

A Testing Approach to Assess the Weathering Behaviour of Lithotypes Characterised by a Trace-sulphide/Carbonate-deficient Mineralogy: Application to Altered Andesites/Diorites at the Boddington Gold Mine, Western Australia

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Abstract

Altered andesites/diorites at the Newmont Boddington Gold Mine (NBGM) locally contain pyrrhotite and pyrite as trace components (e.g. total-S values less than 0.5 %). The groundmass of these waste rocks is typically deficient in carbonates (e.g. CO₃-C values less than 0.02 %). Sulphide oxidation rates (SORs) have been obtained via weathering columns in which cycles of flushing and desaturation to residual moistures are conducted on a weekly basis. During 65 weeks of testing, samples with sulphide-S values up to 0.40 % have been characterised by leachate-pH values of 6+, and peak SORs ranging up to approx 5 mg SO₄/kg/day during the summer months. The extent of sulphide oxidation during the testing period corresponds to approx 0.5 kg H₂SO₄/tonne. Acid consumption rates (ACRs) were independently determined via jar-soak tests at constant temperature (18-22 °C) using 0.005 M-HAc (acetic acid) solutions adjusted to pH 5. At pH 5-6+, the ACRs have been approx 10+ mg H₂SO₄/kg/day for the consumption of approx 5-6 kg H₂SO₄/tonne.

The current study is providing a sound geochemical basis upon which to propose a S-cut-off value (e.g. 0.3 %) for differentiating between reactive and benign varieties of the andesite/diorite waste rocks at the NBGM. Results to date indicate that the silicate groundmass of these lithotypes contains a "pool" of alkalinity forms sufficient to sustain circum-neutral pH during oxidation of waste rock containing at least 0.1-0.2 % of sulphide-S.

Key Words: sulphide oxidation, acid consumption rate, circum-neutral, silicate buffering

Introduction

This study presents results of kinetic testing on samples of altered andesites/diorites at the Newmont Boddington Gold Mine (NBGM) in the south west of Western Australia. These waste rock types locally contain pyrrhotite and pyrite as trace components, and the groundmass is typically deficient in carbonate minerals. Lithotypes of this kind present difficulties in assessing acid forming characteristics (e.g. Li 2000; Morin and Hutt 2007). The key objective of this study is to determine a S-cut-off value (e.g. 0.3 %) for differentiating between reactive and benign varieties of the andesite/diorite waste rocks at the NBGM. The current work forms part of a wider programme of investigations on mine waste and environmental management at the NBGM to assist with life of mine and closure planning (Amoah *et al.* 2011).

Static Testwork Programme

The fine-earth (< 2 mm) fractions of cuttings from a range of blast-holes were initially tested, and then composites prepared to span a range in sulphide-S and CO₃-C values of 0.14-0.69 %, and 0.01-0.18 %, respectively (Table 1). The methodology described in AMIRA (2002) was used to determine Acid Neutralisation Capacity (ANC), and is listed as Bulk-ANC in Table 1. Calcite-ANC values were calculated assuming that all CO₃-C occurs as calcite.

Mineralogical studies (powder x-ray diffraction, and scanning electron microscopy with electron-probe analysis) showed that the sulphide-mineral suite in the samples comprised pyrrhotite and pyrite (Table 2), and Maximum Potential Acidity (MPA) values were calculated accordingly from sulphide-S. The

assemblage of primary silicates in the groundmass of each sample was very similar, as expected for andesites and diorites generally (viz. differences largely textural compared with composition and mineralogy).

Table 1. ABA results for composite samples of andesites/diorites

Sample No.	Total-S (%)	Sulphide-S (%)	CO ₃ -C (%)	Bulk-ANC	Calcite-ANC	MPA
				kg H ₂ SO ₄ /tonne		
GCA8644	0.15	0.14	0.02	20	1-2	4.3
GCA8645	0.25	0.23	0.01	15	1	7.1
GCA8646	0.35	0.33	0.11	34	9	10.0
GCA8647	0.42	0.40	0.02	19	1-2	12.2
GCA8649	0.63	0.61	0.18	42	15	18.7
GCA8648	0.72	0.69	0.01	16	1	21.1

Note: Samples GCA8646 and GCA8649 gave a "fizz" rating of 2. The "fizz" rating for all other samples was zero.

