

**ADVANCES IN ACID DRAINAGE PREDICTION USING  
THE NET ACID GENERATING (NAG) TEST**

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## ABSTRACT

This paper describes the results of a completed 3 year research project carried out under industry sponsorship through the Australian Mining Industry Research Association (AMIRA). The project aims were to develop the static net acid generation test (NAG) test as a stand alone prediction test for pre-mining and operational monitoring uses and to evaluate the capability of the kinetic NAG test for predicting the lag period required for the development of acid conditions.

The study involved evaluation of a range of mining and processing wastes from gold, copper, lead, zinc, iron ore and coal mining operations. The results demonstrated that the static NAG test, using hydrogen peroxide as an oxidant, is a reliable stand alone predictive tool that can also be used to in conjunction with the net acid producing potential (NAPP) to significantly reduce the uncertainty which is inherent with acid base accounting procedures. The study also confirmed the robustness of the test for non-coal operations but highlighted the need for caution with coal wastes containing a high content of organic material.

The simplicity of the static NAG test and the short turn-around of the test procedure is ideal for field use. The test and assessment criteria are amenable to site specific calibration for identifying specific material types appropriate for the site and waste management operation. The static NAG test is now routinely used in the field at a number of mining operations within the Asian Pacific region and is well accepted by operators and regulatory authorities.

The development of the kinetic NAG test as a tool for predicting the lag period or exposure period required for the development of acid conditions in a mine waste samples was a significant outcome of the research. The results showed that for potentially acid forming samples, there was a direct relationship pH trend observed during the kinetic NAG test (in minutes) and the time (in weeks) for the pH of column weathering test leachates to fall to pH 4. This relationship and the appropriate evaluation procedure are discussed in the paper.

## Keywords

Acid drainage, net acid generation (NAG) test, net acid producing potential (NAPP), lag period prediction, Asia Pacific region.

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## 1.0 INTRODUCTION

This paper describes the results of a 3 year research project carried out under industry sponsorship through the Australian Mining Industry Research Association (AMIRA) to develop the net acid generation (NAG) test as a simple low cost procedure for the identification and characterisation of acid forming mine rock and process residues.

The companies sponsoring this research were Billiton Australia, BHP Australia Coal Limited, Carpentaria Gold Pty Limited, Hamersley Iron Pty Limited, Peak Gold Mines Pty Limited, Placer Pacific Limited, P.T. Kelian Gold Equatorial Mining, and Western Mining Corporation Limited - Kambalda Nickel Operations.

The objectives of the study were to:

- Evaluate and develop the static NAG procedure as a simple low cost test for identifying acid-forming mine rock and waste materials for use in premining and operational phases of mining ventures;
- Evaluate the suitability of the NAG test for operational monitoring and identification of acid-forming materials; and
- Investigate the kinetics of the NAG test reaction for predicting the field lag period (i.e. the exposure period required for acid conditions to develop under natural weathering conditions).

## 2.0 STUDY METHODS

### 2.1 Sample Selection and Preparation

A total of 119 samples representing 17 mine sites were received from the 8 sponsor mining companies. Samples included waste rock, ore, process residues (both fresh and weathered) and coal reject. The range of samples included in the project are displayed in Table 1.

*Table 1: Samples provided for acid-base and NAG testwork program.*

Sample Type	Number of Samples	Number of Mines	Sample Numbers by Mine Type				
			Gold	Iron Ore	Nickel	Base metal	Coal
Process Residues and Reject	23	11	8	0	11	1	3
Waste Rock	78	15	54	8	6	3	7
Ore	18	8	13	0	3	2	0
<b>TOTAL</b>	<b>119</b>		<b>75</b>	<b>8</b>	<b>20</b>	<b>6</b>	<b>10</b>

Between 2 and 20 kg of each sample was received and prepared on the basis of sample type. For samples of waste rock, reject and ore received by EGi, approximately 4 kg was sub-sampled and crushed to nominal < 10 mm. About 1 kg of this sub-sample was then crushed to < 4 mm and a final 250 g sample pulverised to approximately 200 Mesh (<75µm). Process residue samples were normally received in the form of slurries which were air-dried for a week and then sub-sampled.

