

MEASURED RATES OF SULFIDE OXIDATION AND ACID NEUTRALIZATION IN KINETIC TESTS: STATISTICAL LESSONS FROM THE DATABASE¹

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Abstract: Kinetic tests involve repetitive oxidation and leaching cycles on a sample, and are frequently used for the prediction of acid generation and neutralization. The two most common types of kinetic tests are humidity cells and columns. Summary statistics from 250 humidity cells and 31 columns were compiled into a database in order to search for correlations and relationships among the data. The statistics included rates of sulfide oxidation and acid neutralization, highest and lowest pH values, particle-surface areas, and acid-base accounts (ABA), although all this information was not available for all kinetic tests. The rate of sulfide oxidation, as indicated by sulfate production, ranged from 0.036 to 5520 mg SO₄/kg of sample/week. In general, samples with the lowest rates of sulfate production yielded water with consistently neutral pH, and the samples with the highest rates yielded acidic water. However, there was a wide range between 10-1000 mg SO₄/kg/wk where pH was either neutral or acidic. The rate of sulfate production was not strongly correlated with Net Neutralization Potential or Net Potential Ratio, indicating these ABA parameters did not strongly reflect the potential rate of acid generation in a sample.

The rate of neutralization ranged from 1.43 to 5750 mg CaCO₃/kg/wk and did not correlate well with pH in the test water. However, a correlation was noted with the rate of sulfate production. In other words, the rate of neutralization is a response to the rate of sulfide oxidation, and the development of acidic or neutral conditions reflects the ratio of the rates rather than their absolute values. The ratio of the rates suggested that carbonate minerals accounted for most of the pH-neutral tests, but feldspar minerals and virtual absence of acid generation apparently accounted for the others.

Key Words: kinetic tests, humidity cells, sulfide oxidation rates, acid generation rates, acid neutralization rates, acidic mine drainage

1. Introduction

For sulfide-bearing materials, the potential for acid generation from sulfide oxidation exists when these materials are exposed to oxygen and moisture. In spite of sulfide oxidation and associated acid generation, acidic conditions may not develop if a sufficient quantity of acid-neutralizing minerals co-exists with the sulfide minerals (Ferguson and Erickson, 1987) and can react at a rate equal to, or greater than, the rate of acid generation (Ferguson and Morin, 1991).

The balance of acid-generating and acid-neutralizing minerals in a sample of rock or tailings can be determined through static tests, such as acid-base accounting (ABA), and reaction rates can be

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measured with laboratory- or field-based kinetic tests (e.g., British Columbia Acid Mine Drainage Task Force et al., 1989). All of these tests have weaknesses (e.g., Lawrence et al., 1989; Ferguson and Morin, 1991; Leaver and Bowman, 1994) and no one test alone can provide "the answer" to whether a sample will one day become acidic. In fact, Morin and Hutt (1994a) argued that the proper interpretation of one test's results depends on the results of all others, and thus all tests are required and should be interpreted together. This paper focusses on kinetic tests, but considers static-test data where available.

In general terms, a kinetic test involves continuous or repetitive oxidation and leaching of a sample with periodic rinses by water to remove the reaction products. Although international standard methods do not exist for kinetic tests, the most popular laboratory-based test appears to be humidity cells. The second most popular is apparently various types of columns, which are not always as well suited for determining reaction rates as explained below. In-field kinetic tests are not as common (Lapakko, 1994; Norecol, Dames, and Moore, 1994; Donovan and Ziemkiewicz, 1994), but have been recommended to calibrate laboratory tests (Price and Errington, 1994).

A humidity cell consists of an enclosed chamber holding a known weight of sample (e.g., Figure 1), often 0.5-1.0 kg and crushed if necessary to less than 0.25 inches. Air is continuously pumped into the cell, through or over the sample, and out an exhaust port. Either humid air is used continuously or alternated with dry air.

A "cycle" for a humidity cell, typically one week, is defined as the time between two consecutive rinses of the sample with water. The purpose of the rinse water is to remove all reaction products accumulated in the sample during a cycle (e.g., 100 mg SO₄ over 1 week). Based on the weight of the samples (e.g., 1 kg) and the volume of rinse water (e.g., 0.5 L), a reaction rate can then be calculated (200 mg SO₄/kg sample/week). If particle-surface area is known (e.g., 5 m²/kg), a surface-normalized rate can also be calculated (40 mg SO₄/m²/wk).

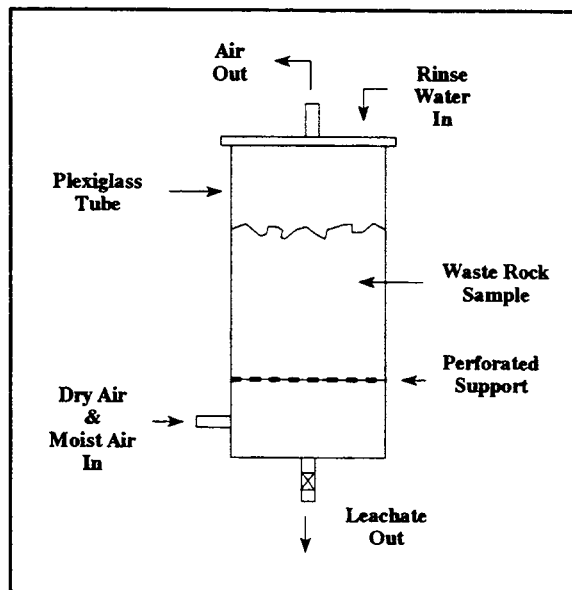


FIGURE 1. Example of a Humidity Cell.