Table 2. Mineralogical results for composite samples of andesites/diorites

Mineral	GCA8644	GCA8645	GCA8646	GCA8647	GCA8648	GCA8649
quartz	major	major	major	major	dominant	major
plagioclase	major	major	major	major	major	major
biotite	accessory	accessory	accessory	minor	accessory	accessory
chlorite	accessory	accessory	accessory	accessory		accessory
clinozoisite	trace	accessory	accessory	accessory	accessory	accessory
Ca-amphibole	accessory	accessory	trace	trace	trace	trace
pyrrhotite	trace	trace	trace	trace	trace	trace
pyrite		trace		trace	trace	trace
chalcopyrite					trace	trace
carbonate			trace			trace

Note: dominant is greater than 50 %; major is 20-50 %; minor is 10-20 %; accessory is 2-10 % and trace is less than 2 %.

Samples GCA8648 and GCA8649 are low grade ore samples with Cu contents of 2,400 mg/kg and 1,200 mg/kg, respectively. Results for these samples are discussed only briefly, since the focus of this paper concerns the weathering behaviour of the waste rock samples.

Kinetic Testwork Programme: Weathering Columns

Variation to AMIRA (2002) methodology

The weathering-columns and the geometry of the gantry housing the columns and flood-lamps are based on those described in AMIRA (2002). The main departures from AMIRA (2002) are:

- power, and operation, of the flood-lamps in order to constrain maximum and minimum temperatures of the sample-beds during the drying phase; and,
- weekly flushing, and a greater rate of deionised-water addition during flushing.

In order to constrain variations in the sample-bed temperatures, 80W flood-lamps were employed, and turned on intermittently during the night-time (via automatic timers) as follows:

- June to September: 9 hrs 17.00-19.00, 22.00-24.00, 2.00-5.00 and 7.00-9.00
- October to May: 6 hrs 22.00-24.00, 2.00-5.00 and 8.00-9.00

The above differs from that described in AMIRA (2002) where 150W flood-lamps are turned on continuously during the daytime for 8-10 hrs. Although it is implied in AMIRA (2002) that the sample-bed surface temperature does not exceed approx 30-35 °C, this was not the case under the conditions of our laboratory. In-house research (unpublished) using columns instrumented with thermistors and soil-moisture sensors, and automatically logged hourly, has shown that during the latter stages of drying when residual-moisture contents are attained, the methodology described in AMIRA (2002) results in summer-peak temperatures (for 1-2 hrs in mid-afternoon) up to approx 70-80 °C in the top 5-10 mm on the side of the sample-beds closest to the centre of the flood-lamps (i.e. near-lamp-side). However, with 80W flood-lamps operated intermittently during the night-time, the near-lamp-side temperature in the top 5-10 mm ranges up to approx 40 °C only during extreme hot spells in summer. Since peak temperature occurs in the top 5-10 mm on the near-lamp-side, the remainder of the sample-bed has a temperature no greater than 30-40 °C.

Related monitoring during the winter months has shown that there is an approx 10 °C difference between the summer-peak and winter-peak temperatures. Winter- and summer-peak SORs correspond to mean temperatures of approx 20 °C and 30 °C, respectively. As this study progresses, temperature variation in SORs will allow estimation of the activation energy (E_a) for sulphide oxidation in the andesites/diorites, and so serve as input to kinetic geochemical modelling.

Each weathering column contains 1.5 kg of the fine-earth fraction of the composites and is flushed with 1.00 kg of deionised-water each week. The column leachates are weighed for mass-balance calculations, and filtered and preserved, as appropriate, for specific analyses. Such weekly flushing differs from the 4-weekly flushing regime advocated in AMIRA (2002). In the present work, it generally took up to 8-10 hours for drainage to be completed during the flushing phase of each cycle.