## 2.2 Laboratory Methods

All samples were analysed in duplicate for total sulphur, pH, electrical conductivity (EC) (on a 1:2 sample:deionised water extract), acid neutralising capacity (ANC) and static net acid generation (static NAG). Based on the results of the characterisation work, 33 selected samples were analysed using the kinetic NAG test. A summary of the testing procedures is presented below.

*pH and EC.* The pH and EC were determined on a 1:2 (sample:deionised water) slurry. 50 g of pulverised sample was immersed in 100 mL of deionised water with mixing. The mixture was allowed to stand for 12 hours and the pH and EC measured directly on the slurry.

*Acid neutralising capacity (ANC).* The ANC of the sample was determined by a method based on Sobek et. al. (1978) which involves adding a known amount of standardised hydrochloric acid to 2.0 g of sample and allowing the sample time to react with heating, then back-titrating the cooled mixture with standardised sodium hydroxide to pH 7.0. The amount of acid consumed by reaction was then used to calculate the ANC.

*Total Sulphur.* Total sulphur determinations were carried out by Enviromet Ltd (Sydney, Australia) using a Leco Automatic Sulphur Analyser according to Australian Standard Method AS1038 Part 6.3.3.

*Net Acid Producing Potential (NAPP).* The Net Acid Producing Potential (NAPP) is calculated by from the maximum potential acidity (MPA) and ANC using the following equation (Miller and Murray, 1988):

$$\text{NAPP (kg H}_2\text{SO}_4\text{/t)} = \text{MPA} - \text{ANC}$$

where:

$$\text{MPA} = S_{\text{pyr}} (\%) \times 30.6$$

$$\text{ANC units kg H}_2\text{SO}_4\text{/t}$$

Note: if pyritic sulphur ( $S_{\text{pyr}}$ ) is not determined directly on the sample then total S or sulphide S (total sulphur less sulphate and organic sulphur) can be used for an initial estimate of NAPP.

Total S will over estimate the NAPP if significant amount of sulphate S, organic S or other non-pyritic forms occur in the sample.

*Static Net Acid Generation test (Static NAG).* 250 mL of 15 % H<sub>2</sub>O<sub>2</sub> (unstabilised 30 % H<sub>2</sub>O<sub>2</sub> diluted 1:1 with deionised water) was added to a 2.5 ± 0.1 g sample of pulverised waste rock or tailings contained in a 0.5 litre conical flask. Oxidation of sulphides in the sample generated heat and samples were considered to have finished reacting when: (1) the sample solution boiled and then cooled, or (2) after at least 12 hours had elapsed. Any excess H<sub>2</sub>O<sub>2</sub> left in the sample solution was removed by heating on a hotplate at 110 °C until all effervescence ceased. The pH of the solution was measured after cooling to ambient temperature and the solution was titrated with standardised NaOH (0.100 N NaOH if pH > 2.5 and 0.500 N NaOH if pH < 2.5) to pH 4.5 using an Orion 960 autochemistry system. Note: titration to pH 4.5 and pH 7 can provide additional information for sample characterisation. The NAG<sub>pH 4.5</sub> value indicates the contribution from free acid, Al and Fe while the NAG<sub>pH 7</sub> provides information on the additional acidity due to other metals such as Cu and Zn. NAG<sub>pH 4.5</sub> is used to determine the NAG capacity reported in this paper.

The NAG test is based on hydrogen peroxide oxidation procedure developed by Finkelman and Giffin (1986) for determining the pyrite content of coal overburden. A number of other researchers have also investigated the use of peroxide for assessing sulphide content and acid potential of mine waste materials (Smith *et al.*, 1974; Sobek *et al.*, 1978; O'Shay and Hossner, 1984; O'Shay *et al.*, 1990). Coastech Research Inc. (1989) applied the test as a prediction method to estimate the net acid production from waste rock and tailings.