Secondary-mineral precipitation can also occur in a cell if insufficient rinse water is added, preventing the removal of all of a cycle's reaction products. This biases the resulting rate towards unrealistically low values. Consequently, the appropriate volume of rinse water should be sufficiently large to ensure all concentrations are below solubility limits, but above analytical detection limits. Because rates of sulfide oxidation and acid generation are often defined through sulfate production rates, gypsum solubility is a particular concern (Ferguson and Morin, 1991). A few cells in this study were limited by gypsum solubility, placing an artificial maximum limit on their apparent rates of sulfide oxidation.

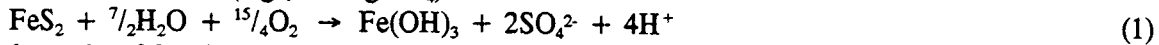
In some cases, secondary-mineral precipitation (Figure 2) is critical to water-chemistry predictions (Morin and Hutt, 1994b; Morin et al., 1995, these proceedings). However, humidity cells are not the optimum test for evaluating mineral precipitation expected under field conditions. Therefore, cells should focus on primary reaction rates and these rates are the focus of this paper.

Columns of various dimensions and heights holding various weights of samples are not always successful at measuring primary reaction rates. Many of the columns examined in this study appeared to be limited by gypsum precipitation. This often seemed to be the result of (1) adding or trickling a

relatively small amount of water into the columns at the end of, or during, a cycle and (2) incomplete rinsing of all particle surfaces due to channelling. When sulfide oxidation is high and gypsum precipitates, the column rinse water incorrectly provides a relatively low rate. At some point when sulfide oxidation falls below some critical value, the accumulated gypsum begins dissolving so that the rinse water incorrectly provides a relatively high rate.

2. Theory

According to the "standard" pyrite-oxidation equation, two moles of acidity (e.g., 200 mg as CaCO₃ equivalent) are generated with every one mole of sulfate (e.g., 96 mg SO₄):



This molar ratio of 2:1 (or weight ratio of 2.083:1) for acidity to sulfate is based on many assumptions about conditions within the sample which are not always applicable (Morin and Hutt, 1994a) and, in any case, are rarely measured. Also, measurements of acidity cannot be used to the rate of acid generation because some portion of original acidity has been neutralized, even in acidic tests. As a result, this study uses the rate of sulfate production as the direct indicator of the rate of acid generation. The rate of acid neutralization needed to successfully maintain near-neutral pH is then indicative of the ratio of acid generation to sulfate production (Section 4).

The rates of acid neutralization can be difficult to assess due to complexities such as mineralogy and initial pH prior to neutralization. For example, if pH were 2.0 around sulfide grains and neutralizing minerals raise the pH of the rinse water to 7.0, the measured alkalinity which begins to appear at pH 4.5 may represent only a small portion of total neutralization that had occurred.

A method for determining neutralization rates in cells and columns involves the sums of elemental molar concentrations, namely (Ca + Mg + Sr + Ba) and (Ca + Na/2 + K/2) (Ferguson and Morin, 1991; Morin and Hutt, 1994a). The first sum represents dissolution of carbonate minerals ("the carbonate sum"), and is often simplified to (Ca+Mg) because concentrations of Sr and Ba rarely exceed those of Ca and Mg. The second sum is attributed to dissolution of feldspar minerals ("the feldspar sum"). In several of the kinetic tests examined here, calcium was the dominant cation and thus both sums could be simplified further to simply (Ca). For pH-neutral kinetic tests, the molar ratio of Ca/SO₄ should theoretically be between 1.0 and 2.0 if carbonate minerals are dissolving solely in response to acid generation, and near or just below 1.0 if calcium-dominant feldspar is dissolving (Morin and Hutt, 1994a). For this study, the ratios of both sums against sulfate were examined where sufficient data were provided.

If it is not already clear from the discussions above, humidity cells and columns are not research-level instruments. They are empirical in nature, providing only bulk sample-size rates. Rigorous interpretations of such kinetic tests as particle-surface and microenvironmental reactions are not justified. Nevertheless, humidity cells are becoming a standard test for determining bulk reaction rates, and can play an important role in spite of sample size, as discussed below.

There have been some arguments regarding (1) the scaling of kinetic-test results to full-scale mine components and (2) the biased enhancement or acceleration of oxidation in kinetic tests. First, scaling

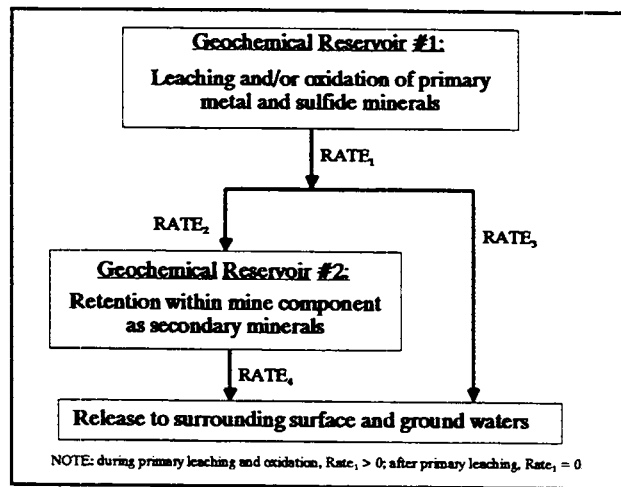


FIGURE 2. Primary and Secondary Geochemical Pathways Affecting Effluent Water Chemistry (from Morin et al., 1995).

