saturation for the two types of leaching – the flooding technique and the drip-trickle technique. Higher saturations appear to be associated with the flooding technique, probably due to better water distribution and elimination of air pockets.

The pure quartz sample came from a crystal and the field sample was from a mine in New Mexico. Both the field sample and the quartz sample were crushed to 100% passing through a 6.3 mm screen and used in the twelve humidity cells for this study (see Table 2 for conditions). The field sample was split for all eight of its humidity cells using a conventional splitter. The original sample contained a large amount of fines and there was a concern that these fines would prevent drainage of leachate over time. Thus, a decision was made to remove most of the fines from several of the samples before humidity cell testing. The fines were removed from the original sample with a wet sieving technique using an 85 mesh (175 μm) screen. But even after removing the fines in this manner, a small fraction of fines were still present due to fines sticking to larger particles. The amount of fines removed from the original sample varied from 14.8-21.8wt %. The field sample with the minus 85-mesh fines removed was loaded into cells, G1, G2, G19, G20, G25 and G26. Fines were not removed from samples placed in two of the cells (G27 and G28) and these cells were tested without a felt pad at the bottom of the cell. Two control cells used a

crushed pure quartz sample made with the same PSD as the other samples (G9 and G10) and two control cells used glass beads (GB1 and GB9).

Each cell was initially charged with 1 kg of sample. Cells G1 and G2 used synthetic water based on the composition of the first wash water of the field sample (Table 3). As shown in Table 2 two of the samples were studied at a higher temperature.

Table 1. Glass bead packed bed properties for duplicate cells GB1 and GB9

	GB1	GB9
Weight of glass beads, g	1374±0.1	1370±0.1
Height, cm	11.0±0.1	11.6±0.1
Porosity (unitless)	0.350±0.001	0.35±0.001
Initial water saturation, S_w flooding technique (unitless)	0.170±0.007	0.160±0.019
Initial saturation, S_w Drip-Trickle technique	0.062±0.017	0.067±0.006

Table2. Operating conditions for the humidity cells used in this study. Eight of the cells contained rock material from one field sample while two control samples were used - pure quartz and uniform glass beads.

Sample	Duplicate Cell Numbers	Temp	Leach Water	Fines Removed
Field Sample	G1 and G2	RT*	Synthetic	Yes
Field Sample	G19 and G20	50C	DDH2O	Yes
Field Sample	G25 and G26	RT	DDH2O	Yes
Field Sample	G27 and G28	RT	DDH2O	No
Control Sample Pure Quartz	G9 and G10	RT	DDH2O	No
Control Sample Glass Beads – 5 mm	GB1 and GB9	RT	DDH2O	No

*room temperature

Table 3 Composition of the Synthetic Water used in Cells G1 and G2.

Species	pH	Fe	Ca	Na	Mg	Al	K	F	NH ₃	SO ₄
mg/l(except pH)	3.0	35.7	250	2.58	100	98	2.9	7	1.36	1608

Mineralogy before Testing

The mineralogy of the field sample was determined by personnel at New Mexico Tech (McLemore, 2009a) and the results are given in Table 4. Note that the difference in mineralogy between the fines and the original sample particularly for K-feldspar, plagioclase, illite/sericite, epidote and jarosite. It is interesting that potassium feldspar was not detected in the fines and epidote was significantly present in the fines. Also, gypsum and jarosite seem to be more present in the fines, probably indicating less adhesion of these particles to the larger particles than other minerals. The differences could also be due to small mineral inclusions found in the original field sample that were released upon crushing (McLemore, 2009b). The error in the weight percent values depends somewhat on the mineral but generally 2-4% is common.

Table4 Mineralogical analysis for the field sample before testing, wt. %(McLemore, 2009b)

Mineral:	Original sample	Fines (-85 mesh)	Sample without Fines
quartz	29	23	30
K-feldspar	22	nd	26
plagioclase	19	26	19
illite/ sericite	15	22	13
chlorite	4	6	4
smectite	2	1	1
kaolinite	2	1	1
epidote	0.01	11	nd
Fe oxides	3	0.2	3
rutile	0.4	0.6	0.4
apatite	0.5	0.2	0.3
pyrite	0.9	0.3	1
calcite	0.2	0.1	0.3
gypsum	1.1	3	1
Jarosite	0.4	5	nd
TOTAL	99.51	99.4	100

nd = not detected

Results and Discussion

Drying Cycle

Temperature profiles for one of the glass bead cells, GB1, are shown in Figure 1 for three different air flow rates. Data for the duplicate cell, GB9, were very similar. As shown in Figure 1 temperature decreases as drying starts, reaches a plateau and then increases as water saturation approaches zero. RTD 16 is the inlet air probe which is inserted in the bottom compartment of the cell, below the packed bed. The temperature of this probe is not constant due to some water remaining in the bottom of the cell and/or the dripping of water from the rock sample during testing. This is essentially equivalent to a “wet-bulb” temperature used in psychrometric charts. There is an apparent movement of some water downward through the cell due to diffusion. Since the amount of interstitial water that moves down the cell varies somewhat from run to run, the temperature profile will also vary as shown in Figure 1. The “dry-bulb” air temperature entering the cells was also measured using RTD-14 and, as shown, it remained essentially constant during the drying phase.