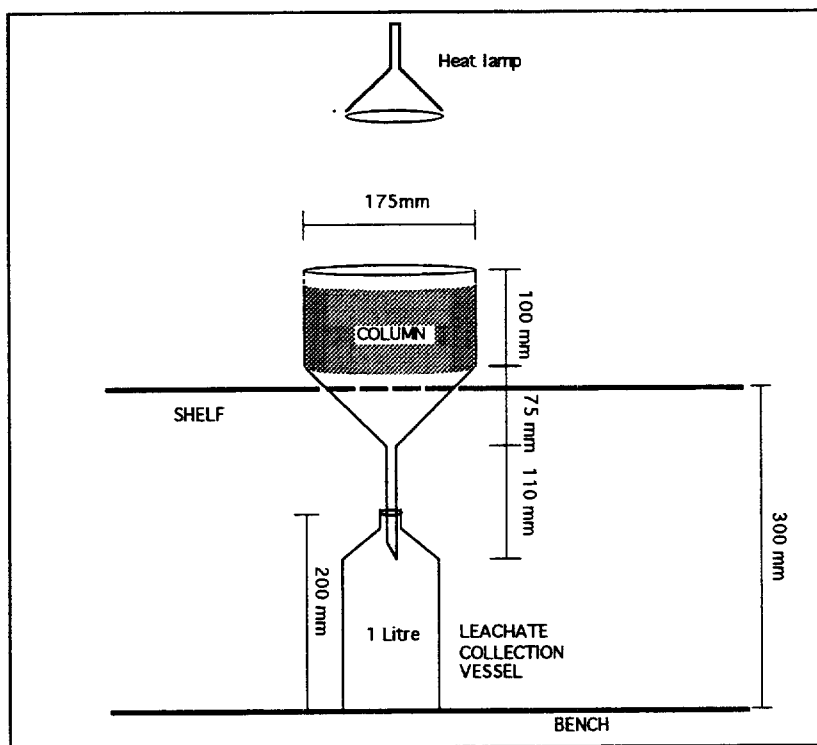
*Kinetic Net Acid Generation (Kinetic NAG) test.* The procedure described previously for the NAG test was again utilised, however, pH and temperature probes were immersed in the sample solution and the pH, temperature, and time were continually recorded. The starting temperature for the kinetic NAG test is 20°C.

*Mineralogical Study.* A total of 14 crushed mine rock samples were submitted to Harris Exploration Services, North Vancouver, B.C., Canada for petrographic and mineralogical examination. The mineralogical proportions in each sample (with special reference to sulphides and carbonate) were estimated by microscopic point counting. In those samples where carbonate occurred in sufficient abundance, the carbonate species was determined by partial XRD scans.

*Column leaching test work.* A schematic of the leach column set-up is displayed in Figure 1. For the column leach test, tailings samples were loaded in the as-received particle size condition while waste rock samples were crushed to minus 4 mm size. Approximately 2 kg of sample was placed in each column and leached with deionised water at a rate of 100 mL/kg/week for 3 weeks and 400 mL/kg/week on the fourth week. The water was added on the same day each week (Friday) and the leachate collected on the Monday after the fourth week addition. Between water additions, each column was exposed to a heat lamp to dry the column and allow excess oxygen

into the material. The column leachate was analysed for pH, EC, acidity or alkalinity, Fe, Ca, Mg and SO<sub>4</sub>.

Figure 1: Leach column set-up for kinetic test work



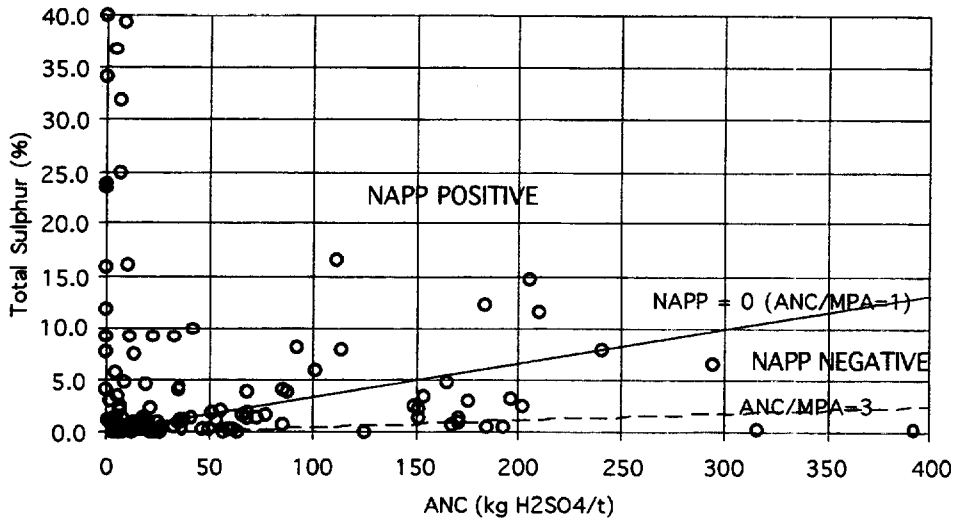
### 3.0 NAPP AND STATIC NAG TEST RESULTS