is not necessary in some cases since fine material similar to a cell sample (less than 0.25 inches) often dominates bulk reaction rates in coarser rock dumps (Morin and Hutt, 1994b). These authors reported similar bulk rates (per kg) for five scales of kinetic tests ranging from 1 kg to 30,000 kg. Second, active movement of moist, or alternating dry and moist, air as well as periodic rinsing by water are not unusual in some rock dumps and mine walls (Morin et al., 1991; Morin et al., 1994a and b; Morin and Hutt, 1995, these proceedings). As a result, kinetic tests should not often enhance the rate of oxidation over that expected in the field. In fine-grained materials such as tailings, humidity cells can provide a reasonable oxidation rate representative of full exposure to the atmosphere and moisture. In cases where oxygen is expected to be limited by diffusion (Jambor and Blowes, 1994) or submergence (Morin, 1993), lower rates can be calculated from the maximum rate. Consequently, scaling of reaction rates and enhancement of oxidation rates are not considered problems with properly operated kinetic tests.

3. The Database

Published or publicly available summaries, files, or reports on proposed, operating, and closed mines were searched for kinetic-test data. Data were entered into a spreadsheet whenever the reaction rates normalized to the weight of the sample, the volume of rinse water, and the time interval of the cycle were given or could be calculated. This resulted in a total of 281 tests from 53 mines, of which 250 were humidity cells and 31 were columns. The tests were operated for an average of 29 weeks with a range of 6 to 159 weeks (Table 1). Also, 219 tests were from western Canada, 20 were from eastern Canada, and 42 were from other countries. In 127 cases, calcium and/or magnesium concentrations were not available, so the corresponding rate of neutralization and the elemental ratios (Section 2) could not be determined.

Because this study included comparisons of reaction rates to initial solid-phase sulfur levels, paste pH, and grain-surface area, the reports and files were also searched for static-test data and particle-size analyses. Static-test data were often found (249 tests), but particle-size analyses were rarer (91).

Ferguson and Morin (1991) defined a number of curves that represent changes in sulfate production through time. Many tests examined here produced a concave curve for cumulative sulfate, indicating the rates decreased through time until they stabilized for several years (e.g., Norecol, Dames, and Moore, 1994). For this reason, rates were calculated over the entire test as well as for the last few weeks (often 5 weeks) of the test (Table 1).

4. Results and Discussion

In order to identify relationships, scatterplots of one rate were plotted against another rate or one of the sample's physical or chemical characteristics. Correlations of two parameters cannot establish cause-and-effect relationships; however, knowledge of sulfide oxidation and acid neutralization can help to clarify the relationships. The following subsections discuss the correlations and relationships, or lack thereof.

4.1 General Observations

The 281 kinetic tests could be divided into two classes on the basis of pH of the rinse water: neutral and acidic. The detailed definition of the classes depends on the critical value of pH that separates them. A reasonable choice may be pH 6, because water-quality guidelines often call for pH above 6. However, distilled water passing over an inert sample could have a pH around 5.3 and would thus be strictly declared acidic, but not from the perspective of acidic drainage. A few tests apparently had low

TABLE 1

**RANGES AND MEANS OF RATES AND ACID-BASE PARAMETERS
FOR 281 KINETIC TESTS**

<u>Parameter</u>	<u>Count</u>	<u>Minimum</u>	<u>Maximum</u>	<u>Mean</u>
Physical and Static-Test Geochemical Characteristics				
Number of weeks	199	6	159	29
Surface area ¹	91	0.47	88.3	12.1
Total Sulfur, %S	233	0.006	37.2	5.03
Sulfide, %S	129	0.01	37.2	5.10
Neutralization Potential ²	249	-20.8	532	40.6
Carbonate NP ²	56	0	88	19.3
Paste pH	160	2.38	9.72	6.91
NNP ² (NP-%S*31.25)	249	-1110	+263	-109
NPR (NP/[%S*31.25])	249	-0.26	515.2	6.38
Lowest test pH	281	1.7	8.2	5.5
Highest test pH	281	2.6	9.1	7.0
Average Rates over Entire Test / Average Rates over Last Weeks				
SO ₄ Weight Rate ³	269/175	0.036/0.022	5516/3128	285/301
SO ₄ %S Rate ⁴	237/144	0.794/0.186	3380/1000	139/112
SO ₄ Surface Rate ⁵	87/81	0.042/0.034	2051/1980	320/267
NP (CaCO ₃) Weight Rate ³	154/144	1.43/0.919	5751/3261	187/127
NP (CaCO ₃) Surface Rate ⁵	80/75	0.379/0.169	321/200	73.0/17.7
Molar (Ca+Mg)/SO ₄	153/144	0.002/0.0017	205/85.9	4.95/4.32
Molar (Ca+Na/2+K/2)/SO ₄	20/20	0.16/0.12	1.68/2.21	0.96/0.916
¹ units are m ² /kg				
² units are tonnes CaCO ₃ equivalent / 1000 tonnes of sample				
³ units are mg of parameter / kg of sample / week				
⁴ units are mg of parameter / kg of sample / week / %S from ABA				
⁵ units are mg of parameter / m ² of particle-surface area of sample / week				

rates of acid generation and produced rinse water with a pH as low as 5.0. As a result, a pH of 5.0 was used here to separate tests of self-generated acidic and neutral conditions.