At the bottom most probe (RTD17), temperature decreases rapidly compared to the others. This is due to a higher saturation compared to the top portion. The saturation in the cell is not uniform after draining and the saturation at the bottom is always higher due to gravitation effects. The plateau region indicates a relatively steady-state evaporation situation. Once the inlet air loses its humidity and the “wet-bulb” temperature starts to increase, there is a drop in temperature for all RTD probes indicating a faster evaporation rate. After essentially all the water is removed, temperatures rise to the normal inlet air temperature. Air flow rate definitely affects evaporation and the temperature change can be as high as 12°C, at least for this packed bed of uniform glass beads.

The temperatures in the quartz control cell were relatively similar to those in the glass bead cell at the same air flow rate. However, in the case of the field sample, the cell never seemed to dry out completely

(temperature difference remains below zero) and took much longer to dry. We attribute this to the presence of clays and fines that are in the field sample but not in the other samples which hold water more tightly and make evaporation more difficult. This effect may be important in determining water distribution in a field rock pile when convective air currents are present.

Particle Size Distribution after testing

Three different PSDs were measured for each sample - that of the original sample, after removing fines (before testing) and after testing (top and bottom sections). Some field sample cells showed a significant difference in the distribution, with a trend towards smaller particles during testing, but, for others, there was not a large difference. In almost all cells, the percentage of coarse fraction decreased. This may be related to the degradation of some minerals, but the relation between this phenomenon and mineralogy was not clear. All PSDs were measured in the dry state where very fine particles tend to adhere to larger particles and are difficult to separate. Regardless, dry measurement of PSD provides a close estimate of PSD for these experiments.

Leaching Over Time

The concentrations of selected metals and anions in the humidity cell leachates for the field and quartz samples are plotted in Figures 2 and 3. Cells G9 and G10 are the quartz control cells and show the typical behavior of a pure quartz cell during humidity cell testing, pH relatively constant, around 6.0. Evidently there was some minor contamination of the sample since there are some measurable amounts of iron, calcium and sulfate initially present. For the field sample cells, pH is also relatively constant around 3.5 and appears to be rising in some of the cells. Metal and sulfate concentrations steadily fell by one to two orders of magnitude over 30 weeks and then stabilize between weeks 31 and 52 except for the G1, G2, G27 and G28 cells. Cells G1 and G2 of course were being leached with synthetic water (Table 3) and the values given are approximately those in the leach water. However, cells G27 and G28 show significantly different behavior, particularly for sulfate and calcium. Note that for these cells, concentrations never really stabilize even after 52 weeks. This may be due to the presence of more fines in these samples. The presence of these fines affects leaching rates since preferential flow patterns may develop among the courser fractions making diffusion the limiting transport mechanism for the finer fractions. Thus, it takes longer for the ions to be removed from the column.

The Ca/SO_4 molar ratio for cells G27 and G28 (with the higher gypsum concentrations) was close to 0.9 ± 0.1 (gypsum dissolution ratio=1.0) which shows that gypsum is perhaps the major source for both SO_4 and Ca in those cells. But when it comes to the other field cells the Ca/SO_4 molar ratio ranges from 0.4 to 0.8 indicating another significant mineral source for sulfate such as pyrite. However, the SO_4/Fe molar ratio for all cells was very high, between 100 and 1000. This indicates that pyrite oxidation is not the main SO_4 contributor and that the dissolution of gypsum might be the dominate sulfate source for these samples.

High Temperature Cells

Experimental data show that the increase in temperature to 50°C for the field sample cells G19 and G20 did not have a large effect on leachate data but there was a noticeable effect in some of the effluent ions (see Figure 4 for potassium). Due to a malfunctioning RTD probe, the electrical power to the high temperature cells (G19 and G20) was shut off after week 27. Cell 19 was heated to excessive temperatures at that time and was removed from the study. Effluent concentrations of several ions in the high temperature cell G20, such as aluminum, K, and Mg dropped significantly during that period and matched the effluent from the room temperature cells. However, effluent concentrations resumed to their higher values when the temperature returned to 50°C after week 37. This shows that temperature effects are reproducible. In general most of the ions in the higher temperature cells were at slightly higher effluent concentrations compared to room temperature humidity cells.

