SHORT-TERM ACID ROCK DRAINAGE CHARACTERISTICS DETERMINED BY PASTE pH AND KINETIC NAG TESTING: CYPRESS PROSPECT, NEW ZEALAND¹

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Abstract. The paste pH test (1 part solid: 2 parts water) is one method used to determine the acidic nature of a rock/soil sample. In conjunction with kinetic NAG testing a classification scheme has been developed for the Cypress Prospect within the Stockton coal mining region, West Coast, New Zealand. Samples having a paste pH of < 4.0 are considered potentially acid forming (PAF) and contain significant acidic sulfate salts (up to $30.1 \text{ kg H}_2SO_4/t$ equivalent) that will immediately produce acid upon exposure to water. Samples with a paste pH of 4.0 - 5.0 are considered PAF, but have a lower stored acidic salt content (up to 9.0 kg H_2SO_4/t equivalent). In the field the lithologies represented by both these rock types are likely to generate ARD immediately upon exposure to water. Circum-neutral paste pH values (> pH 5.0) for samples classified PAF indicated that they have a short-term acid neutralization capacity (ANC) that is greater than the readily available short-term acid generating capacity of the sample. This resulted in a time lag (2 - 356 minutes) prior to decrease to pH 4 in the kinetic NAG test. Samples having a paste pH > 6.0 typically produced a longer lag period than those with a paste pH of 5 - 6. As previous researchers have demonstrated this represents a lag period prior to the onset of laboratory acid rock drainage in larger column leach tests. These results have direct application to strategic mine planning at the proposed Cypress mine including separating waste rock into immediate acid generators (high management priority) from acid generators with a lag to acid formation (lower priority) and non-acid forming. Field validation of this classification system is still needed.

Additional Key Words: Acid Rock Drainage, Kaiata mudstone, New Zealand

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Introduction

Acid rock drainage (ARD) is the most significant environmental problem facing the global mining industry. For instance, there are over 200,000 ARD sites within the United States of America (Hochella et al., 1999); greater than 5,000 km of water-courses are polluted by contaminated drainage from abandoned mines in Europe (Younger, 2002); and Harries (1997) reported the presence of significant ARD within Australia, which has been compared to the problems facing the Canadian mining industry (Bell, 2003).

The overall acid generation process that produces ARD is associated with the oxidation of sulfide minerals. The ARD acid formation process is typically represented by the oxidation of pyrite (Equation 1). For every mole of pyrite oxidised, 4 moles of acidity are released.

$$FeS_2 + \frac{7}{2}H_2O + \frac{15}{4}O_2 \implies Fe(OH)_3 + 2H_2SO_4$$
 (1)

A variety of predictive tests are available to determine the acid-base account characteristics of a rock sample, which is a sum of the inherent acid generating and acid neutralizing reactions. The overall objectives of these test procedures are to determine aspects of the ARD evolution trend such as the overall acid nature, lag period to acid onset, ARD duration and geochemical acid-base kinetics (e.g., Fig. 1). Determining where on the ARD evolution trend a current ARD system is, and thus the duration of the remaining acid/metal load, is particularly important to operators and regulatory bodies. For instance, the peak ARD zone, identified by high sulfide oxidation and acid generation in excess of ANC is the period where effective active ARD management and mitigation procedures are required. A steadily decreasing sulfate load would indicate that the peak ARD load has passed, whereas an increasing sulfate load would suggest that the peak has yet to be achieved. Generally a decreasing SO_4^{-2} and metal load together with increasing pH values would suggest that eventually sulfide acidity generation rates will match the longer-term inherent acid neutralization rates of the sample usually derived from silicates. This paper investigates the front-end of the ARD evolution trend and uses geochemical tests to develop an improved understanding of any ARD lag period. Particularly we assess the paste pH test and conclusions that can be drawn after comparison to the kinetic NAG test.

Historically, the paste pH test has been considered a static test in that it provides no indication of reaction kinetics. However, as this paper will demonstrate, paste pH measurements are a good indicator of the immediate acid-base reactivity of a sample. This is demonstrated by comparison to other standard geochemical tests such as the kinetic NAG test (IWRI and EGi, 2002). Numerous soil-rock paste pH tests exist (e.g., Stevens and Carron, 1948; Ferrari and Magaldi, 1983; Meek, 1996; Miller et al., 1997; Price and Kwong, 1997; Morin and Hutt, 2001; IWRI and EGi, 2002; Cruywagen et al., 2003) using either pulverized or as received uncrushed sized materials. Combined with experimental tests such as the kinetic NAG test and the column leach test (IWRI and EGi, 2002) they can provide valuable information regarding lag time to ARD evolution.

Previously a strong correlation has been demonstrated for the time lag (in minutes) to acidic conditions in the kinetic NAG test compared to the time lag to acid generation in the column leach test (Miller et al., 1997). Furthermore, recent scale-up field trials using purpose constructed 500 tonne test pads (designed to ensure oxygen was not limiting) of screened (<300 mm) preferentially mined PAF material at the Freeport Grasberg mine, Papua Province,

Indonesia, suggested that column leach tests (of similar material) provided a good indication of field response to ARD generation (Miller et al., 2003). Thus any correlation between paste pH measurements, time lag prior to acidic conditions in the kinetic NAG test and thus the column leach test, may also be of relevance to the field.



Figure 1. Schematic ARD Evolution Trend for low carbonate - high pyrite waste rock. The plot shows simplified hypothetical acidity and ANC release rates in comparison to pH. Short-term ANC is derived from carbonates and near-surface high-energy sites on silicate minerals. Longer-term ANC is determined by stoichiometric silicate dissolution kinetics after carbonate exhaustion. Peak ARD zone is associated with sulfide mineral oxidation in excess of available ANC. The ARD recovery zone occurs near sulfide exhaustion when longer-term silicate ANC is in excess of acid generation. The ARD recovery zone can be extended by significant Fe(OH)₃ buffering, i.e., jarosite dissolution and subsequent Fe(OH)₃ precipitation and acid release. Fe(OH)₃ buffering can also occur during initial pH decrease.

