

Difficulties of Interpretation of NAG Test Results on Net Neutralizing Mine Wastes: Initial Observations of Elevated pH Conditions and Theory of CO₂ Disequilibrium

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ABSTRACT

Hydrogen peroxide leach tests (NAG tests) are increasing in use by the mining industry for determining acid rock drainage characteristics of mine wastes. The test uses an excess of hydrogen peroxide (H₂O₂) to rapidly oxidise sulfide minerals in a sample, thus releasing acidity related to sulfide oxidation. The acidity released is neutralized by available (carbonate) neutralizing potential, theoretically resulting in a measure of Net Acid Generation (NAG) based on material reactivity. In addition, chemical analysis of the NAG leachates can be undertaken to provide an indication of solutes that may be mobilised during sulfide weathering.

NAG tests are complimentary tests to standard Acid Base Accounting (ABA), allowing clarification of ABA characteristics and better characterization of samples that show uncertain characteristics based on ABA testwork. However, experience has shown that the use of the NAG test method on samples with elevated carbonate content can result in excessively basic pH conditions which may be misleading and result in uncertainty in interpretation.

To understand the uncertainty mentioned above, tests were completed to measure and evaluate the pH change throughout the NAG test method. The results indicate that much of the pH change can be attributed to carbon dioxide (CO₂) disequilibrium during the heating stage of a NAG test. The study is supported by diagnostic mineralogical and geochemical investigation. In addition, the influence of CO₂ on NAG test pH conditions is demonstrated through numerical interpretation using the USGS thermodynamic equilibrium code PHREEQC.

Keywords: ARDML, NAG, PHREEQC, carbon dioxide, hydrogen peroxide

INTRODUCTION

Environmental concerns coupled with the strengthening of environmental legislation, has necessitated the development a range of testwork methods for the prediction of Acid Rock Drainage and Metal Leaching (ARDML) from mine wastes. One of the most accepted and widespread methods is the Acid Base Accounting (ABA) test, which is used to determine the balance between acid generation potential (AP) and acid neutralization potential (NP) of a material based on sulfide and carbonate content, respectively. Recently, the complimentary NAG) test method has seen increasing use within the industry. This test allows for the rapid determination of ARD characteristics of tailings and waste rock samples through the use of hydrogen peroxide (H₂O₂). The typical NAG test procedure is based upon H₂O₂ oxidation testing that has been developed over the past 40 years by: Smith *et al.* (1974), Sobek *et al.* (1978), O'Shay & Hossner, (1984), Finkelman & Giffin (1986), O'Shay *et al.*, (1990), Miller *et al.* (1997), and most recently by Stewart *et al.* (2003); Stewart (2005) and Stewart *et al.* (2006).

Solutions generated during the NAG test can additionally be analysed to provide an indication of metal and metalloid (e.g. zinc, copper, lead, cadmium, arsenic) mobility during weathering and oxidation of the waste. Furthermore, there are possible advantages to using NAG tests and derivations of the NAG test (such as kinetic NAG) as a substitute for kinetic tests (Miller *et al.*, 1997; Stewart *et al.*, 2006; Sapsford *et al.*, 2010) and for preliminary prediction of seepage water quality from mining waste facilities (Barnes *et al.*, 2013; Barnes *et al.*, 2015).

Although NAG tests allow for improved understanding of ARDML characteristics and classification of samples that show uncertain acid generating characteristics from ABA testing, it is not without its limitations. These limitations have been highlighted in studies undertaken on the NAG testing of coal spoils by Stewart, (2005) and Stewart *et al.*, (2006). In addition, other authors have highlighted the sensitivity of the oxidising agent H₂O₂ to: base metal cations and dissolved carbonates (Lee *et al.*, 2000 and references therein); and to elevated reaction temperatures (Yazici & Deveci, 2010).

Further to the above limitations with respect to the instability of H₂O₂, it has also been observed by the authors that the use of the NAG test method on samples with elevated transition metal carbonate content (such as ankerite) can produce excessively basic NAG pH conditions which may be misleading and result in uncertainty in interpretation. This paper intends to shed light on the mechanisms that induce the generation of excessively basic solution. It is hypothesised that the elevated pH conditions can be attributed to carbon dioxide (CO₂) disequilibrium during the heating stage of a NAG test.