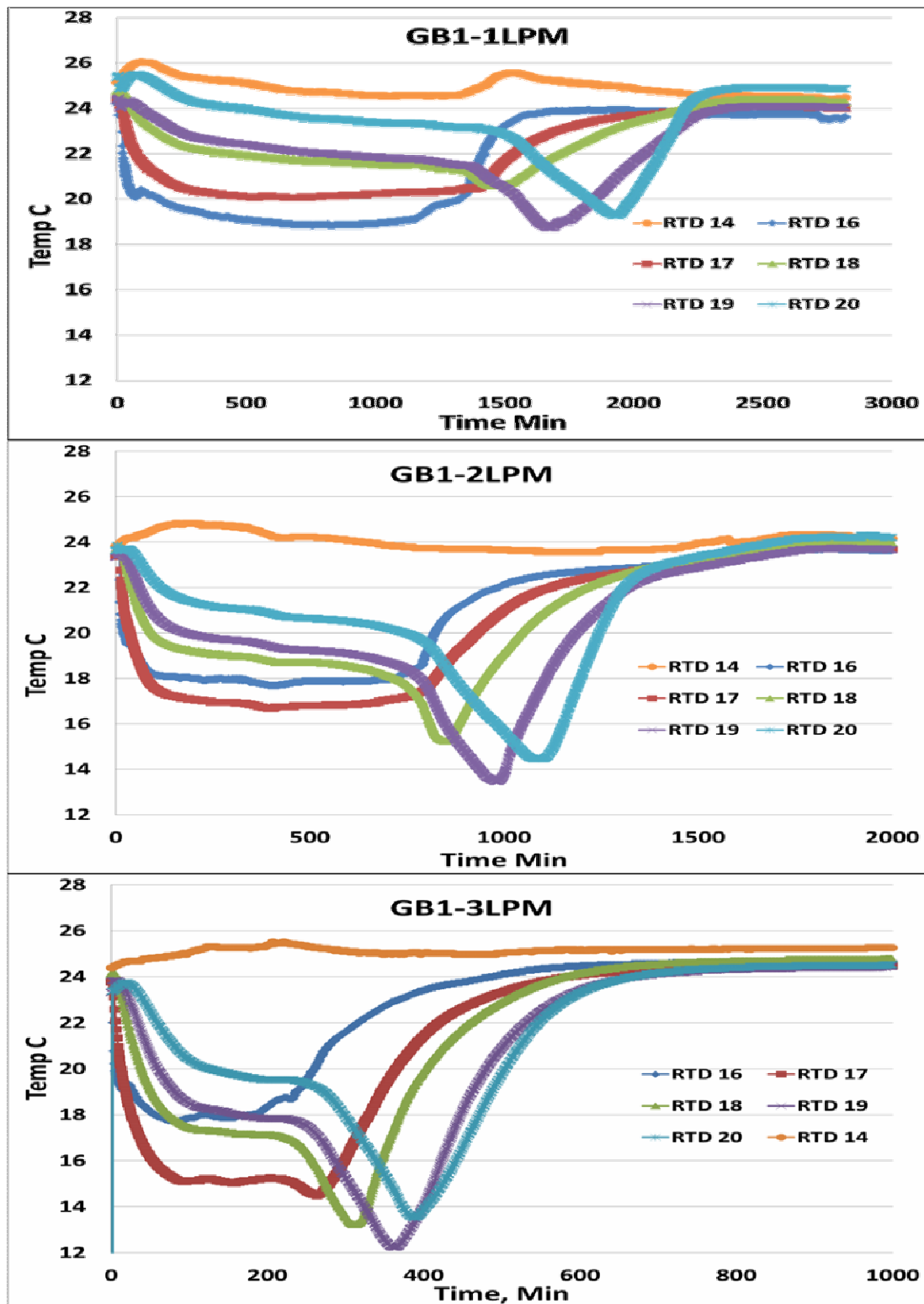


Figure 1. Drying profiles for GB1 with respect to air flow rate. Probe RTD14 measured the dry air temperature during testing. RTD 16 measured the air inlet temperature while probes RTD17-20 measured the temperature from the bottom of the rock sample to the top.