Paste pH is a relatively rapid, simple method used to assess the readily available acidity/alkalinity of a sample and is easily conducted in the field. Because the test is of short duration (<12 hr) and non-vigorous (deionised water), only soluble salts and reactive minerals are assessed. Minerals typically assumed to be assessed by paste pH tests include acid generating sulfate salts such as melanterite, reactive sulfides such as greigite, and high surface area pyrite and carbonate. However, the results of Stevens and Carron (1948) suggested that other minerals can also influence the paste pH. They investigated the abrasion pH (similar to paste pH) of over 200 minerals. Results indicated that the abrasion pH values of several (Fe,Al)SO₄ minerals were very acidic, i.e., coquimbite pH 1; melanterite pH 2-3; alunogen pH 2; and jarosite pH 4-6. Minerals such as chlorite (pH 7-8), calcite (pH 8), vermiculite (pH 8-9),

dolomite (pH 10), and actinolite (pH 11) had much higher neutral to alkaline pH values. Meek (1996) reported that paste pH can be an indication of the amount of sulfide oxidation in the sample, which is also an indication of the weathering to which the sample has been subjected. Ferrari and Magaldi (1983) reported that the degree of weathering of a soil sample can be determined by the abrasion pH, which is influenced by the quantity of easily displaced ions (e.g., Na, K, Mg, Ca). In weathered soils, there are fewer easily exchanged cations compared to fresh rock that can be exchanged for H^+ ions and thus the abrasion pH is lower, although this is not usually below pH 5.0 (Ferrari and Magaldi, 1983).

As part of the paste pH test rock samples are crushed and ground increasing the highly reactive surface area for H^+ ion exchange with base cations and thus neutralization. To avoid the formation of these highly reactive surface areas Price and Kwong (1997) proposed the rinse pH test, which involved rinsing a sized un-pulverized sample with water and measuring the pH of this liquor. Comparison of the results for the two tests on similar sized samples (one pulverized only) indicated that paste pH measurements on pulverized rock can be 3 to 4 pH units higher for acidic samples than in the rinse pH test, although samples that had a rinse pH of 7.0 also had a paste pH of 7.0. This suggested that crushing increased the reactive surface area of carbonates and silicates, which increased the proton neutralization capacity of the sample.

Paste pH provides no indication of the sample's total capacity to generate acidity or alkalinity, but rather, provides an indication of the immediate pH characteristics of the sample should it be mixed with water. Morin and Hutt (2001) compared paste pH to ANC and MPA. Results indicated an increasing paste pH with decreasing total wt% S and that paste pH decreased with decreasing ANC. Cruywagen et al. (2003) stated that samples with a pH < 4.0 are classified as acid/toxic regardless of their overall NAPP or NAG characteristics as acid drainage will be immediately generated. Weber et al. (2004) suggested that samples with paste pH values of < pH 5 would contain acidic SO_4^{-2} salts, which would release acidity during the ANC test thereby obscuring part of the acid neutralization capacity of the sample and biasing the NAPP calculation towards positive NAPP values (e.g., classification of the sample as potentially-acid-forming).

When paste pH values are compared to other ARD geochemical tests a number of conclusions can be drawn regarding sample reactivity and lag time to ARD onset in potentiallyacid-forming samples. This paper investigates acid-base accounting (ABA) data for six rock samples that were characterized as part of the Australian Minerals Industry Research Association (AMIRA) P387a project: Prediction and Kinetic Control of Acid Mine Drainage (e.g., IWRI and EGi, 2002; Weber et al., 2004; Weber et al 2005a,b); two rock samples were also assessed as part of research allied to the AMIRA P387b project: Neutralizing Mineral Reactions in Acid Mine Drainage Control (Weber, 2003); and 31 samples that were assessed as part of research projects funded by Solid Energy (NZ) Ltd (SENZ) that included work by Hughes (2004) and Connor (2005). One prerequisite for sample acceptance into this test suite was that it contained negligible quantities of sulfides other than pyrite. This was defined as < 20 wt% of the sulfide content are sulfides other than pyrite, which is important as the oxidation kinetics of some sulfides are slower than pyrite and/or do not contribute to acid formation (e.g., Stewart et al., 2003). Both would affect the assessment of the acid generation kinetics in the kinetic NAG test and relevance to any lag period to ARD onset as compared to paste pH. These results are thus most applicable to ARD prediction for coal measures and pyrite dominated waste rocks.

The objectives of this work were to determine whether paste pH could provide an indication

of the ARD nature of a sample and after comparison to other standard ABA data whether it could predict any lag to ARD onset. The sample suite was derived from the proposed Cypress mine, West Coast, New Zealand, although additional samples from other international mine sites were assessed.

Methodology

Site description

The Stockton coal mining region is part of the Buller coalfield, which includes the Stockton mine, the proposed Cypress mine, Island Block mine, and the historic Sullivan underground mine. It is located 40 minutes drive NNE of Westport on the West Coast of the South Island of New Zealand. Economic coal seams within the Buller coalfield are associated with the Brunner Coal Measures (BCM), which unconformably overlie the basement rocks comprising Greenland Group turbidite sequences, intruded by granites and porphyries. The Brunner Coal Measures is Eocene in age and is dominated by sandstones inter-layered with siltstones, mudstones, carbonaceous mudstones, and coal seams. The sequence is 70-130 m thick and was deposited in a fluvioparalic environment consisting of braided and meandering streams within a deltaic, barrier, tidal environment (Flores and Sykes, 1996). The BCM are conformably overlain by, and laterally interfinger with, the Eocene marine Kaiata mudstone formation (Flores and Sykes 1996).