METHODOLOGY

Ten waste rock samples were collected from a gold mine site in the UK. Local geology at the site consists of clastic marine sediments, minor volcanic units, sandstone and auriferous quartz-carbonate veins. The major gold-bearing formation comprises mixed semi-pelites, quartz semi-pelites, and psammites. The project area contains mesothermal gold mineralization, with gold disseminated in quartz-sulfide veins.

Acid base accounting testwork

ABA testing was undertaken in accordance with the method described in EN15875 (CEN, 2011). The method assesses the speciated carbon and sulfur content of the samples to determine the balance of acid generating sulfides (AP) and acid neutralizing carbonate minerals (NP). ABA testing does not make allowances for mineralogy and assumes all sulfide is present as pyrite and all carbonates are present as calcite. AP is calculated from the sulfide sulfur content of the samples whilst the NP is based on a modified Sobek procedure in which acid addition is determined from the Total Inorganic Carbon (TIC) content of the sample. The determination of a samples AP and NP in kg CaCO₃ equivalents per tonne allows for the calculation of net neutralizing potential (NNP) and neutralization potential ratio (NPR) which describe the samples potential for acid generation:

$$\text{Net Neutralizing Potential (NNP)}(\text{kg CaCO}_3) = \text{NP} - \text{AP}$$

$$\text{Neutralization Potential Ratio (NPR)} = \text{NP} / \text{AP}$$

An assessment of paste pH was also undertaken to determine the immediate sample pH without oxidation of the sample.

Mineralogical analysis

A mineralogical investigation was undertaken on the samples to assist in interpretation of the NAG test results. This consisted of optical and scanning electron microscopic (SEM) analysis and spectroscopic analysis using SEM electron dispersive X-Ray (EDX) analysis of grab samples from the drill core sample intervals. The investigation focussed on quartz, carbonate, and sulfides within mineralized veins.

Net acid generation testwork

The static NAG test differs from the ABA test in that it provides a direct empirical estimate of the overall sample reactivity, including any acid generated by semi-soluble sulfate minerals (e.g. alunite), as well as other potentially acid-generating sulfur-bearing minerals. As such, the NAG test provides a better estimate of potential for field acid generation than the more widely-used ABA methods. ABA defines acid potential based solely on sulfide sulfur, or total sulfur content, and assumes pyrite to be the dominant sulfide mineral present.

NAG testing was carried out in accordance with the method described in the AMIRA guidelines (2002) as follows: 250 mL of 15% H₂O₂ was added to 2.5g of pulverised sample. The H₂O₂ was allowed to react with the sample overnight, and the following day the NAG solution was gently heated, using a hot plate to near boiling for a minimum of 2 hours; deionised water was added as required to maintain an approximately constant volume of solution. The NAG solution is heated to remove excess H₂O₂ and also facilitate the release of inherent neutralizing capacity (carbonate buffering). The solution was then allowed to cool to room temperature, and at this point the NAG pH was measured and recorded. The test also reports the NAG value, measured in equivalent kilograms of sulfuric acid (H₂SO₄) per tonne.

The above method was adapted for the test samples in order to track the pH change through the latter portion of the test. pH was measured at the following times to determine the evolution of solution pH:

- *Step 1:* Prior to solution heating following completion of NAG test procedure (20 °C)
- *Step 2:* Following 2 hour heating step (at approximately 100 °C) after the solution is allowed to cool to room temperature (20 °C) - and then made up with deionised water - typical NAG pH
- *Step 3:* After solution allowed to equilibrate with atmosphere for 24 hours
- *Step 4:* After solution allowed to equilibrate with atmosphere at room temperature for 1 week.

The leachate generated during the NAG test was analysed for major ions, dissolved metals and metalloids using inductively coupled plasma mass spectrometry (ICP-MS). The aggressive oxidising conditions of the H₂O₂ used in the NAG test, effectively oxidises all physically exposed or chemically available sulfide minerals. Analysis of the NAG solution provides an indication of the potential for high level metal(loid) release that would occur during exposure of the mine-waste material to oxygen.