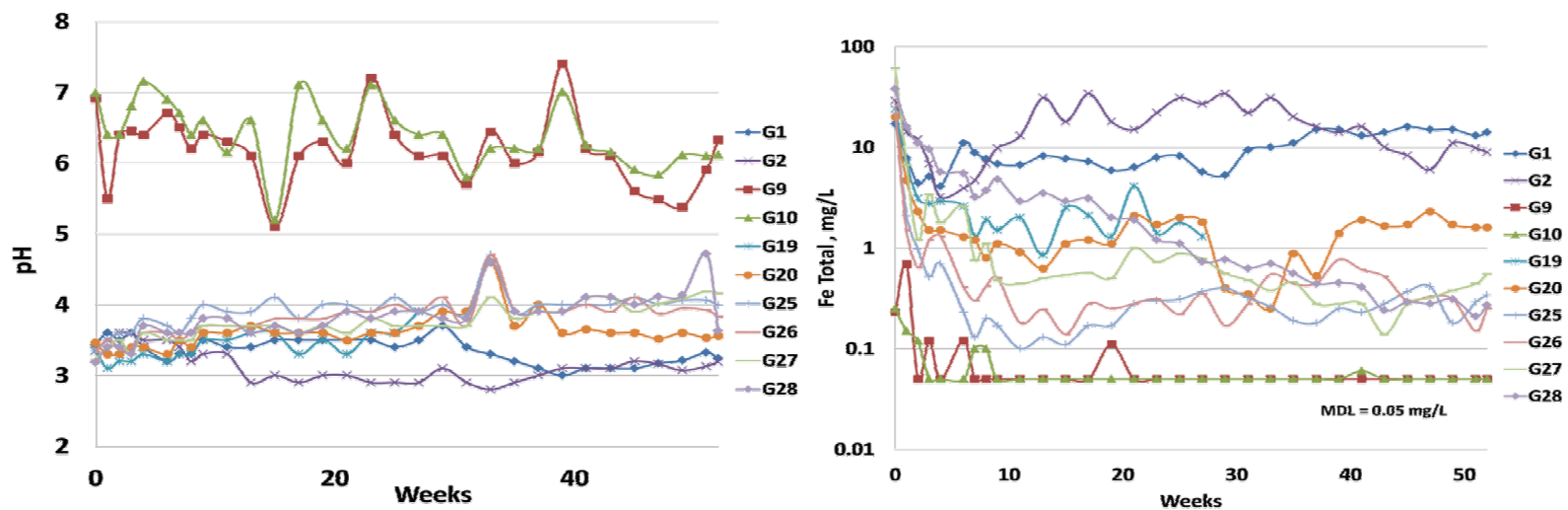


Figure 2. pH and total iron for all 8 field sample cells and the 2 control cells over time. Cells G9 and G10 are the control cells with pure quartz.