Samples

Geological settings for the SENZ samples are provided by Hughes (2004). They are derived from the Stockton mine area, West Coast, New Zealand. In general the mudstones and sandstones are dominated by quartz, muscovite, and kaolinite with varying minor proportions of microcline, albite, illite, and pyrite. Pyrite morphology includes both euhedral and framboidal forms up to 10 μ m in diameter, although larger nodules also occur. Significant carbonate is restricted to the upper stratigraphic layers of the Kaiata mudstone.

In addition to the 39 samples investigated in detail here, the acid-base account data is provided for an additional 102 samples from the proposed Cypress mine. These results are from the SENZ ARD database and details are provided in Appendix 1. The AMIRA samples have previously been discussed and detailed mineralogical assessments provided (Weber et al., 2004b, 2005a, b). Samples are from a variety of localities including the Kaltim Prima coal mine, Kalimantan, Indonesia (Samples MS and PF); the PT Freeport Grasberg Mine, Papua Province, Indonesia (sample PAF-H); Newcrest Cadia mine, Australia (sample C); Placer Pacific Osborne mine, Australia (sample U – tailings composite); Newcrest Telfer mine, Australia (sample T); and Kalgoorlie Consolidated Gold Mines, Australia (samples BS and UM). These AMIRA samples are used to determine whether the developed classification scheme has application beyond the Cypress prospect.

Paste pH Test

Paste pH was determined by mixing 1 part solid to 2 parts deionized water and recording the pH. Paste pH for the AMIRA samples was determined 12 hours after mixing on pulverized $< 75 \,\mu\text{m}$ material (25 g : 50 mL) as per standard methods (IWRI and EGi, 2002). Paste pH was measured by a Meterlab PHM201 pH – temperature meter calibrated against standard buffers at pH 4.01 and 7.0. Paste pH for the SENZ samples was assessed 5 minutes after mixing 5g of pulverized $< 75 \,\mu\text{m}$ material with 10 mL of deionized water to provide an accurate assessment of

any acidic salt content without subsequent interference by neutralization reactions. Paste pH was measured by a Cyberscan 1100 desktop pH – temperature meter calibrated against standard buffers at pH 4.01 and 7.0 for all samples except SENZ samples 4,5,6,7,8,10,12, which were assessed by a WinLab Data-Line pH Meter (pH 4.01 and 7.0 buffers).

Net Acid Production Potential

The net acid production potential (NAPP) is a measure of the samples overall acid generating capacity. NAPP (Equation 2) is the difference between the inherent acid neutralization capacity (ANC) and the maximum potential acidity (MPA) of the sample. A negative NAPP indicates that the sample has a net neutralizing capacity and a positive NAPP indicates that the sample has a net acid generating capacity. NAPP, ANC, and MPA are expressed in kg H₂SO₄/t equivalent. ANC is determined by acid digestion (HCl) of the sample followed by back-titration (NaOH) to determine the quantity of acid consumed (e.g., as per Sobek et al., 1978; IWRI and EGi, 2002). MPA is based on total wt% S as determined by LECO analysis (e.g., Crock et al., 1999) multiplied by the stoichiometric conversion factor 30.6. This conversion factor is determined from the stoichiometry of the pyrite oxidation (Equation 1). Further details of these tests have been reported previously (IWRI and EGi, 2002; Weber et al., 2004). Results are presented in Table 1.

$$NAPP = MPA - ANC$$
(2)

Argon purged leach test

To determine the readily available soluble sulfate acidity (SA) present in the sample due to oxidation of pyrite, an argon purged deionized water leach (Weber et al., 2004) was conducted on samples that had paste pH values < pH 7.0. It is assumed that minimal O₂-induced pyrite oxidation would occur under these conditions (e.g., Equation 1), and likewise, minimal pyrite oxidation by Fe⁺³ would occur due to the circum-neutral pH values and the lack of O₂ that is needed as a terminal electron acceptor. Thus any pH drop is likely to be related to the dissolution of soluble sulfate salts (e.g., pyrite oxidation products).

Three AMIRA samples were tested (PF, MS, PAF-H) using 1g of sample (< 75 μ m) and leaching them (2 h) in 100 ml argon purged deionized water for 1 h with continual purging. Eighteen SENZ samples were assessed using 5g of sample (<75 μ m) and leaching them in 50 mL argon purged (2 h) deionized water for 1 h with continual purging. After the 1 h leach period the pH was recorded and the unfiltered sample was back titrated to pH 7.0 and the SO₄⁻² acidity (SA) calculated from the amount of base added (Equation 3).

$$SA (kg H_2SO_4/t) = \underline{ml NaOH \text{ titrated } x \text{ molarity } NaOH}_{wt \text{ sample } (g)} x 49$$
(3)

Kinetic NAG Test

The NAG test and variations thereof (Miller et al., 1997; IWRI and EGi, 2002) evaluates the net acid generation potential of a sample without specific separate estimation of MPA or ANC (Miller et al., 1997) by using un-stabilized H_2O_2 to accelerate the oxidation of sulfides, particularly pyrite. The NAG test was refined by Environmental Geochemistry International (EGi) and is based on earlier similar procedures (Sobek et al., 1978; Finkelman and Giffin, 1986; O'Shay et al., 1990). The NAG test involves the addition of 250 mL 15 vol% (un-stabilized) H_2O_2 (e.g., 6.35 mol/L H_2O_2) to 2.5g of pulverized sample (< 75 µm), to encourage the rapid

oxidation of reactive sulfides. Un-stabilized H_2O_2 with a pH of 4.5 is used rather than stabilized H_2O_2 with a pH of 3.5 to prevent the sample being classified as acid generating, when it may simply be a function of the pH 3.5 H_3PO_4 acid stabilized H_2O_2 . Un-stabilized H_2O_2 has a determined titrated acidity of 0.05 kg H_2SO_4/t (using a default of 2.5g as the reactant material).