Daily weighing indicates that following commencement of each drying phase with the flood-lamps on Monday nights, it takes approx 3 days to approach residual moistures. Residual moistures correspond to total suctions greater than approx 1-2 MPa, as determined in preliminary testing using a WP4 Dewpoint Potential Meter (Decagon).

Weathering behaviour and rates recorded to date

pH and bicarbonate alkalinity

The variation in leachate-pH values is shown on Figure 1. Leachate-pH values have been consistently 6+, except for the low-grade-ore sample GCA8648 where values below 6 first occurred after about 25 cycles. By cycle-65 leachate-pH for this sample was 4.4.

Variation in leachate- HCO_3 concentrations is shown on Figure 2. Samples GCA8644, GCA8646, and GCA8649 have been characterised by leachate- HCO_3 concentrations within the tens-of-mg/L range, especially for GCA8646 and GCA8649. The latter samples had $\text{CO}_3\text{-C}$ values above 0.1 % (Table 1) and traces of carbonate minerals in these samples were identified in the mineralogical study (Table 2). The designation "bulk-carbonate buffering" on Figure 2 signifies the role played by traces of carbonate minerals (e.g. calcites) in the samples *in situ*.

Beyond cycle-30, samples GCA8645 and GCA8447 were characterised by leachate- HCO_3 concentrations less than 5-6 mg/L, and leachate-pH values above 6.3. The recorded HCO_3 concentrations may reflect dissolution of minute amounts of carbonate minerals present in the samples *in situ*, and/or absorption of atmospheric CO_2 into pore-fluid films during the unsaturated, drying phase of the weathering cycles. That is, in effect, the hydroxide alkalinity associated with the hydrolysis and/or dissolution of silicate minerals is transiently converted to carbonate (e.g. " CaCO_3 ") during weathering, and hence a source of HCO_3 in circum-neutral buffering (Morin and Hutt 2007).

Figure 1. Variation in leachate-pH values for weathering columns

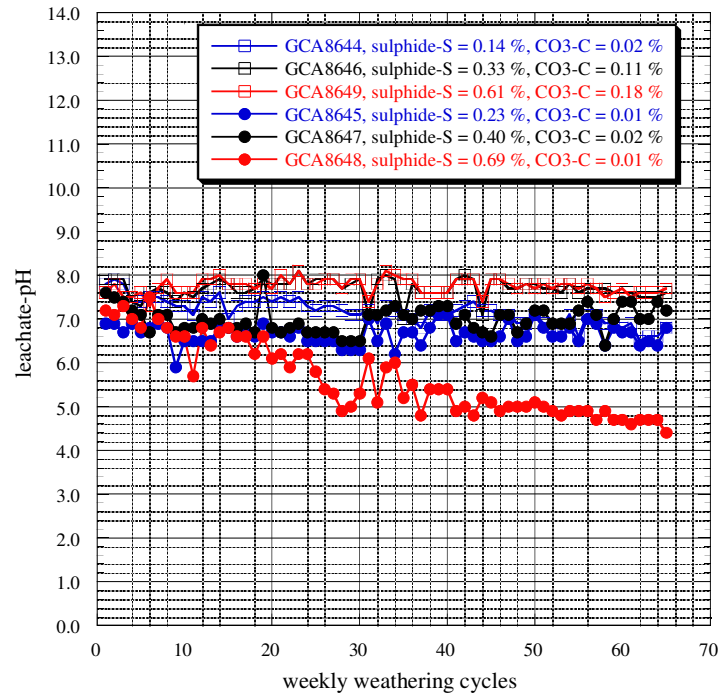
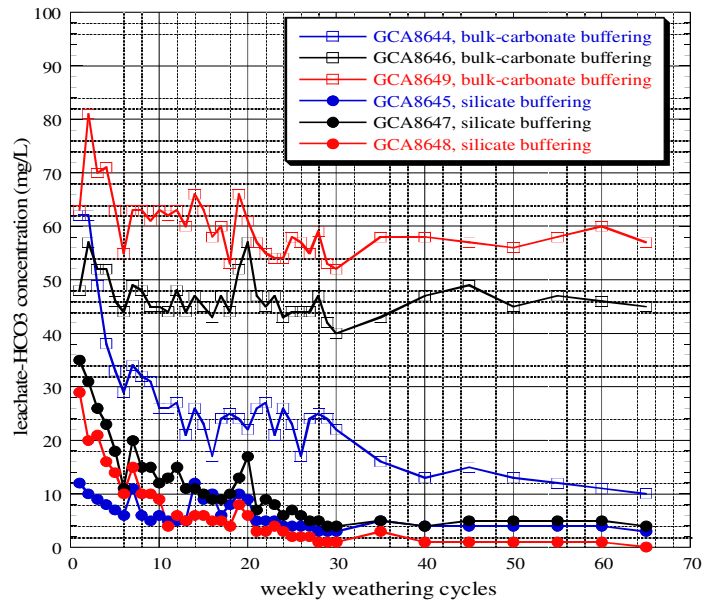