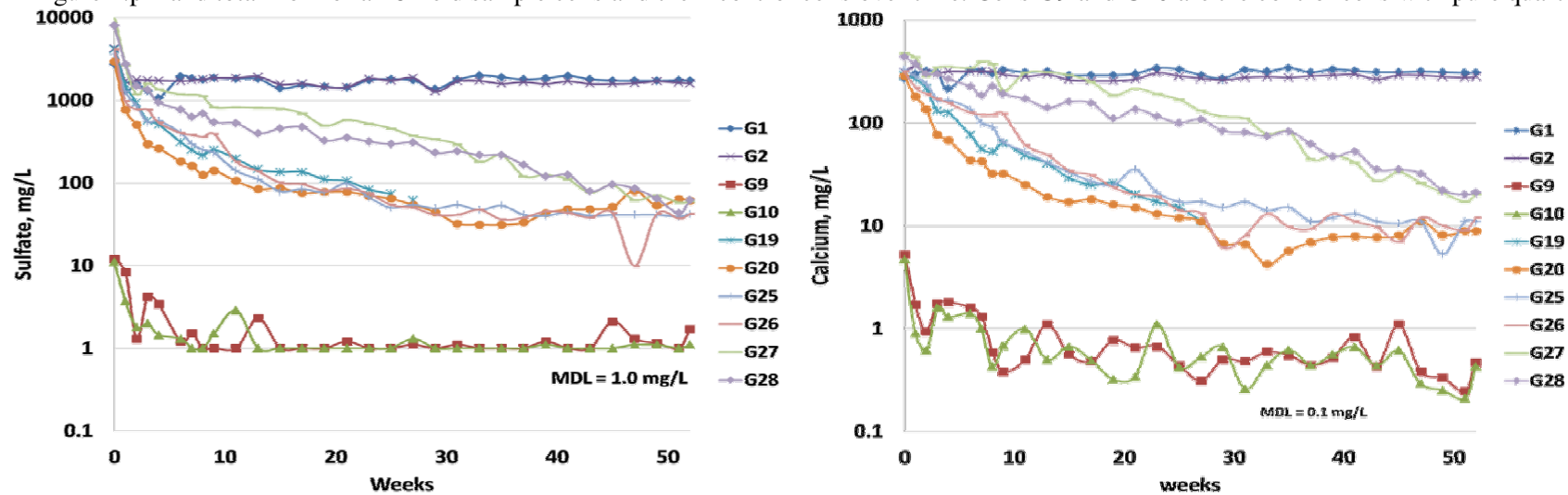


Figure 3. SO_4 and Ca as a function of weeks for all the field sample cells and control cells

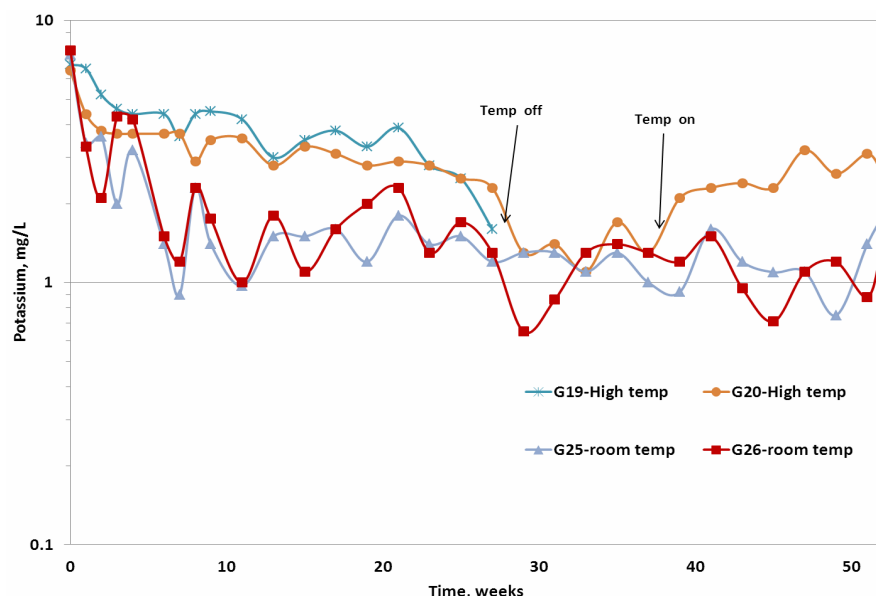


Figure-4: Comparison of potassium profiles between room temp and 50°C cells. The G19 cell was terminated prematurely at week 27. All four cells had the same initial field sample.

Mineralogical Analysis after Testing

All field samples were analyzed after 52 weeks of testing and the results are given in Table 5 below. As mentioned earlier, the contents of each humidity cell were divided into an upper section and a lower section in order to determine if the mineralogy might be different in those two sections over time. As shown in the table, there are some differences after 52 weeks of humidity cell testing, particularly for the samples retaining the fines which are somewhat confirmed by the leachate data. The only significant difference is that gypsum content decreased in all the field samples after testing compared to the amount before testing confirming the dissolution of that mineral found from the leaching data. No noticeable difference was seen in pyrite but there had to be some oxidation in order to maintain the low pH in these samples. The other source to lower pH could have been jarosite which was detected in the original sample but was not detected in the sample after fines were removed. Differences in the other minerals are slight and probably within experimental error.

Conclusions

It is shown that, during the normal dry-air-cycle of humidity cell testing, temperatures drop due to evaporation and the extent and duration depends on the properties of the rock sample. This can affect the leaching rates since mineral dissolution depends on temperature. The extent of drying also affects leaching rates. This should be an important consideration for predictive modeling studies. The presence of fines has been shown to affect the leaching rate. The time to reach stable values is much longer in the presence of fines since the higher surface areas increase dissolution rates. Dissolution of minerals at higher temperatures (50°C) is significant and reproducible. Replicate samples produced similar results.

Acknowledgement

We would like to thank Chevron Mining Inc. for their financial support and their valuable input during this project. We would also like to thank all the personnel at New Mexico Institute of Mining and Technology and New Mexico Bureau of Geology and Mineral Resources, especially Dr. Virginia McLemore, for the mineralogical analyses as well as the people on our staff, Danilo Garcia and Sally Kaiser, for their help with the humidity cell experiments.

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Acronyms

ARD	Acid Rock Drainage
ASTM	American Standard Testing Methods
DDH ₂ O	Deionized Distilled water
RTD	Resistance Temperature Detector
RT	Room Temperature