Acid generated during the NAG test by sulfide oxidation reacts with any carbonates and silicates present and may result in their dissolution. Silicate neutralization in the NAG test is a function of the high surface area of the pulverized (< 75 μ m sample) and can be more important than carbonate neutralization in low carbonate samples (Weber et al., 2005b). Rapid pH decrease indicates reactive sulfide minerals and/or acidic sulfate salts (Weber et al., 2004), whereas a high final NAG pH (>6.0) indicates the presence of carbonates (and silicates) with acid neutralizing capacities in excess of acid produced by sulfide oxidation. After digestion the final pH is recorded (NAG pH), and the sample is titrated with NaOH first to pH 4.5 and then to pH 7.0 and the net acid generation (NAG) capacity calculated. The sample is back titrated initially to pH 4.5 as this is the starting pH of the solution. To obtain information on the acid generation rate of a sample, the pH and temperature of the NAG liquor are monitored during the single addition NAG test to produce a pH-temp profile for the duration of the test. This is known as the kinetic NAG test and is conducted at an initial starting temperature of $20^{\circ}C \pm 2^{\circ}C$. A kinetic NAG temp-pH profile was obtained for 30 samples that were assessed as part of this work to provide a reasonable population base for trend identification.

Results (Miller et al., 1997) indicated that there is a correlation between the time for 1 unit pH decrease in the kinetic NAG test (minutes) and the time to pH 4 in the AMIRA column leach test (weeks). However, a pH decrease of 1 pH unit in the kinetic NAG test is ambiguous for samples that initially increase in pH; any decrease thereafter, i.e., from pH 7.0 to pH 6.0 would indicate cessation of the lag period when it is clearly still above the starting pH. For this work we assume that the lag period terminates at pH values < 4.0. This agrees with new research finding by Stewart (2005). Any observed lag will thus provide a reasonable indication of the lag expected in kinetic column leach experiments.

The use of NAG pH and NAPP can be used to classify samples into potentially acid forming (PAF) and non-acid forming (NAF) (IWRI and EGi, 2002) for rapid classification of samples. An uncertain category is also present, which is generated when NAPP and NAG pH are in conflict.

Results and Discussion

Acid - Base Accounting

NAPP data, including ANC and MPA is provided in Table 1 together with NAG results (NAG pH, NAG acidity: pH 4.5 and pH 7.0), and Ar leach results if conducted. A standard classification scheme (IWRI and EGi, 2002) is used to present this data (Fig. 2), which compares NAG pH to NAPP and identifies non-acid forming samples from potentially-acid forming samples. Four samples are non-acid forming, three are borderline uncertain, and the remainder are potentially-acid forming. NAPP results compared reasonable well with NAG results for potentially-acid forming samples (Table 1), generally confirming that they would produce acidity upon exposure to oxygen and water. NAG values did not provide a good acid-base account for non-acid forming samples as the test does not assess the total neutralization present in these samples as the test is designed to determine the net acid generation capacity rather than the net



Figure 2. NAPP- NAG pH classification scheme.

Paste pH and acidic salt effects

Thirteen samples had paste pH values < 4.0 suggesting a significant acidic salt content (up to 30.1 kg H₂SO₄/t) that was released immediately after the addition of water to the sample. Samples with a paste pH of 4.0 - 5.0 also had a significant acidic salt content (up to 9.0 kg H₂SO₄/t), although these acidic salts are generally less than in samples with a paste pH < 4.0. Similar effects would be expected in the field and ARD is likely to commence following first contact with water. Further work is in progress, however, the determined acidic salt content can provide an indication of the limestone dose requirements needed to return the pH to circumneutral conditions and generate a lag prior to encapsulation by an oxygen excluding cover.

For strategic ARD management samples with a paste pH < 5.0 should be highest priority. For samples with a paste pH < 5.0, in all instances, the acidic salt content (SA) as determined by the argon purged leach test is less than the acidic salt content observed in the ANC test as identified by negative ANC values (Table 1). This suggests that the argon leach test using deionized water only assesses the immediate reactive acidic salt content, whereas it may not fully account for less soluble acidic salts released during the ANC test. Further work is needed to refine this test. Samples that had a paste pH > 5.0 can either be potentially-acid-forming or nonacid forming depending on their acid-base account. Some may experience a time lag prior to ARD onset.