Figure 2. Variation in leachate- HCO_3 concentrations for weathering columns



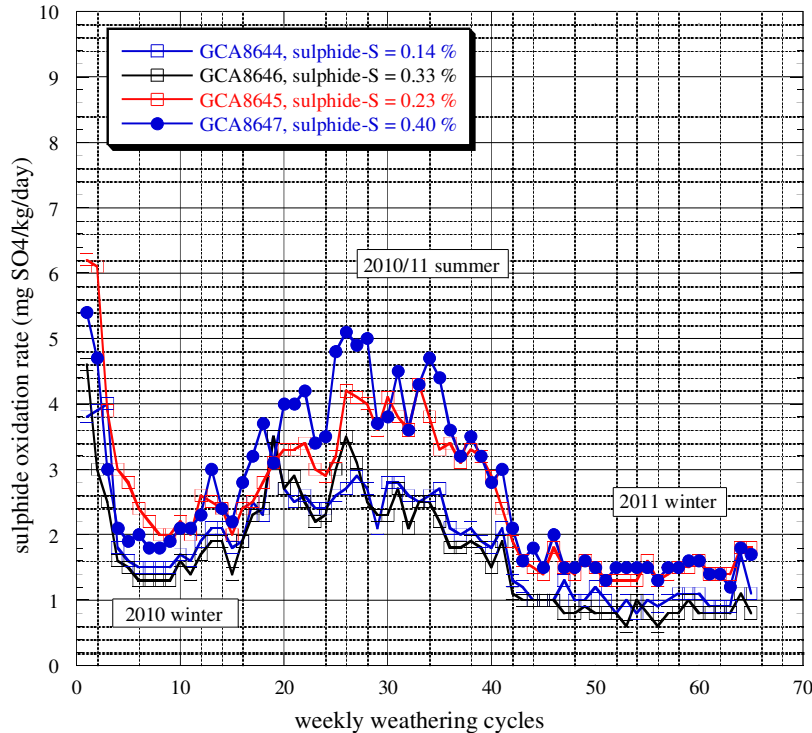
Beyond cycle-25, sample GCA8648 had leachate- HCO_3 concentrations near the detection limit of 1 mg/L, as expected, due to the leachate-pH tracking towards 4.3 (i.e. the methyl orange end-point for bicarbonate alkalinity determination).

Sulphide oxidation rates

The SORs (in mg $\text{SO}_4/\text{kg}/\text{day}$) correspond to the amount of sulphate eluted from the columns normalised with respect to the sample weight (i.e. mg SO_4/kg) and divided by 3 days (i.e. the approximate period during each weathering cycle when the moisture status of the sample-bed was neither at a relative saturation above 80-85 % where oxygen diffusion is limiting, nor at residual moistures). The so-calculated SORs broadly correspond to sulphide oxidation being restricted to when the sample-beds were appreciably unsaturated, yet still moist. Since sulphide oxidation, albeit at subdued rates, would also occur on other days following completion of draining each week, these calculated SORs are variously biased on the high side. Expressing the SORs in this way allows for a more consistent comparison with the ACRs obtained from the pH5-soak tests (see below) which correspond to saturated conditions.

The weathering columns were commissioned in late-June 2010. The variation in the SORs is shown on Figure 3 for samples GCA8644 to GCA8647 (i.e. waste rock samples that are the focus of this study).