The lowest pH measured during a test, which often occurred towards the end, was used as the general indicator of whether the test could be simply labelled either acidic or neutral. Additionally, the highest measured pH, which often occurred near the beginning of a test, was used to determine if an acidic test was initially neutral. On this basis, there were 191 neutral and 56 acidic tests, plus an additional 34 tests that became acidic (considered part of the acidic class for a total of 90). If a cutoff pH of 6.0 were used instead, there would be 160 neutral and 121 acidic tests, with the acidic class including 52 tests that were initially neutral. At least half of the neutral tests were expected to eventually become acidic at later time (discussed below).

The comparison of the lowest measured pH to initial solid-phase sulfur showed that acidic conditions can be generated by sulfur levels as low as 0.1%S (Figure 3a). No clear relationship was noted between lowest pH and initial Neutralization Potential (NP, Figure 3b and Table 1) or Carbonate Neutralization Potential (not shown), probably because the initial NP was eventually depleted in some tests (Section 4.3) thereby allowing rinse pH to become acidic. Relative to Net Neutralization Potential (NNP, Table 1), all acidic cells (lowest pH < 5.0) had negative values except for one with a negligible positive value which was essentially zero (Figures 3c and 3d). Relative to Net Potential Ratio (NPR), all acidic cells had values less than 1.0 except one with a value close to 1.1 (Figure 4a). For both NNP and NPR, there were samples with values below 0.0 and 1.0, respectively, that were not acidic, but expected to become so later. This lag time to acidic conditions could be decades (Lapakko, 1994; Morin et al., submitted), but can be roughly estimated from kinetic-test data prior to the onset of acidification (Morin and Hutt, 1994a).

No correlation was noted between lowest pH and calculated particle-surface area (Figure 4b). Also, the surface area did not correlate well with either the rate of sulfide oxidation or acid neutralization, and is thus not examined further in this paper.

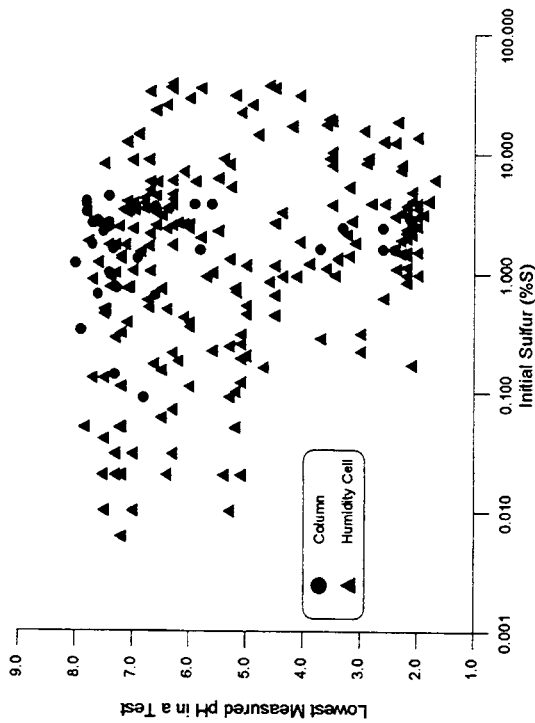
In general, samples generating the lowest acidic pH typically had the highest average rate of sulfate production (Figure 4c) and vice versa. Rates did not become tightly clustered until the rate exceeded 1000 mg/kg/wk and lowest pH was below 2.4. However, there was a large range from 10 to 1000 mg SO₄/kg/wk in which many acidic and neutral tests were located. As explained above for NNP and NPR, many of the neutral tests were expected to become acidic at some point in time when NP is depleted. On the other hand, the rate of NP depletion showed no clear relationship to lowest pH (Figure 4d). These rates are examined further in Sections 4.2 and 4.3.

A comparison of lowest pH to the carbonate ratio showed that many neutral samples had ratios clustered between 1.0 and 2.0 (Figure 5), indicative of carbonate-mineral dissolution (Section 2). A few neutral tests had ratios less than 1.0, suggesting feldspar dissolution was neutralizing the acidity. However, some tests with ratios between 1.0 and 2.0 were acidic, highlighting the importance of site-specific conditions in using the ratios (Morin and Hutt, 1994a). The carbonate ratios above 10 probably represented simple dissolution of neutralizing minerals into rinse water in the near absence of acid generation. This "preferential depletion" of NP caused simply by water movement can cause the onset of acidic conditions earlier than expected based only on the rate of acid generation, but is apparently only a major concern at the lowest rates of acid generation in this study (Section 4.2).