Sample	Paste pH	MPA	ANC	Ar Leach pH	SA	NAPP	Final NAG pH	NAG	Lag (min.)
PF	2.9	143	-37.7	3.26	30.1	136##	2.0	96*	0
MS	4.7	40	7.4	4.92	0.5	22.6##	3.0	10*	0
PAF-H	3.8	141	11	4.47	4.8	130	2.4	96*	0
С	8.0	24	11	NC	0	13	4.0	4.7*	240
U	7.9	73	18	NC	0	55	2.3	39*	75
Т	8.0	46	6	NC	0	40	2.8	22*	225
BS	7	97	215	NC	0	-118	7.7	0	NA
UM	7.8	7	128	NC	0	-121	9.8	0	NA
BCM Pad 1	3.4	32.1	-7.9	3.28	4.9	32.1	2.41	32.5	0
BCM Pad 2	2.8	46.8	-12.8	2.95	6.9	46.8	2.19	42.1	0
BCM mudstone	2.9	58.5	-16.8	2.87	13.5	58.5	2.17	51.5	0
BCM Sandstone	2.9	34.9	-6.6	3.42	4.8	34.9	2.50	31.4	0
50:50 Sst:Mst	3.3	36.7	-6.5	3.03	5.8	36.7	2.3	35.1	0
Granite	4.5	3.7	-2.8	3.69	0.7	3.7	3.4	7.1	0
SPF	4.2	16.2	-8.9	3.39	0.4	16.2	2.5	53	0
K1 (fresh)	4.3	58.1	-3.6	3.59	1.3	58.1	2.3	41	0
K2 (weathered)	3.7	41.6	-4.9	3.49	1.4	41.6	2.4	37	0
DH1565 56 - 56.5	4.0	87.7	-5.6	3.41	9.0	87.7	2.2	44.7#	0
DH1613 48 - 48.03	2.9	78.4	-9.1	2.89	4.9	78.4	2.2	60.4#	0
DH1545 16.55 -17.05	2.8	102.8	0**	2.78	9.1	102.8	1.8	73.3#	0
DH1613 10-10.03	3.0	64.9	-9.9	3.28	5.7	64.9	2.3	36.6#	0
DH1545 9.72-10.22	4.4	86.8	-1.2	3.93	3.3	86.8	1.9	54.4#	
DH1565 29.4-30m Ox	3.8	68.1	6.2	3.7	3.7	61.9	2.28	54.1	0
DH1565 29.4-30m Fr	5.8 - 6.1	68.1	6.2	7.05	0	61.9	2.35	53.9	4
DH1565 23.5-24.2	7.6	65.8	14.3	NC	-	51.5	2.21	58.6	21.5
DH1717 73.35-74.23	8.1	52.9	19.2	NC	-	33.7	2.6	42.9	54
DH1613 22-22.02	7.7	58	51.9	NC	-	6.1	2.75	33.3	41
DH1513 45.65-45.69	7.9	42.1	20.5	NC	-	21.6	2.68	32.3	9.5
DH1613 44.93 - 45.41	7.6	67.9	17.6	NC	-	50.3	2.45	56.6	48.5
DH 1717 16.32-16.35	7.6	50.2	58	8.1	0	-7.8	9.21/8.4	0	NA
DH 1613 33.0 - 33.40	7.6	36.7	57.7	8.2	0	-21	6.1	7.4	NA
DH 1613 34.3 - 34.33	7.5	47.2	68.2	NC	-	-21	7.0	0	NA
SENZ 4	3.6	98.8	0.5	IP	IP	98.3	2.21	61.6	0
SENZ 5 (BCM Kaiata)	6.6	0.2	0.5	IP	IP	-0.3	4.05	$0.4^{\#}$	NA
SENZ 6 (BCM Kaiata)	5.0	30.2	3.1	IP	IP	27.1	2.44	31.7	2
SENZ 7	6.3	43.1	10.3	IP	IP	32.8	3.69	10.4	9
SENZ 8	7.3	52.6	47.9	NC	-	4.7	3.18	19.21	356
SENZ10	5.4	53.9	3.6	IP	IP	50.3	2.44	37.94	5
SENZ 12	7.6	61.5	20.8	NC	-	40.7	2.55	31.44	26

Table 1. Acid-base account data for waste rock samples.

[#] NAG acidity to pH 4.5; ^{##} NAPP corrected for the influence of acidic salts on the ANC test (See Weber et al., 2004). * indicates NAG as determined by sequential NAG test (IWRI and EGi, 2002); ** ANC corrected to zero for negative ANC values (Hughes, 2004). NC = not conducted as Ar leach test pH > 7.0; IP = testing in progress; Lag as determined by the kinetic NAG test (minutes) is considered zero if <1 minute). NA indicates not applicable as material is non-acid-forming and remains at neutral pH.

Classification of samples based on NAG pH and paste pH provided a general trend (Fig. 3) that can be used to divide samples with paste pH > 5.0 into non-acid-forming and potentially-acid-forming. Samples that are potentially-acid forming (e.g., NAG pH < 4.0) and have a paste pH > 5.0, therefore have an immediate neutralization value that is greater than any immediate acid generating capacity. This equates to a potential lag period prior to the onset of acidity. The length of this lag cannot be determined by the paste pH test, hence kinetic testing is required to validate this lag.

Kinetic NAG testing

Thirty two samples were tested by the kinetic NAG test. Kinetic NAG profiles (pH and temperature) for samples PF, MS, and PAF-H have been published previously (Weber et al., 2004; Weber et al., 2005a) and no lag period was observed prior to pH decrease to below a pH of 4.0. This is expected for samples that are acidic as identified by the paste pH test. Similarly other samples (K1 (fresh), K2 (weathered), Silt pond fines, BCM mudstone, DH1565 29.4-30m (Ox) and SENZ 4) that had paste pH values < 5.0 also had no lag prior to pH decrease to below 4.0 in the kinetic NAG test. These representative samples are shown in Fig. 4. Although these samples generate acid immediately, this does not mean they are all severe in regards to ARD. For instance, the granite sample generates only 7.1 kg H₂SO₄/t, but the onset of ARD is rapid.



Figure 3. Paste pH – NAG pH classification scheme. Potentially acid-forming samples are divided in high, medium and low risk in regard to time to ARD. Low risk samples may have a lag period due to carbonate and silicate neutralization buffering. NAF samples are unlikely to generate ARD. Uncertain Category classification suggests additional re-testing is required. BCM are samples from the Brunner Coal Measures; AMIRA are from the P387a,b projects; Kaiata samples are from the Kaiata mudstone at the proposed Cypress mine.