Figure 3. Variation in sulphide oxidation rates for weathering columns



The influence of the seasonal-temperature wave on the variation in the (circum-neutral) SORs is clear. Future weathering cycles over the 2011/12 summer and 2012 winter should reveal (in)consistencies in the seasonal amplitude of the SORs. Related seasonal variations in SORs due to temperature swings were reported by Lapakko (2003) for humidity-cell testing. In this study, the daily heat loads applied during summer correspond to pan-evaporation rates (independently monitored) up to approx 5-6 mm/day. During the winter months, the pan-evaporation rates drop to around 3-4 mm/day which are more than sufficient to attain residual moistures in the sample-beds (i.e. rate of drying non-limiting).

The SOR for sample GCA8647 (i.e. sulphide-S = 0.40 %) peaked at approx 5 mg SO₄/kg/day during the 2010/11 summer, and corresponds to a mean temperature of approx 30 °C. The winter-peak SOR for this sample was 1-2 mg SO₄/kg/day, corresponding to a mean temperature of approx 20 °C.

Kinetic Testwork Programme: pH5-Soak Tests

Basis of test for quantifying circum-neutral buffering kinetics of silicate groundmass

pH5-soak tests employing dilute solutions containing acetic acid have been used to estimate acid consumption rate (ACR) over the pH range 5-6 (nominal). The raw data were adjusted (see below) to take into account the potential enhancement of silicate dissolution due to the use of acetic acid rather than sulphuric acid.

"Single-mineral" dissolution rates for a range of primary silicates (viz. feldspars, Ca/Mg-silicates and ferromagnesian-silicates) at 20-25 °C using solutions containing acetic acid for pH buffering are reported in Ganor *et al.* (2009). "Single-mineral" dissolution studies are typically conducted on specimen minerals of high-purity and using size fractions within the range 100-500 µm (e.g. fine sand grading). Sample preparation methods minimise the creation of defects and other imperfections on mineral surfaces, so that unrepresentative, initial rates of dissolution are as short-lived as possible. Varying pre-treatments (e.g. ultrasonification) are also undertaken to remove ultra-fines for the same reason.

A summary of test conditions employed in research on the dissolution of primary silicates in solutions which contain dilute concentrations of acetic acid is presented in Table 3.

Table 3. Literature dissolution rates for primary silicates in solutions containing acetic acid

Mineral	[Acetate] _{Total} (mM)	Final- pH	Temp. (°C)	Dissolution- Enhancement Ratio (DER)	Reference
<i>This Study</i>	5	5-6	18-22		
labradorite	0.1-1	4-6	20-22	1-2	Welch and Ullman (1993)
bytownite	1	6	20	1+	Welch and Ullman (2000)
albite	10	5	20-25	2	Oelkers and Scott (1998)
diopside	up to 10	6	25	1-2	Golubev and Pokrovsky (2006)
hornblende	10	4	25	3-4	Zhang <i>et al.</i> (1996)
Fe-chlorite	up to 10	4-5	25	1+	Hamer <i>et al.</i> (2003)

Notes: The DER for bytownite from the work of Welch and Ullman (2000) was barely discernable when compared with the runs in either deionised-water, or KNO₃. Accordingly, this DER is given as 1+. The DER value of 1+ listed for the work by Hamer *et al.* (2003) for Fe-rich-chlorite is based on Lowson *et al.* (2005) which indicated a value of 1.01. The DER for albite from the work by Oelkers and Scott (1998) is based on their model calculations. The DER from the work by Welch and Ullman (1993) is for labradorite.

The dissolution-enhancement ratio (DER) is based on a comparison of the dissolution rate obtained at the same pH and temperature values, and similar ionic-strength, but for:

- a solution containing varying concentrations of acetate ions, and undissociated acetic-acid; and
- a solution without acetic acid where mineral acids (e.g. HCl or HNO₃) are employed to control pH.