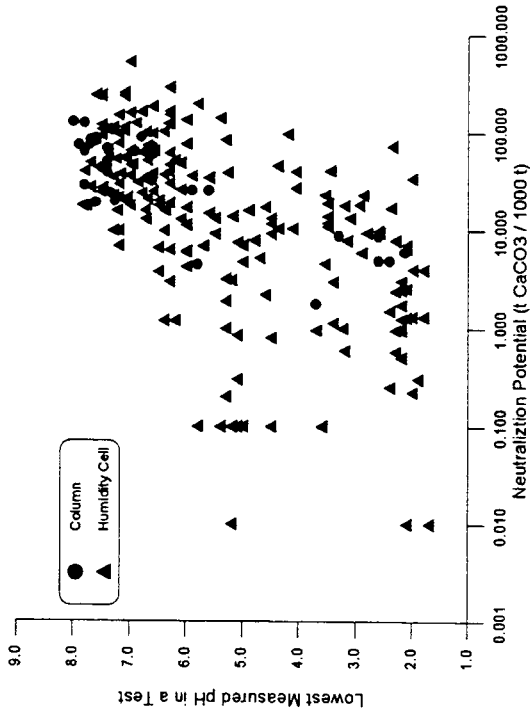
4.2 Rates of Sulfide Oxidation

As explained in Section 2, the rate of sulfate production is related to the rate of sulfide oxidation and acid generation, although sulfate production was limited in some tests by gypsum precipitation. Overall, the rates of sulfate production in the tests spanned over 5 orders of magnitude (Table 1). The comparison of sulfate production to initial solid-phase sulfur revealed a general trend of increasing rate with increasing sulfur (Figure 6a), although the rates typically ranged over two or more orders of

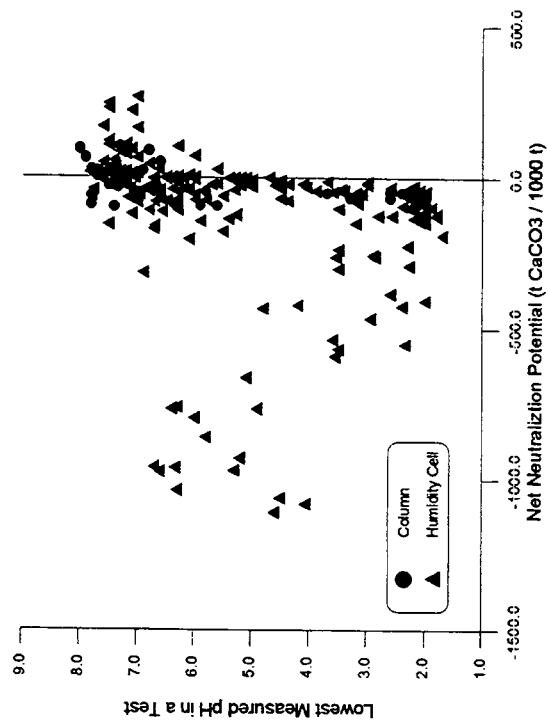
3a Lowest pH vs. Initial Sulfur



3b Lowest pH vs. Neutralization Potential



3c Lowest pH vs. Net Neutralization Potential



3d Lowest pH vs. Net Neutralization Potential

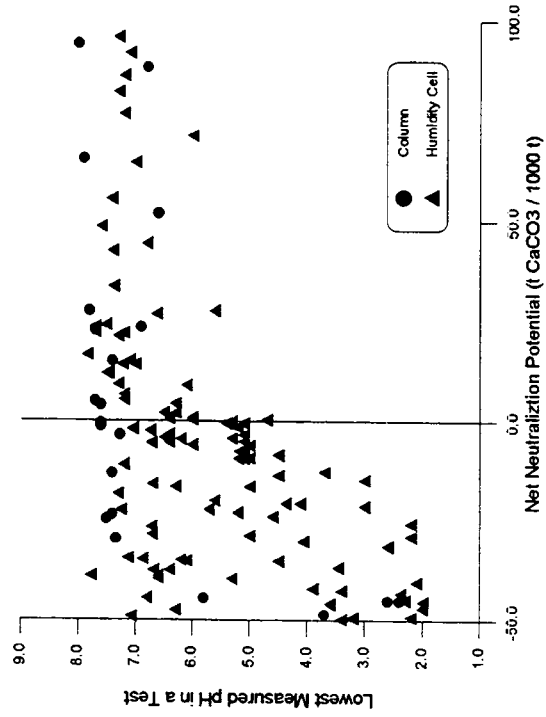
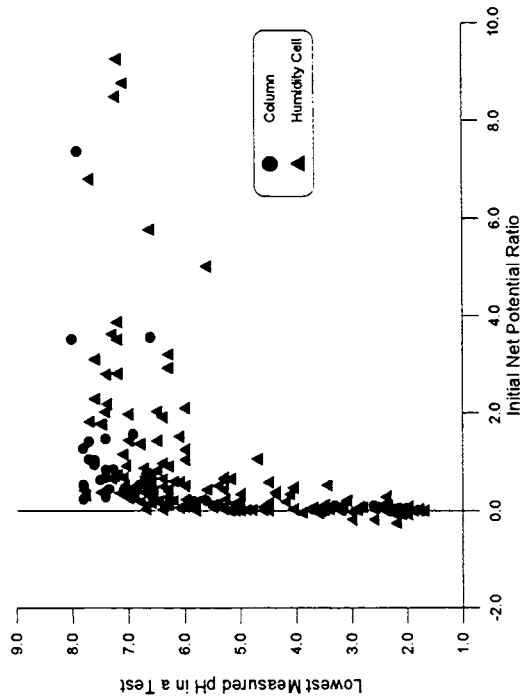
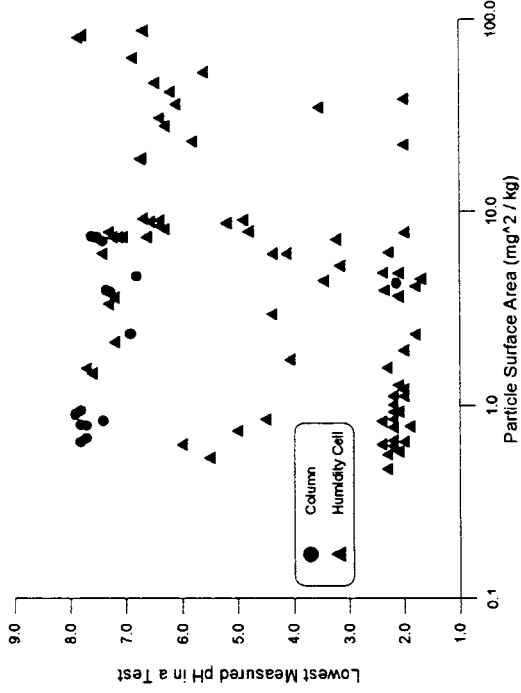