#### 3.1 NAPP Characteristics.

Total sulphur results for all samples range from 0.01 to 40 % S; ANC from 0 to 392.4 kg H<sub>2</sub>SO<sub>4</sub>/t; NAPP from -388.7 to 1,224 kg H<sub>2</sub>SO<sub>4</sub>/t; and NAG values ranging from 0 to 309 kg H<sub>2</sub>SO<sub>4</sub>/t. The NAPP results for all the samples are displayed on a conventional acid-base plot in Figure 2 with total sulphur (%) plotted against ANC (kg H<sub>2</sub>SO<sub>4</sub>/t). The NAPP = 0 line shown on the figure defines the boundary between NAPP positive and NAPP negative samples. NAPP=0 is equivalent to ANC/MPA=1. The results illustrate the wide range of samples included in the study and indicate that 60 samples represent NAPP negative materials and 59 samples represent NAPP positive materials. The majority of the samples fall in the range of total S <10% and ANC

< 200 kgCaCO<sub>3</sub>/t. The ANC/MPA =3 line is shown on the figure and identifies a number of samples which fall in the ANC/MPA range from 1 to 3.

Figure 2: Total Sulphur versus ANC of all Test Samples



### 3.2 Relationship between NAPP and NAG data.

The relationship between the pH of the NAG solution after reaction (NAGpH) and the NAPP value for all samples is shown on Figure 3 and Figure 4 shows the relationship for the NAPP range -200 to +200 kg H<sub>2</sub>SO<sub>4</sub>/t. The results show a clear NAGpH separation between NAPP negative and NAPP positive materials with NAPP positive materials having a final NAGpH generally less than 4 and NAPP negative materials having a final NAG pH generally greater than 4 with most having NAGpH values greater than 6.

The two NAPP negative samples with NAGpH less than 4 (see Figure 4) are coal reject samples and the low pH is possible due to peroxide reaction with organics. The three outliers which have NAPP values greater than 40 kg H<sub>2</sub>SO<sub>4</sub>/t but NAGpH less than 4 are from a single mine site with high S (4 to 15%S) and high ANC (80 to 210 kg H<sub>2</sub>SO<sub>4</sub>/t characteristics). For these outlier samples, test work demonstrated significant variability in repeat analysis and with and without boiling. This suggests a complex mineralogy and mineral assemblage which was confirmed by mineralogical and petrographic investigations identifying a variable content of ankerite [Ca(Mg,Fe,Mn)(CO<sub>3</sub>)<sub>2</sub>] and a veining pyrite occurrence. The results highlight the need for particular care when interpreting the results for samples with high sulphur contents and high ANC's. Field behaviour of these material types can be highly dependent on particle size and scaling up from laboratory test work must be interpreted with caution.

Only 8 of the 119 samples tested had NAGpH values between 4 and 6, and 5 of these were NAPP negative.