Thermodynamic equilibrium modeling

Thermodynamic equilibrium calculations were carried out to validate and numerically recreate the observations of the NAG testwork and demonstrate the influence of CO₂ on NAG pH. Thermodynamic calculations were carried out using the geochemical modelling code PHREEQC, version 3.1.2.8538 (Parkhurst & Appelo, 1999) together with the MINTEQ v8 database. This database is largely consistent with the MINTEQ v4 provided with PHREEQC, including updated thermodynamic data for arsenic and manganese. The NAG solutions defined in TABLE 1 were run initially in PHREEQC without any carbonate equilibrium phases, then the same simulations were run with CO₂ at atmospheric pressure (CO₂ partial pressure of 0.0003 bar). Forty PHREEQC simulations (four per initial starting solution; TABLE 1) were carried out in order to replicate the NAG experiment as described below:

- *Simulation 1:* Initial solution – at room temperature (20 °C)
- *Simulation 2:* Boiling initial solution (100 °C) - degassing of CO₂ in solution to atmospheric equilibrium
- *Simulation 3:* Cooling of solution back to room temperature (20 °C) – system at disequilibrium with respect to CO₂ and carbonate minerals with CO₂ in solution much lower than equilibrium CO₂ concentration
- *Simulation 4:* System allowed to reach equilibrium with respect to CO₂ (20 °C)

The input solution chemistries outlined in TABLE 1 were experimentally derived from the NAG test. Bicarbonate alkalinity was not measured directly and was determined via major ion charge balance.

Table 1 PHREEQC initial input solutions derived from NAG leachate analysis

Sample ID	1	2	3	4	5	6	7	8	9	10
Temp (°C)	20	20	20	20	20	20	20	20	20	20
units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
NAG pH (pre boiling)	7.03	7.38	7.21	7.42	7.22	7.35	7.22	7.00	7.37	7.12
HCO ₃ ⁻	49.5	57.9	56.2	50.9	48.4	52.3	50.1	29.9	39.2	21.0
SO ₄	1	1.2	6	3.8	1.6	1	11	1	1	1
Ca	18	19	22	19	18	17	22	12	15	8.6
K	1.9	4.8	2.7	3	2.1	4.6	3.5	1.4	2	2.5
Mg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Na	0.5	1.1	0.5	0.5	0.5	1.6	1	0.5	0.5	0.5
As	0.06	0.0055	0.028	0.14	0.011	0.024	0.076	0.018	0.125	0.017
Cu	0.0012	0.0023	0.0026	0.0017	0.0036	0.001	0.0068	0.0011	0.012	0.0038
Fe	0.056	0.048	0.059	0.105	0.062	0.032	0.054	0.063	0.056	0.035
Mn	0.35	0.16	0.21	0.225	0.245	0.04	0.195	0.455	0.215	0.13
Ni	0.0047	0.005	0.012	0.024	0.0078	0.0038	0.011	0.0057	0.071	0.0085
Zn	0.001	0.001	0.001	0.0025	0.001	0.001	0.0011	0.001	0.099	0.001

RESULTS

ABA testwork

ABA testwork results for the ten samples are summarised in TABLE 2. Based on the results all samples are classified as Non Acid Forming (NAF). Paste pH of the samples ranged from 8.12 to 8.96 with an average of 8.47. The total sulfur concentrations range from 0.01 wt% to 0.16 wt%. The samples are classified as NAF based on their NNP and NPR values which were calculated from the NP and AP values. Nine of the 10 samples have a greater NP than AP and are therefore classified as net neutralizing mine waste. An NNP result of <-20 and an NPR result of <1 is indicative of a potentially acid forming (PAF) sample, whereas an NNP of >20 and an NPR of >3 is representative of a NAF sample. All samples except for sample 10 report an NPR of >3.

Table 2 Sample summary

Sample ID	Total S (%)	Sulfide S (%)	Paste pH	TIC (%)	AP (kg CaCO ₃ eq/t)	NP (kg CaCO ₃ eq/t)	NPR	NNP (kg CaCO ₃ eq/t)	ARD Classification
1	0.01	0.01	8.52	0.09	0.31	1.463	4.68	1.15	NAF
2	0.06	0.06	8.96	0.24	1.88	20.30	10.83	18.43	NAF
3	0.05	0.05	8.55	0.21	1.56	12.79	8.18	11.23	NAF
4	0.16	0.16	8.22	0.23	5.00	17.29	3.46	12.29	NAF
5	0.07	0.07	8.31	0.11	2.19	22.25	10.17	20.06	NAF
6	0.03	0.03	8.32	0.36	0.94	19.03	20.29	18.09	NAF
7	0.15	0.15	8.14	0.19	4.69	17.13	3.65	12.44	NAF
8	0.01	0.01	8.67	0.06	0.31	5.46	17.48	5.15	NAF
9	0.07	0.07	8.12	0.16	2.19	19.24	8.79	17.05	NAF
10	0.03	0.03	8.86	0.21	0.94	0.00	0.00	-0.94	Uncertain