FIGURE 3: Lowest pH vs. (a) Initial Sulfur, (b) Initial Neutralization Potential, (c) and (d) Initial Net Neutralization Potential [different scales].

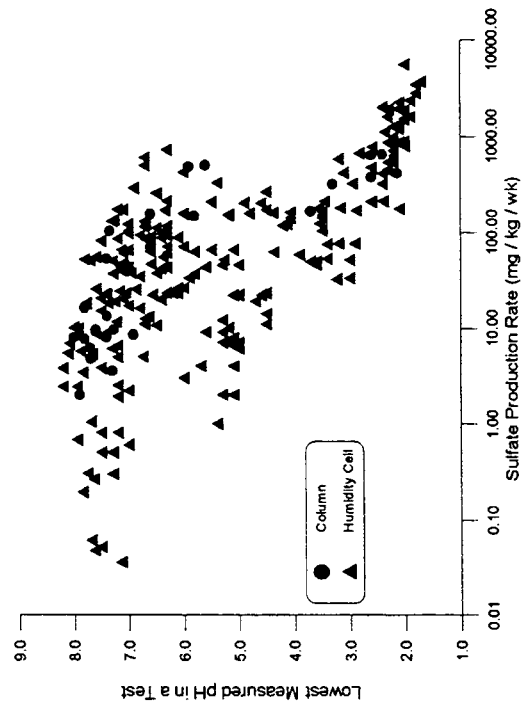
4a Lowest pH vs. Initial Net Potential Ratio



4b Lowest pH vs. Particle Surface Area



4c Lowest pH vs. Sulfate Production Rate



4d Lowest pH vs. NP Depletion Rate

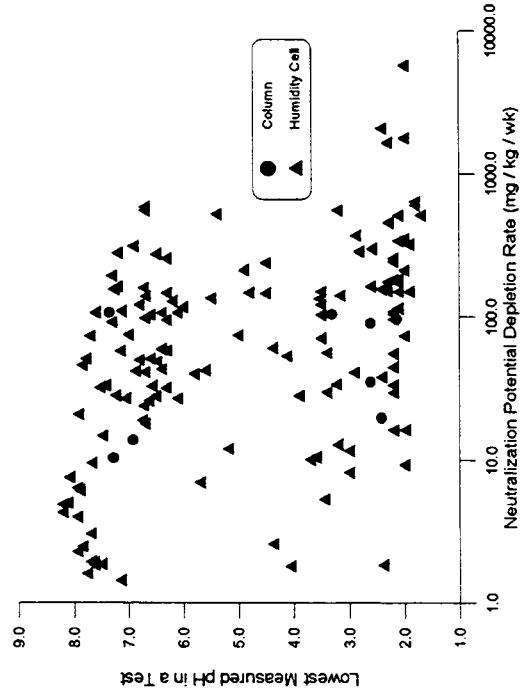


FIGURE 4: Lowest pH vs. (a) Initial Net Potential Ratio, (b) Surface Area, (c) Sulfate Production Rate, and (d) NP Depletion Rate.

magnitude at a particular amount of initial sulfur. Of special note, the sample with the highest measured rate did not have the highest sulfur content, but one of 13.61%S. This sample was of coal tailings. The next four highest rates contained only 4-6%S and were crushed crystalline rock. Therefore, there is little justification for the belief that the highest sulfur levels generate the highest rates of acidity. Similarly, the rates for some samples with sulfur levels above 20%S were comparable to those with sulfur as low as 0.1%S. Obviously, there are variables other than solid-phase sulfur levels, such as sulfur mineralogy and crystallinity, that affect the rate of sulfate production.

There was no correlation of sulfate production with initial NP (not shown). However, when NP and initial sulfur were combined into NNP and NPR (Table 1), general correlations with sulfate production were seen. For NNP (Figure 6b), only rates less than 1 mg SO₄/kg/wk were consistently correlated with positive values of NNP, whereas all rates above 500 mg SO₄/kg/wk correlated consistently with negative-NNP values. Notably, similar to initial sulfur, the highest rates above 500 mg SO₄/kg/wk did not have the most negative NNP values, but were preferentially clustered around -50 to -100 t CaCO₃/1000 t. As a result, a general belief that NNP reflects the intensity of acid generation is incorrect.