Kinetic NAG results for samples that were classified as non-acid-forming (NAF) and had past pH values greater than 6.0 remained circum-neutral throughout the test (Fig 5) This includes samples Black Shale, Ultramafic, DH1613 34.3 - 34.33, DH1717 16.32 - 16.35, SENZ 5 and DH1613 33.0 - 33.60, although the last two samples are marginal in respect to being classified as non-acid-forming. SENZ 5 is very low in sulfides (MPA = $0.2 \text{ kg H}_2\text{SO}_4/\text{t}$) and ANC ($0.5 \text{ kg H}_2\text{SO}_4/\text{t}$) and is marginally NAPP negative indicating its nature as NAF. This is in agreement with a NAG pH of 4.05, just above the NAG PAF classification threshold of pH 4. Sample DH1613 33.0 - 33.60 is also very marginal in that it also only just maintained a pH in excess of 4.0 after carbonate exhaustion at 1000 minutes. Although these samples do contain sulfides, the acidity generated is neutralized by the carbonate and silicate neutralization inherent in the sample.

Of most interest to this research are samples that have a paste pH in excess of 5.0, and have NAG pH values less than 4.0. Kinetic NAG testing indicated that all these samples experienced a lag in the kinetic NAG test prior to pH drop below pH 4.0. This includes samples DH1513 45.65m - 45.69; DH1565 23 - 24m; DH1565 29.4 - 30m; DH1613 22 - 22.02m; DH1613 44.93 - 45.41m; DH1717 73.35 - 74.23m; SENZ 6; SENZ 7; SENZ 8; SENZ 10; SENZ 12; and AMIRA samples C, U, and T (Fig. 6 and 7). These results still require field validation, however it would suggest that there will be a lag prior to the onset of ARD in the field for rocks represented by these samples. The lag appears to be greater for samples having a paste pH > 6.0and these can be considered lower priority for ARD mitigation efforts than samples with lower paste pH values. Although there is a time lag in the kinetic NAG test identified for samples that have NAG pH values below 4.0 and have a paste pH between 5 - 6 (Fig. 7), the onset of ARD as identified by the kinetic NAG test (i.e., pH < 4.0) is likely to be quick and should be classified as medium risk in regard to ARD severity. The lag time is due to the inherent ANC being available to neutralize the acid generated by the sulfides under NAG conditions. However, the lag time does not compare to ANC (as determined by acid titration) due to several unqualified variables that most likely include wt% sulfide, sulfide form (e.g., framboidal versus euhedral) that affects reaction rates and hence acid generation rates, acidic SO_4^{-2} salt content, and carbonate reactivity and grain size. Thus it is imperative to test all samples by the kinetic NAG test to determine lag period; paste pH cannot provide any indication of lag duration.

Testing of fresh samples representative of the rock in the field is essential for modeling of waste rock as PAF and NAF. Coal measures containing framboidal pyrite are extremely susceptible to oxidation and acidic salts can form within a few weeks to months thereby masking any lag present. Sample DH1565 29.4 – 30m (Ox) was tested after 4 months storage in a plastic bag at room temperatures after crushing. The result was a paste pH of 3.8 in comparison to the same sample DH1565 29.4 – 30m (Fr) that was crushed and assessed immediately by paste pH and kinetic NAG testing. Using fresh rock the paste pH was 5.8 -6.1 and the lag in the kinetic NAG test was 4 minutes.



Figure 4. Selected samples having paste pH values below 5.0 and displaying immediate pH decrease to below a 4.0 in the kinetic NAG test. Other samples displaying these characteristics are shown in Table 1 and have paste pH values below 5.0.



Figure 5. Selected non-acid-forming samples having paste pH values > 6.0 and NAG pH values > 4.0.



Figure 6. Selected PAF samples that display a lag period prior to acid formation in the NAG test and have paste pH values > 6.0 and NAG pH values < 4.0.



Figure 7. PAF samples that have paste pH values between 5 and 6 and have NAG pH values < 4.0.

A classification system based on these results is proposed for rocks associated with the Kaiata mudstone having variables levels of carbonate and pyrite (Table 2). The classification system is based on the immediate ARD severity and the duration to significant acid release. Field validation is still required.

Samples that have NAG pH values < 4.0 and paste pH values < 4.0 are already generating significant quantities of acid and should be classified as high risk. Mitigation methods for these rock types should include immediate dosing with neutralizing materials such as agricultural grade limestone (<2 mm) and encapsulation to reduce oxygen-water ingress. Results from the Ar leach test suggest that 1.4 to 30.1 kg CaCO₃/t is needed to return pH to circum-neutral conditions prior to encapsulation. Samples with a paste pH of 4 - 6 are medium risk in that ARD has not become extreme. These samples should be dosed with a neutralizing material and encapsulated to reduce oxygen-water ingress. Results from the Ar leach test suggest that 0.4 to 9.0 kg CaCO₃/t is needed to return pH to circum-neutral conditions prior to encapsulation. Samples that have a paste pH >6 and have a NAG pH > 4 will most likely have a reasonable lag period prior to ARD formation in the field, although this will need field validation. These samples will only need encapsulation to permanently exclude oxygen and water provided this

occurs prior to pH drop. Samples having a paste pH > 6 and NAG pH > 4.0 are classified as non-acid-forming and can be used as a non-acid-forming engineering material if required, although further testing is needed on marginal samples.