One of the key outcomes of the research findings shown in Table 3 is the low value of the resulting DER. The DER is typically within the range of 1-2 for pH values of 4-6 for [acetate]_{Total} concentrations up to approx 10 mM. This reflects the weakly complexing nature of acetate as a ligand (i.e. derived from an aliphatic, monocarboxylic acid). Use of dilute solutions containing acetic acid therefore results in minimal enhancement of primary silicate dissolution through complexing reactions (c.f. other carboxylic acids such as oxalic acid [Ganor *et al.* 2009]).

The "background-electrolytes" employed in the cited research are inferred to have Electrical Conductivity (EC) values less than 300 µS/cm, especially for runs at circum-neutral pH. In the pH5-soak tests, the feed-solutions have a [acetate]_{Total} of approx 5 mM, and an EC value of approx 300-400 µS/cm. The pH values of the feed-solutions and reacted-solutions are 5.0 (± 0.1), and 5.7-6.0, respectively.

Given the above, the DER is assumed to be 1.25, and raw rate data from the pH5-soak tests are divided by 1.25 to yield (circum-neutral) ACRs, reported in mg H₂SO₄/kg/day. The units of mg H₂SO₄/kg/day for the ACRs, and mg SO₄/kg/day for the SORs, permit direct comparison of the ACRs and SORs as a function of progressive weathering (viz. cumulative consumption of acid in the pH5-soak tests, and cumulative production of acid in the weathering columns).

In order to minimise the creation of reactive sites on the surfaces of primary silicates through particle-particle abrasion, a "stagnant-soak" approach was selected rather than the use of continuous agitation (e.g. bottle rolling) in the pH5-soak testing. Typically, 0.20 kgs of sample (< 2 mm) is placed in a plastic jar to which 1.5 kg of the pH5 feed-solution is added. After mixing via hand tumbling, the jar is left standing in a

constant temperature room (18-22 °C). Every 1-2 days, the jar is hand tumbled, the pH of the slurry is determined, and then left to stand again. This process is repeated until the pH of the slurry reaches 5.7-6.0 when the supernatant (i.e. reacted-solution) is decanted, weighed, and filtered, for analysis. The next lot of feed-solution is then added to the reacted solids remaining in the jar. The weight of feed-solution added is the same as that of the reacted-solution removed.

The acidity (in mg H₂SO₄/L) of the feed- and reacted-solutions are determined by titrating to pH 7.0 using a standardised NaOH solution; 1-2 drops of 5 % (v/v) H₂O₂ are added during the titration as the end-point is approached to ensure that any latent acidity associated with dissolved Fe(II) does not bias the "net-acid consumption" on the high side. Preparation of the feed-solutions is tightly controlled to ensure that the acidity (to pH 7.0) is 80 (± 5) mg H₂SO₄/L at pH 5.0 (± 0.1).