In contrast to NNP, NPR showed better correlation (Figure 6c) with the highest rates having the lowest ratios. However, even at NPR ratios close to zero, the rates of sulfate production extend almost over their full range. Therefore, there was little correlation between the rate of acid generation and NNP and NPR values.

The ratio between the rate of sulfate production and the rate of NP depletion should theoretically lie between 1:1 and 1:2 (1:2.083 by weight) during successful neutralization by carbonate minerals to pH > 6 (Section 2). As a result, there should be, and was, a straight-line relationship between the two rates for neutral tests (Figure 6d) with an error bar below the 1:1 line of roughly 0.3 log cycles (a factor of 2). The tests above the 1:1 line had a proportionally much higher rate of sulfate production and acid generation, and thus many were acidic despite a detectable rate of NP dissolution. In other words, some neutralization was occurring in many acidic cells but the rate was not sufficient to result in neutral rinse water. This is the reason that acidity in the rinse water cannot provide the rate of acid generation.

At sulfate-production rates below 1 mg SO₄/kg/wk, the rates of NP depletion did not fall below 1 mg CaCO₃/kg/wk. This was probably due to the preferential depletion of NP discussed in Section 4.1.

4.3 Rates of Neutralization

The rate of neutralization is synonymous with the rate at which Neutralization Potential (NP) in a sample is consumed and depleted. This rate spanned over 3 orders of magnitude in the database (Table 1).

The rate of NP depletion determines if and when an acid-generating sample will become acidic.

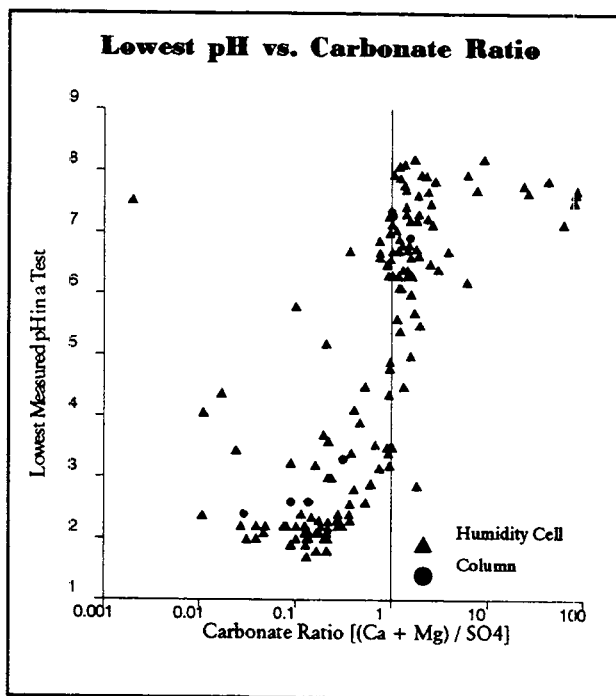
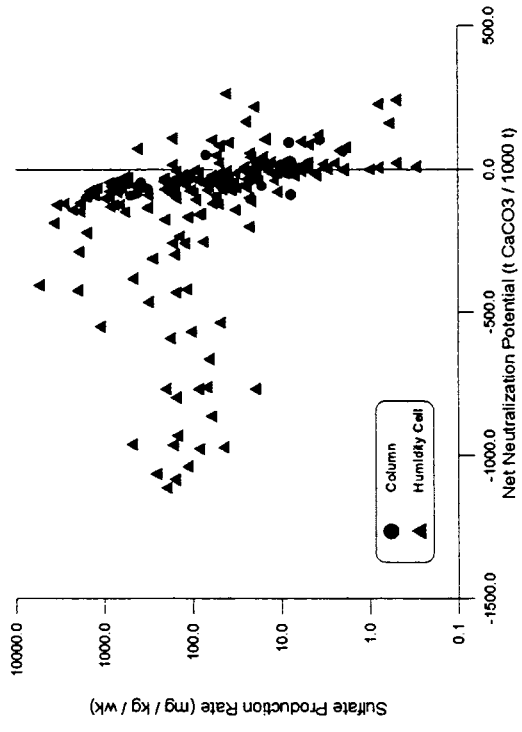
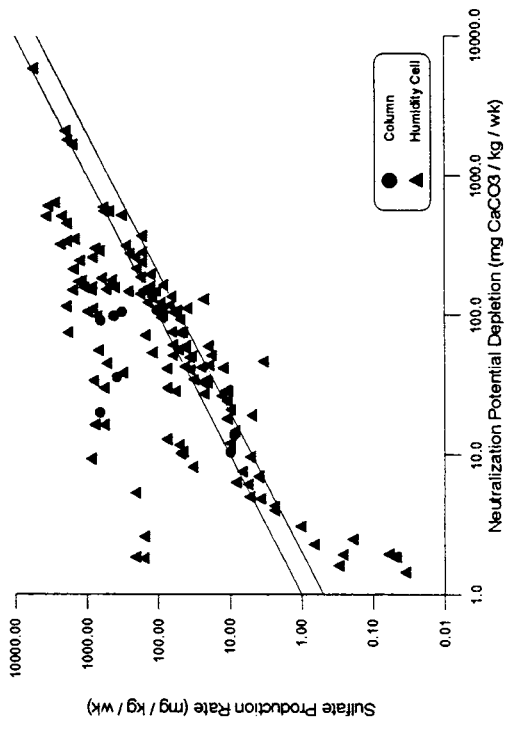


FIGURE 5. Lowest pH vs. Carbonate Ratio.