	-						
Classification	Test Criteria	Strategic Plan					
NAF	Paste pH > 6	Possibly suitable for use as an engineering					
	NAG pH > 4	material (further testing required)					
PAF - Lag to ARD	Paste pH > 6	Encapsulation to reduce subsequent oxygen-					
	NAG pH < 4	water ingress prior to ARD formation					
PAF – Medium risk	Paste pH 4 - 6	Neutralisation dose required followed by					
	NAG pH < 4	immediate encapsulation					
PAF – High risk	Paste pH < 4	Neutralisation dose required followed by					
	NAG pH < 4	immediate encapsulation. Dose rate is greater					
		than medium risk.					

Table 2. Classification system based on NAG pH and paste pH for Kaiata mudstone. (Dose rates still need to be quantifies as part of ongoing research).

Conclusions

A simple classification scheme has been developed to help predict high, medium and low risk ARD rock types based on paste pH and NAG pH; two simple rapid screening tools for ARD characterization. This classification system addresses only the hydrogen ion balance of the materials being investigated and does not consider the release of toxic metals commonly associated with acid generation. This classification scheme evolved from a database of ARD classification work associated with the proposed Cypress mine, Stockton area, West Coast, New Zealand. Several samples from the AMIRA P387A project also fit into this classification scheme and suggest that it may have application to other sites, however these results are most applicable to coal measures and pyrite dominated waste rocks.

Results indicate that paste pH is a good indicator of the immediate acid-base reactivity of the sample. It is common knowledge that if rock has paste pH values < 5 then it is high priority for strategic ARD management. However, as a rule, at Cypress, if paste pH > 6 but the sample is NAPP positive or NAG acidity > 0 and ANC is > 5 kg H₂SO₄/t then there will be a lag period prior to acid onset. Kinetic NAG testing should be conducted to determine the lag period. Samples having a paste pH between pH 4 and 5 may have a slight lag but should still be considered medium risk in regard to time to ARD formation.

These laboratory studies aid in our understanding of the front end of the ARD evolution trend (Fig. 1) for each particular sample as assessed in the laboratory. From a simple paste pH test geochemists can predict whether there is likely to be acidic conditions or circum-neutral drainage at the onset of rock weathering. When used in combination with the NAG test, results can be used to predict whether the circum neutral drainage will persist or if there is a time lag to acid generation. Duration of this lag period can only be determined from the Kinetic NAG test; paste pH cannot provide this data.

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		Paste					
SENZ Sample	Description	pН	ANC	MPA	NAPP	NAGpH	NAGpH7.0
SE1	Kaiata	5.0	5.9	72.8	66.9	2.50	54.0
SE2	Kaiata	4.9	1.5	96.4	94.9	2.26	72.9
SE3	BCM	4.2	0.5	18.7	18.2	2.61	26.2
SE4	Kaiata	3.9	0.5	96.7	96.2	2.23	72.1
	Composite						
SE5	BCM/KF	6.6	0.2	0.5	-0.3	4.05	0.4*
	Composite						
SE6	BCM/KF	6.1	3.0	29.1	26.1	2.46	30.0
SE7	Kaiata	6.3	10.29	43.146	32.856	2.7	17.7
SE8	Kaiata	7.3	47.9	52.632	4.732	5.9	1.8
SE9	Kaiata	7.3	49.49	52.02	2.53	6	1.8
SE11	Kaiata	7.3	50.84	37.944	-12.896	5.5	3.9
W01	Kaiata	5.1	1.1	76.3	75.2	2.55	51.9
W02	Kaiata	5.4	1.7	81.5	79.8	2.54	53.1
W03	Kaiata	5.3	4.7	88.5	83.8	2.49	60.1
W04	Kaiata	5.5	1.7	83.0	81.3	2.47	62.2
W05	Kaiata	7.6	11.8	60.0	48.3	2.85	40.0
W06	Kaiata	8.0	19.4	75.6	56.3	2.73	44.0
W07	Kaiata	4.9	3.7	72.0	68.3	2.79	41.8
W08	Kaiata	7.7	54.6	55.7	1.2	5.30	3.2
W09	Kajata	7.8	91.5	45.6	-45.9	5.14	4.6
W10	Kaiata	7.8	221.1	54.2	-166.9	5 54	3.5
W11	Kajata	7.8	36.8	60.6	23.9	3 20	23.1
W12	Kajata	8.1	410.1	28.8	-381.3	6.18	23.1
W12	Kajata	7 5	25.4	55.4	30.0	2.62	19.1
W14	Kajata	6.9	10.0	58.8	48.8	2.62	36.8
W15	Kajata	77	16.0	72.6	56.6	2.33	68.8
W16	Kajata	7.5	9.2	83.3	74 1	2.55	62.4
W10 W17	Kajata	7.9	15.1	73.5	58 <u>4</u>	2.43 2 40	60.8
W18	Kajata	6.6	10.0	50.5	40 5	2.40	44 7
W19	Kajata	6.0 6.4	8 1	67.1	-+0.5 59.0	2.75	50.6
W20	Kajata	6.5	3.2	72.0	687	2.65	68.0
W21	Kajata	0.3 7 2	10.8	71.7	60.9	2.01	52.9
W22	Kajata	7.1	14.4	101.7	87.3	2.71	66.1
W23	Kajata	44	0.0	91.9	91.9	2.61	72.0
W24	Kajata	6.13	7.2	55.1	48.0	2.50	37.1
W25	Kajata	6.70	10.0	67.1	57.1	2.51	49.2
W26	Kajata	7.28	13.9	77.5	63.6	2.62	50.4
W27	Kajata	6.20	2 5	118.5	116.1	2.03	79.6
W28	Kajata	7 78	171.6	44 1	-127.5	6.09	19
W29	Kajata	7.63	31.4	56 <i>4</i>	25.0	3 24	167
W30	Kajata	7.05	35.5	56 0	20.6	3.58	13.7
W31	Kajata	7 73	46 1	<u>4</u> 93	20.0	5.50	3.7
W32	Kajata	7.75	63.8	-7.5 20 8	_24 0	5.05	3.1
W33	Kajata	7.07 7.70	<u>48</u> 6	59.0	- <u>∠</u> -1.0 9.6	5.52	<i>4</i> 6
W34	Kajata	7.70	40.0	50.2 50 1	11 /	<i>J</i> . <i>JJ</i> <i>A</i> 61	0 6 1
W35	Kajata	7.52	767	/0 N	11. 4 77.8	3 27	15 7
11 55	ixalata	1.54	20.2	72.0	22.0	5.57	13.7