Figure 3: NAGpH versus NAPP for All Samples

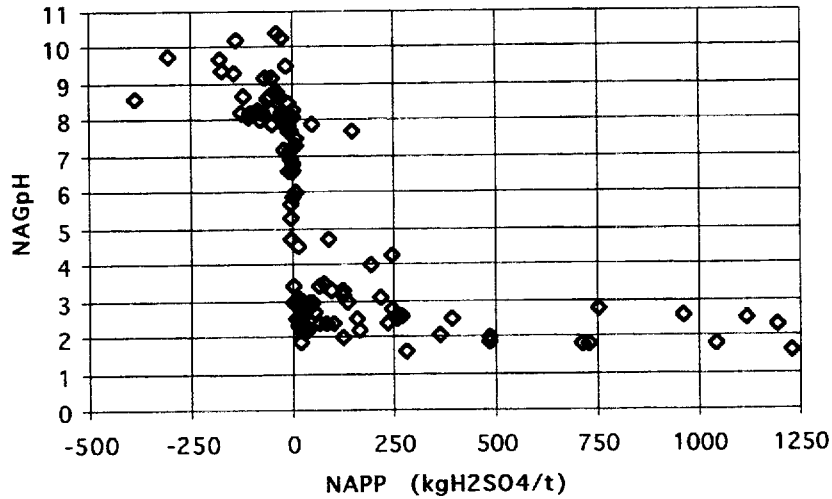
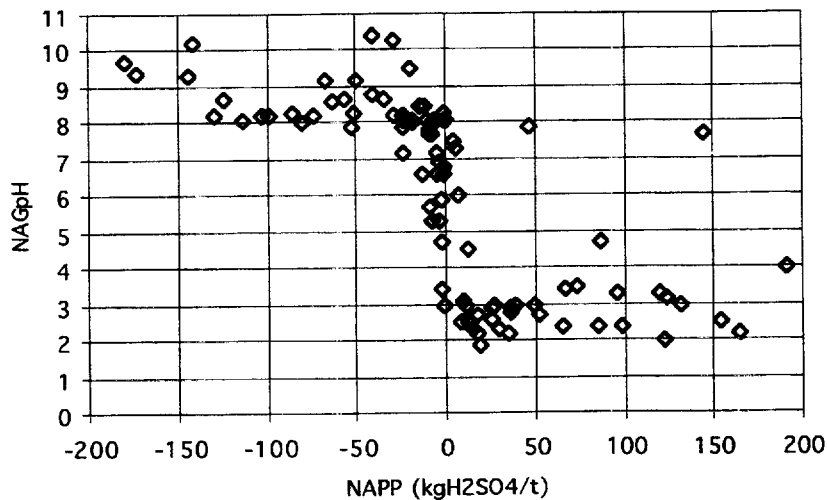


Figure 4: NAGpH versus NAPP for NAPP Range -220 to +200 kg H<sub>2</sub>SO<sub>4</sub>/t



The NAGpH is an ideal parameter for confirming the NAPP calculation results. Outliers are identified and can be further evaluated to determine sulphur forms and carbonate and sulphide mineralogy. The NAG capacity is determined by titration of the sample to determine the net amount of acid generated by peroxide oxidation. For many samples the NAG capacity is less



than the calculated NAPP but should not be considered as an alternative the NAPP value. The NAG capacity is an independent measure of the acid generating potential of a sample.

### 3.2 Static NAG Test Procedures and Material Classification Criteria

Investigations into some of the major experimental variables involved in the NAG test were carried out in order to ensure uniform applicability of the test to the complete range of acid generating materials (and conditions) likely to be found in the field. Some of the most important variables likely to affect the NAG test are temperature, sample:solution ratio, H<sub>2</sub>O<sub>2</sub> concentration and particle size.

Evaluation of the NAG test procedure indicates that temperature, concentration of H<sub>2</sub>O<sub>2</sub>, sample:solution ratio and particle size do not significantly affect the final NAG pH, but can influence the reaction kinetics and amount of acid generated as indicated by the pH and temperature profiles of the kinetic NAG test results. The results suggest that the H<sub>2</sub>O<sub>2</sub> reagent should be added at a constant starting temperature in order to improve the precision of the kinetic NAG test and facilitate a valid comparison of data acquired for a wide variety of samples. The results also illustrate that for a constant mass of sample, the amount of acid produced in the NAG test is a function of the particle size whereas the final NAGpH is effectively independent of particle size.

The findings of this research project have demonstrated that the NAG test is a fast, convenient and low-cost experimental prediction technique for identifying and classifying the acid potential of mine rock and process waste materials. The procedure is robust and adaptable to field use for operation monitoring and identification of waste rock types (Miller et.al.1991). Attachment A presents the Static NAG procedure developed by the study and Table 2 below presents the classification criteria for the initial interpretation of the static NAG test results.