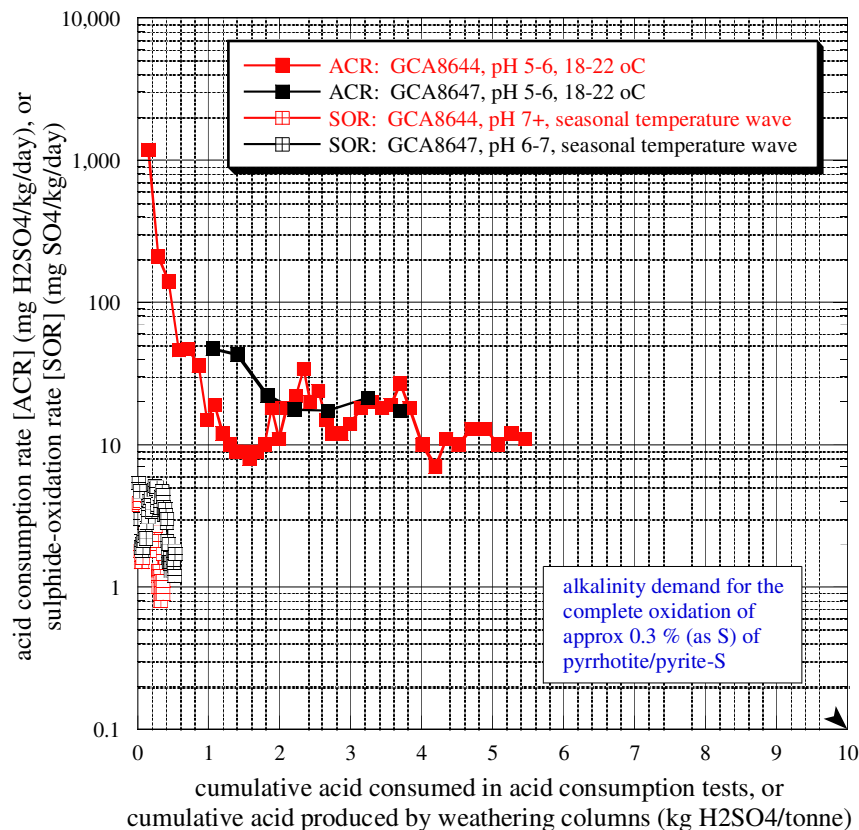
Results recorded to date

The variation in the ACRs for samples GCA8644 and GCA8647 are shown on Figure 4, together with the variation in the SORs for these samples, but expressed as a function of cumulative acid production over the 65 weekly weathering cycles. Also shown on this figure is the "alkalinity demand" required of the rock groundmass for the complete oxidation of 0.3 % (as S) as pyrrhotite, or pyrite. In order for circum-neutral conditions to prevail, the "pools" of alkalinity sources to meet this alkalinity demand need to exhibit ACRs that are at least as fast as the corresponding SORs.

The ACRs above approx 100 mg H₂SO₄/kg/day recorded initially for sample GCA8644 are ascribed mainly to acid consumption by traces of "bulk-carbonates". Thereafter, the ACRs for this sample, as well as those for sample GCA8647, reflect acid-induced hydrolysis, and/or dissolution, of silicate minerals. Silicate minerals of ultra-fine (near-µm) dimensions may be an important "sink" for acid, and source of circum-neutral buffering by the altered andesites/diorites at the NBGM. Mineralogical investigations are in hand to assist interpretation of the results from kinetic testing and identification of the reaction mechanisms involved.

In comparing the variation in the ACRs and SORs shown on Figure 4, due regard needs to be given to temperature differences. That is, the ACRs determined in a constant temperature room at approx 20 °C need to be compared with the peak-winter SORs which also correspond to a mean temperature of approx 20 °C. Results from the 2010 winter (after the initial "settling-down" period following commissioning of the columns, Figure 3), and the 2011 winter, indicate that the peak-winter SORs are approx 1-2 mg SO₄/kg/day. The corresponding ACRs (after decomposition of trace bulk-carbonates initially) have been approx 10-20 mg H₂SO₄/kg/day for a cumulative acid consumption up to 5-6 kg H₂SO₄/tonne (i.e. 50+ % of the alkalinity demand of approx 10 kg H₂SO₄/tonne sought for a cut-off-S value of 0.3 % for screening the andesites/diorites).

Figure 4. Pooled variation in acid consumption rates (pH5-soak tests) and sulphide oxidation rates (weathering columns) as a function of weathering history



Results from the 2010/11 summer indicate peak-summer SORs of approx 5 mg SO₄/kg/day, corresponding to mean temperatures of approx 30 °C. The pH5-soak tests will soon be running at 30 °C by storing the soak-jars in a water-bath in order to assess the influence of temperature on the ACRs.

Summary of Testwork Outcomes and Management Implications

Amoah *et al.* (2011) indicate that a sizeable portion of the andesite/diorite waste rock at the NBGM is characterised by trace quantities of sulphide-minerals, so that a cut-off-S value of 0.3 % for separating benign and reactive rock has major operational and cost benefits for the Project.