PAF – Potentially Acid Forming; NAF – Non-Acid Forming; TIC – Total Inorganic Carbon

Mineralogical assessment

Optical microscope and SEM investigation confirmed the main sulfide mineral present within the deposit as pyrite (FeS₂), which was present in two main textural associations. First, pyrite was frequently present in trace (<1%) proportions within the main quartz-mica-schist (QMS) fabric; usually fine-grained and aligned along the foliation direction. Second, coarse to medium-grained euhedral to subhedral pyrite within cross-cutting veins. This pyrite was closely associated with quartz and ankerite (the main carbonate mineral observed). In addition to pyrite there was subordinate chalcopyrite (FeCuS₂), aikinite (CuPbBiS₃), enargite (Cu₃AsS₄), lautite (CuAsS), and galena (PbS). Mg-poor ankerite was the primary carbonate present in the samples, with an approximate stoichiometry of 0.5Ca0.2Mg0.3(Fe,Mn)CO₃.

NAG testwork

The NAG pHs recorded during the various stages of the NAG tests are reported in TABLE 3 and FIGURE 1. In general, a NAG pH less than 4.5 and a NAG value greater than 5 kg H₂SO₄ equivalents per tonne are indicative of a PAF material. All NAG pH values recorded in the various stages of the testwork are neutral to alkaline (7.0 to 10.67). The table shows that following the NAG test but prior to the pre-heating stage, the measured pH for all solutions is in the range of 7.0 to 7.5 with little deviation. Comparison of the pre-boiling NAG pH with measured paste pH shows that pre-boiling NAG pH is consistently in the region of 1 to 1.5 pH units lower than paste pH.

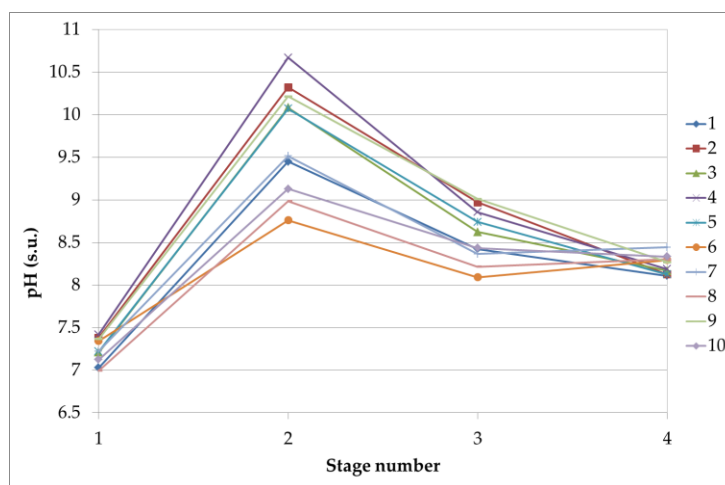


Figure 1 pH variability in NAG experiments

Following the initial pH measurement the NAG solution was heated as per the protocol for a period of approximately 2 hours. Following the heating step, the solution was allowed to cool back to room temperature and was then filled to 250mL with deionised water. Solution pH was then measured a second time (stage 2). The results show that the NAG pH of the solutions greatly increases following the boiling of the solution, suggesting that the solutions are out of equilibration with CO₂ when samples are allowed a standard cooling time. The average pH of the ten solutions increased from 7.23 at stage 1 to 9.72 at stage 2. The average pH dropped from 9.72 at stage 2 to 8.57 at stage 3 following 24 hours of further oxidation and equilibration. After a week, the pH of seven of the ten samples showed further decline, with the average pH decreasing from 8.57 at stage 3 to 8.24 at stage 4.