Table 2: Material Classification Criteria

SAMPLE CATEGORY	FINAL NAGpH	NAG VALUE	NAPP
		(kg H <sub>2</sub> SO <sub>4</sub> /t)	
<b>POTENTIALLY ACID FORMING</b>			
Higher capacity	< 4	> 10*	Positive
Lower capacity	< 4	≤ 10*	•
<b>NON-ACID FORMING<sup>@</sup></b>	≥ 4	0	Negative
<b>UNCERTAIN<sup>#</sup></b>	≥ 4	0	Positive

\*recommended that 5 is used for some coal mining situations

<sup>#</sup>Further evaluation including sulphur forms and mineralogy

<sup>@</sup> Acid consuming materials are identified by NAPP values less than about -100.

#### 4.0 KINETIC NAG TEST AND LAG TIME PREDICTION

Kinetic NAG tests and column leach test were carried out on 33 selected samples and some of the column tests are continuing. The samples selected included thirteen non-acid generating samples, six low capacity potentially acid generating samples and fourteen high capacity potentially acid generating samples. In the kinetic NAG test the pH and temperature of the sample are monitored continually.

The temperature and pH profiles varied widely and Figure 5 represents a material which is fast reacting showing a rapid drop in pH and a temperature peak occurring after about 35 minutes. Figure 6 also represents a potentially acid forming material but the pH trend indicates a significant buffering capacity and the temperature peak did not occur until after about 375 minutes of reaction time.

Figure 5: Kinetic NAG Test pH and Temperature Trends: PAF - Fast Reacting Sample

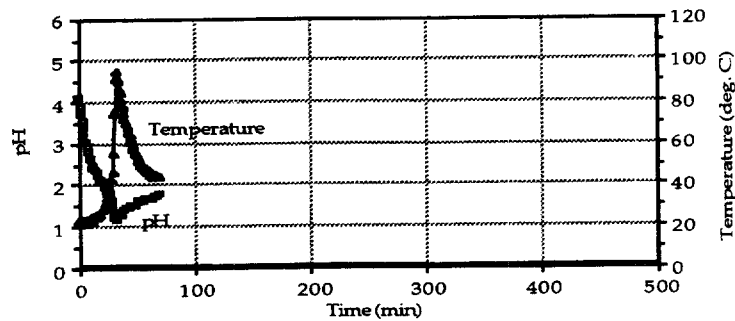
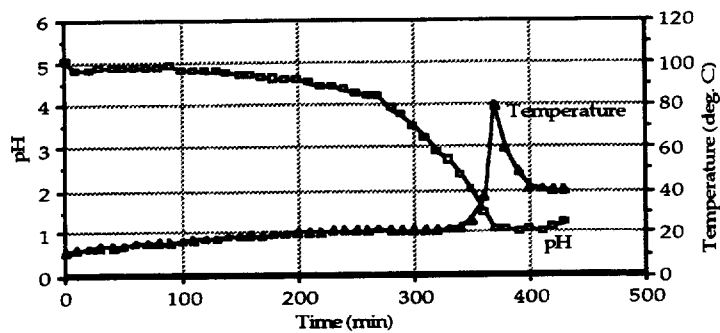