A review of historical geochemical studies on mine wastes at the NBGM was performed by Golder (2004), and it was concluded that the kinetic testing previously conducted was adequate for project planning. Since samples with total-S values as low as 0.1 % produced leachate-pH values near 4 on occasions during the testing period, it was recommended that all andesite/diorite waste rock should be treated as reactive, and thus require isolation. However, Amoah *et al.* (2011) pointed out that the suppression of leachate-pH values occurred during the summer months, and was more likely the result of using weathering columns containing coarse drill-core fragments that drained rapidly (e.g. within minutes) when flushed with deionised-water, thereby limiting the opportunity for circum-neutral buffering by the groundmass silicates. A combination of enhanced SORs during summer, and the limiting residence-time in the pervious rock-beds in the columns, was therefore considered the reason for the observed seasonal leachate-pH values near 4 for samples with total-S values of 0.1 %.

Residence-times during flushing were non-limiting for the kinetic testing performed in this study. Despite completing 65 weathering cycles, the cumulative amount of acid produced in the weathering columns is approx 0.5 kg H₂SO₄/tonne for samples with sulphide-S values up to 0.40 %. This extent of acid production corresponds to approx 5 % of the total amount of acid produced from the complete oxidation of 0.3 % (as

S) of pyrrhotite/pyrite-S (i.e. the alkalinity demand of approx 10 kg H₂SO₄/tonne). In contrast, the cumulative acid consumed during the pH5-soak tests ranges up to 5-6 kg H₂SO₄/tonne, or 50+ % of the alkalinity demand, and these tests have shown that the ACRs are "fast" compared with the (circum-neutral) SORs. Although the current testing programme is continuing, and additional samples are being tested to generate "ACR-envelopes" (to better assess variability), altered andesites/diorites with total-S values of at least 0.1-0.2 % should remain circum-neutral as the traces of pyrrhotite and pyrite fully decompose through weathering.

Though simple in design, the pH5-soak test is proving very useful in quantifying circum-neutral buffering properties (both kinetics, and capacity) of the altered andesites/diorites at the NBGM. It approximates, in essence, a batch-reactor (unagitated) operating in pH-stat mode. This test should also be useful at other sites where the acid forming characteristics of trace-sulphide/carbonate-deficient lithotypes need to be quantified. In its simplest form, the pH5-soak test quantifies the rates of acid-induced hydrolysis, and/or dissolution, of groundmass silicates (and carbonates where present). However, where there are sufficient amounts of pyrrhotite, oxidative dissolution, and pyrrhotite surface alteration, may be important side-reactions to contend with. In related testing of tailings-solids samples containing approx 0.7 % of pyrrhotite-S in a carbonate-deficient gangue (not from the NBGM), the reacted-solutions were Fe-rich (e.g. Fe(II) concentrations in the low tens-of-mg/L range). Also, monitoring of dissolved oxygen during the test showed that the reacted-solutions were anoxic (e.g. concentrations of dissolved oxygen of 1-2 mg/L). The release of Fe(II) forms to solution was accompanied by a marked increase in reactivity of the "leached-tailings", as shown when small subsamples of the tailings-solids were reacted with 15 % (v/v) H₂O₂. It is speculated that under the conditions of the test (viz. pH 5-6 in an acetate buffered system), the surface-zone of pyrrhotite grains altered to a marcasite-like composition (viz. "FeS" altering to "FeS₂"), as noted by Jambor (2003) in discussing the paragenesis and alteration of pyrrhotites *in situ*. In the surface spectroscopy work by Buckley and Woods (1985), pyrrhotite surfaces were shown to become depleted in Fe when reacted in solutions buffered by acetic acid at pH 3. However, side-reactions of this kind in the present study were of secondary importance for the waste rock samples tested herein.

Li (2000) argued that central to quantifying the pH regime during weathering of trace-sulphide/carbonate-deficient lithotypes is determining the rates of alkalinity release (chiefly from silicate sources) at circum-neutral pH, and coupling such alkalinity-release rates to acid-production rates from sulphide oxidation. Rates of alkalinity release (or acid consumption) from silicates may be either taken from the "single-mineral" dissolution literature for use in modelling studies, or measured for site-specific materials (i.e. determination of "mixed-mineral" dissolution rates). The pH5-soak tests employed herein correspond to the latter, experimental approach.

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Acronyms

ACR	Acid Consumption Rate
ANC	Acid Neutralisation Capacity
MPA	Maximum Potential Acidity
SOR	Sulphide Oxidation Rate