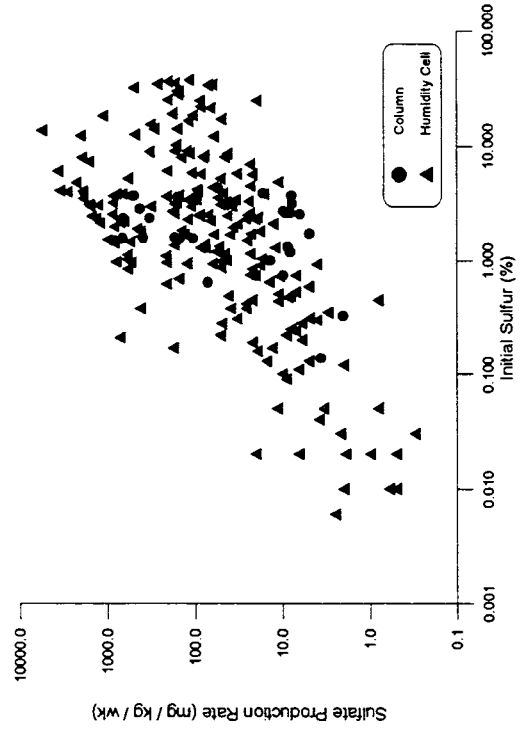
6b Sulfate Production Rate vs Net Neutralization Potential



6d Sulfate Production Rate vs NP Depletion



6a Sulfate Production Rate vs Initial Sulfur



6c Sulfate Production Rate vs Net Neutralization Potential

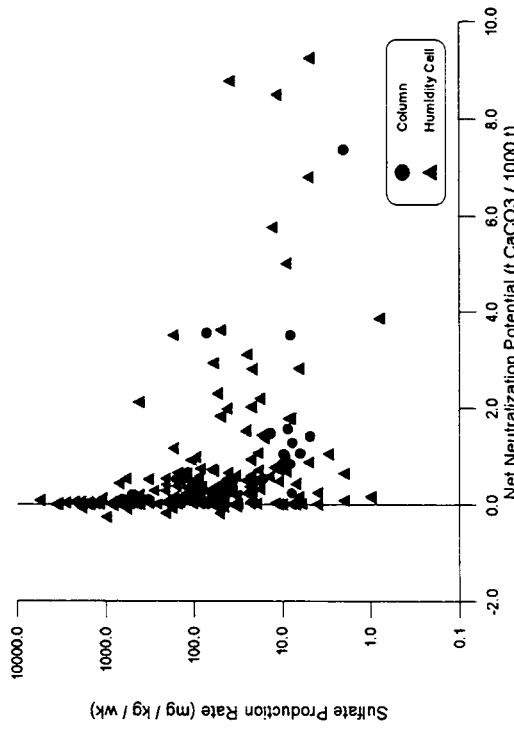


FIGURE 6: Sulfate Production Rate vs. (a) Initial Sulfur, (b) Net Neutralization Potential, (c) Net Neutralization Potential, (d) NP Depletion Ratio, and (e) NP Depletion Rate.

For example, the rate of NP depletion can exceed the rate of acid generation and yet indefinitely maintain neutral conditions, if the initial NPR in a sample is greater than the carbonate ratio (Section 2) measured during a kinetic test (Morin and Hutt, 1994a). In other words, the carbonate ratio from a test is basically the rate of NP depletion to the rate of sulfate production and thus must be roughly equal to, or greater than, the original ratio of NP to acid potential in a sample.

The rate of NP depletion is obviously a response to the rate of acid generation. Therefore, it is not surprising that there was a correlation between the rates, particularly when rinse pH was neutral (Figure 6d). This was discussed at the end of Section 4.2.

For the geochemical parameters of initial solid-phase sulfur, NP, Carbonate NP, and paste pH, there were no clear correlations noted. However, the trend of NP depletion rate with NNP and NPR (e.g. Figure 7) reflected the trend noted for the rate of sulfate production (Figures 5b and 5c). Again, this was the result of the correlation between the two rates.

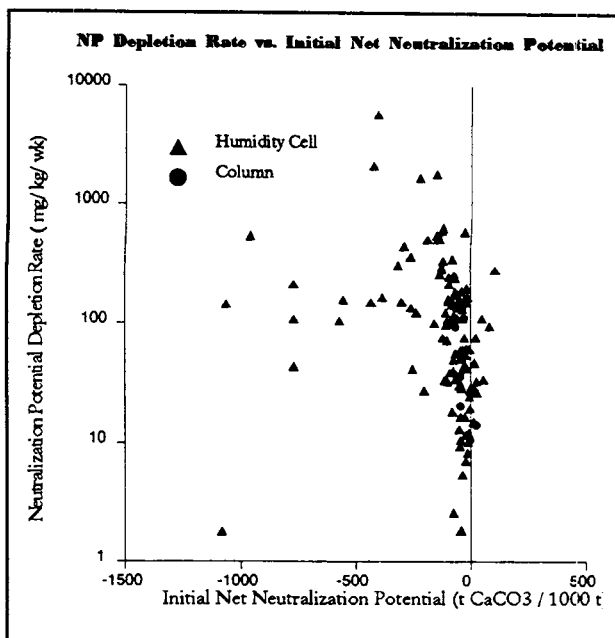


FIGURE 7. NP Depletion vs. Net Neutralization Potential.

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