Figure 6: Kinetic NAG Test pH and Temperature Trends: PAF - Slow Reacting Sample

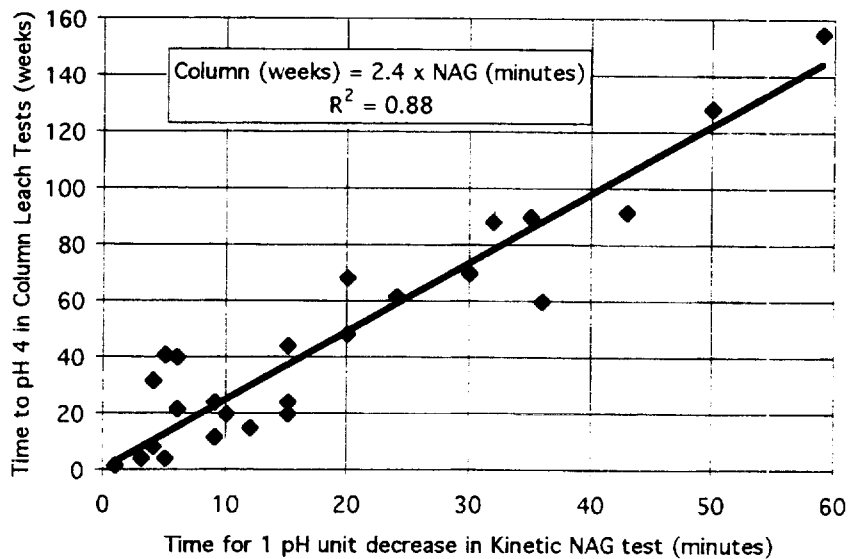


The kinetic NAG test results were compared with the column test results to determine possible correlations between the kinetics of the NAG test and kinetics of the column leach tests. Correlations with ANC and NAPP were also investigated.

Regression analysis identified that the time for the pH of column leachates to fall to pH 4 was directly related to the time for the pH of the kinetic NAG test solution to fall 1 unit. The  $R^2$  for this relationship was 0.88 and is shown on Figure 7. The  $R^2$  for ANC and NAPP versus the time for the pH of column leachates to fall to pH 4 were only 0.14 and 0.01, respectively and hence were not good indicators of the column lag period for the sample set used in this test work.

The regression line in Figure 7 is consistent with a fairly robust relationship taking into account the diverse nature and origin of the samples included in the test work.

*Figure 7: Relationship Between NAG Reaction Kinetics and Lag Period in Leach Columns for Potentially Acid Forming Samples*



The leach column tests were conducted in accordance with the procedures outlined earlier in this paper. The columns were set up to represent conditions on the surface of waste dumps where oxygen is readily available and unsaturated conditions apply. If it is assumed that the leach column results obtained for the samples included in Figure 7 can be extrapolated to the field situation, then there is a basis for using the kinetic NAG test results for prediction of the lag period for materials in the field. With the current level of knowledge, the kinetic NAG test should only be considered indicative for predicting the lag period and identifying fast and slow reacting materials for operational management aspects. However, as more data becomes available, the relationship between the kinetic NAG test, column leach test results and field behaviour will

be further developed providing essential information for managing potentially acid forming material types and further reducing the risk of acid drainage (Miller and Jeffery, 1995).

## **5.0 CLASSIFICATION OF GEOCHEMICAL MATERIAL TYPES FOR MINE WASTE MANAGEMENT**

The results of the NAPP and static NAG tests allow samples to be classified, according to their acid forming potential. Four main categories are defined by the NAPP and static NAG test results as follows:

- Acid consuming (ACM)
- Non-acid forming (NAF)
- Potentially acid forming -low capacity (PAF-L.C.)
- Potentially acid forming - high capacity (PAF-HC)

Acid consuming (ACM) materials are identified either by geology (e.g. limestone) or by the NAPP determination. For practical purposes, ACM materials need to have an excess ANC of the order of 100 kgH<sub>2</sub>SO<sub>4</sub>/t (equivalent to about 10% CaCO<sub>3</sub>) and a sulphide S content less than about 0.5%S.

Non-acid forming (NAF) materials have NAGpH values greater than 4 and generally negative NAPP values whereas potentially acid forming materials have NAGpH values less than 4 and positive NAPP values. Samples which have conflicting NAPP and NAG results must be investigated further. The PAF low capacity and high capacity materials are separated on the basis of the acid capacity as determined by the NAG test. Low capacity samples represent materials that may be amenable to treatment by blending or light lime application. The low capacity category is generally for materials which have the potential to generate less than about 5 to 10 kgH<sub>2</sub>SO<sub>4</sub>/t.

The kinetic NAG test can also be used to categorise materials on the basis of their lag period or risk of acid generation within a given period of time. This information can then be used to refine the waste management operation to prevent acid generation during the operation and prior to implementation of long term control strategies such as placement of an engineered cover, oxygen flux barrier or infiltration barrier.

The NAG test is now commonly used at mining operations throughout the Asia-Pacific region. The test provides a direct measure of the acid potential and allows various categories of acid forming materials to be defined for management flexibility. The test is simple, low cost and has proven field applications. Further developments are planned which are likely to include a modification to the NAG test for identifying metal leach materials. This is important particularly

for waste rock that may not be acidic but may leach significant quantities of metals such as Cu, Zn and Mn even at near neutral pH.