<u>Appendix 1.</u>	ARD geochemical data derived from the Solid Energy (NZ) Ltd Cypress mine
	database, West Coast, New Zealand

W36	Kaiata	7.60	18.6	64.3	45.7	2.54	45.8
W37	Kaiata	7.34	8.0	83.3	75.3	2.36	61.9
W38	Kaiata	7.55	16.0	59.7	43.7	2.70	30.4
W39	Kaiata	7.26	11.6	66.5	54.9	2.52	47.8
W40	Kaiata	7.65	11.8	74.4	62.7	2.45	52.5
W41	Kaiata	6.70	8.0	83.9	75.9	2.11	92.3
W42	Kaiata	7.24		1.5		3.80	13.4
W43	Kaiata	6.9		8.0		2.24	78.6
W44	Kaiata	7.56	74.5	62.2	-12.3	5.26	11.4
W45	Kaiata	7.23	77.2	58.8	-18.4	5.88	1.7
W46	Kaiata	7.83	42.8	58.5	15.7	4.00	7.3
W47	Kaiata	7.61	38.4	44.7	6.3	4.93	3.8
W48	Kaiata	7.84	66.2	46.6	-19.6	6.14	1.1
W49	Kajata	7.97	34.7	56.7	22.0	3.52	11.9
W50	Kajata	7.88	22.4	54.2	31.8	7.81	24.9
W51	Kajata	7.84	19.1	64.0	44.9	2.81	37.6
W52	Kaiata	7.63	14.8	70.7	55.9	2.62	52.3
W53	Kaiata	7.63	11.0	68.0	56.6	2.70	45.1
1514	Kajata	7 49	50.84	37 944	-12.89	5 5	3 92
1514	Kaiata	7.09	3 55	53 856	50.30	23	31.65
1559	Kajata	7.67	49 49	52.02	2 53	6.0	1 76
1562	Kajata	7.07	47.90	52.02	2.33 4 73	5.9	1.70
1563	Kajata	7.37	10.20	43 146	32.86	27	17.74
1604	Kajata	8 33	23 52	42 228	18 71	3.8	5 08
1694	Kajata	8.55 7.51	23.52	42.220 58.14	33 15	2.8	20.58
1694	Kaiata	7.31 8.20	24.99	16 818	12 52	2.8	20.58
1694	Kaiata	8.20 8.13	20.83	40.818 61 506	12.52	4.2	4.41 27 11
1094	Kalata	8.13 8.20	20.05	55.09	40.08	2.0	27.44
1094	Kalata	8.20 7.05	12.00	57.00	42.22	2.7	27.03
1094	Kalata	7.93 8.02	12.72	62 242	40.08	2.5	10.06
1094	Kalata	8.05 8.20	15.72	64 972	49.02	2.5	40.90
1694	Kalala	8.29 7.69	10.17	04.872	48.70	2.5	59.09
1694	Kalala	7.08	15.19	07.952	52.74	2.4	55.27
1694	Kalala	7.08	10.78	74.558	57.58	2.4	JJ.5/
1694	Kalala	7.80	19.00	12.522	52.92	2.4	48.41
1694	Kaiata	/.98	8.82	60.282	51.46	2.4	36./5
1694	Kaiata	8.10	13.60	67.626	54.03	2.5	30.10
1694	Kaiata	7.38	8.70	/9.56	/0.86	2.2	57.23
1694	Kaiata	7.20	6.37	85.068	/8./0	2.2	65.86
1694	Kaiata	7.25	4.66	92.412	87.76	2.1	73.79
1694	Kaiata	6.85	4.66	82.926	78.27	2.2	64.58
JH3	Kaiata	7.8	79.8	45.9	-33.90	8.3	0*
JH5	Kaiata	8.1	97.8	52.02	-45.78	8.5	0*
JH6	Kaiata	8.2	35	42.84	7.84	3.4	303*
JH8	Kaiata	3	0	64.26	64.26	2.3	36.6*
JH9	Kaiata	7.5	67.1	55.08	-12.02	3.3	2.8
JH11	Kaiata	6.9	60.8	42.84	-17.96	3.6	*
JH18	Kaiata	3	0	76.5	76.50	2.2	60.4*
JH26	Kaiata	4	0	85.68	85.68	2.2	44.7*
JH29	Kaiata	7.6	63.6	55.08	-8.52	7.7	0^*
JH32	Kaiata	2.9	0	85.68	85.68	1.9	54.4*
JH35	Kaiata	2.8	0	100.98	100.98	1.8	73.3*

JH36	Kaiata	7.8	53.5	45.9	-7.60	5.5	0*
BCM Pad 1	Egypt trial material	3.4	-7.9	32.1	32.1	2.41	32.5
BCM Pad 2	Egypt trial material	2.8	-12.8	46.8	46.8	2.19	42.1
BCM mudstone	Egypt trial material	2.9	-16.8	58.5	58.5	2.17	51.5
BCM Sandstone	Egypt trial material	2.9	-6.6	34.9	34.9	2.5	31.4
50:50 Sst:Mst	Egypt trial material	3.3	-6.5	36.7	36.7	2.3	35.1