The NAG test is a useful tool for confirming NAPP test results and investigating and monitoring the acid potential of mine waste materials. However it should not be considered as an alternative to the NAPP approach but as an independent measure of the acid generating potential of a sample.

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**ATTACHMENT A**  
**RECOMMENDED STATIC NET ACID GENERATION**  
**(NAG) PROCEDURE**

**1. Sample Preparation**

Drill core and bulk rock samples should be crushed to nominal 4 mm and a sub sample pulverised to approximately 200 Mesh (<75µm). Tailing and process residue samples can be tested 'as received'.

**2. Reagents**

Reagent 1: H<sub>2</sub>O<sub>2</sub> - BDH 'Analar' Analytical Reagent 30% w/v (100 V), or equivalent, diluted 1:1 with deionised H<sub>2</sub>O to 15%. (Refer to Note 1).

Reagent 2: NaOH - 0.50 M Standardised Solution.

Reagent 3: NaOH - 0.10 M Standardised Solution.

**3. Method**

- a) Add 250 mL of reagent 1 (15 % H<sub>2</sub>O<sub>2</sub>) to 2.5 g of pulverised sample in a 500 mL wide mouth conical flask, or equivalent. Cover with a watchglass, and place in a fumehood or well ventilated area (refer to note 2). The H<sub>2</sub>O<sub>2</sub> should be at room temperature before commencing test.
- b) Allow sample to react until 'boiling' or effervescing ceases. Heat sample on hot plate and gently boil until effervescence stops or for a minimum of 2 hours. Do not allow sample to boil dry - add deionised water if necessary.
- c) Allow solution to cool to room temperature then record final pH (NAGpH).
- d) Rinse the sample that has adhered to the sides of the flask down into the solution with deionised water. Add deionised water to give a final volume of 250 mL.
- e) Titrate solution to pH 4.5 while stirring with the appropriate NaOH concentration based on final NAG solution pH as follows:

<u>NAG Solution pH</u>	<u>Reagent</u>	<u>NaOH Concentration</u>
> 2	2	0.10 M
≤ 2	3	0.50 M

**4. Calculation**

Net Acid Generation

$$\text{NAG} = \frac{49 \times V \times M}{W}$$

where:

- NAG = net acid generation (kg H<sub>2</sub>SO<sub>4</sub>/tonne)  
V = volume of base NaOH titrated (mL)  
M = molarity of base NaOH (moles/l)  
W = weight of sample reacted (g)

NOTE: If NAG value exceeds 25 kg H<sub>2</sub>SO<sub>4</sub> per tonne, repeat using a 1.00 g samples.

#### NOTES AND PRECAUTIONS

1. The pH of the H<sub>2</sub>O<sub>2</sub> used in the NAG test should be checked to ensure it is between pH 4 and 7. If the pH is less than 4 then add dilute NaOH (use a solution made up by adding 1 g NaOH to 100 mL deionised H<sub>2</sub>O) until the pH is greater than 4 (aim for a pH between 4 and 6). The pH is adjusted to greater than pH 4 to ensure that the phosphoric acid, used to stabilise H<sub>2</sub>O<sub>2</sub> in some brands, is neutralised. The pH of the 15 % H<sub>2</sub>O<sub>2</sub> should always be checked to ensure that any stabilising acid is neutralised, otherwise, false positive results may be obtained.
2. The NAG reaction can be vigorous and sample solutions can 'boil' at temperatures of up to 120 °c. Great care must be taken to place samples in a well ventilated area or fume cupboard.
3. Caution should be taken in the interpretation of NAG test results for coal reject samples and other materials which may contain a high content of organic material (such as potential acid sulphate soils, dredge sediments and other lake or marine sediments). All organic material must be completely oxidised otherwise acid NAG results can occur which are unrelated to sulphides. Several aliquots of H<sub>2</sub>O<sub>2</sub> reagent may be added to the sample to breakdown any organic acidity.
4. Samples with positive NAPP value, high sulphur content and high ANC must be carefully evaluated.