PHREEQC output results

The PHREEQC output results are in TABLE 3 and are compared with the results of the laboratory testwork in FIGURE 2. The results of thermodynamic equilibrium modelling using PHREEQC exhibit the same pH variability as seen in the laboratory based NAG testwork. The solutions were reacted at a temperature of 100°C; this resulted in the degassing of CO₂ to equilibrium with the atmosphere. The subsequent cooling of the solution back to room temperature results in the system being at disequilibrium with respect to atmospheric CO₂. Average pH increased from 7.23 in the input solution to 9.93 following the boiling and subsequent cooling of the solutions. Comparison between the laboratory data and the thermodynamic equilibrium simulation show good agreement with respect to average pH characteristics with average values in both the disequilibrium case and fully equilibrated cases showing agreement within 0.5 pH units.

Table 3 Comparison between NAG test pH and simulated pH

Sample ID	Pre heating pH		NAG pH (2 hours)		NAG pH (24 hours)		NAG pH (168 hours)	
	Test - Stage 1	Sim 1	Test - Stage 2	Sim 3	Test - Stage 3	Sim	Test - Stage 4	Sim 4
1	7.03	7.03	9.45	9.95	8.42		8.11	8.17
2	7.38	7.38	10.32	9.98	8.97		8.13	8.23
3	7.21	7.21	10.08	9.96	8.62		8.17	8.22
4	7.42	7.42	10.67	9.95	8.86		8.19	8.18
5	7.22	7.22	10.07	9.95	8.74		8.13	8.16
6	7.35	7.35	8.76	9.97	8.09		8.3	8.19
7	7.22	7.22	9.52	9.94	8.37		8.44	8.17
8	7	7	8.99	9.87	8.22		8.3	7.95
9	7.37	7.37	10.21	9.92	9.01		8.27	8.07
10	7.12	7.12	9.13	9.8	8.44		8.33	7.8
Average	7.23	7.23	9.72	9.93	8.57	-	8.24	8.11

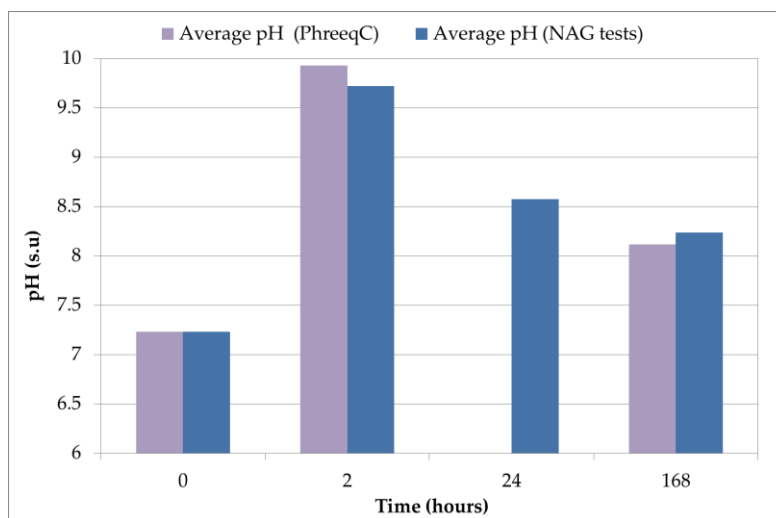
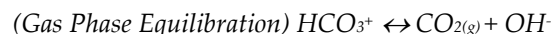
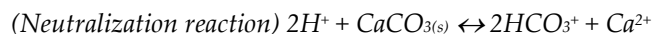
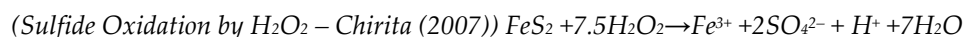


Figure 2 Average of PHREEQC results for all solutions compared to NAG test results

DISCUSSION

The generally accepted NAG test method outlined in Stewart *et al.* (2006) instructs that the NAG solution is boiled to facilitate the breakdown of residual H₂O₂ and also the dissolution of carbonate neutralization potential (NP) buffering. The results of this work have shown that the boiling step in the NAG test methodology can lead to the over estimation of basic pH conditions when the NAG solutions are allowed the standard cooling time. It is postulated that this is the result of the solutions being out of equilibration with respect to CO₂ gas and carbonate minerals. Therefore, the use of such data may lead to a misleading prediction of solution composition, if NAG solution analysis for dissolved metals is to be undertaken.

In order to understand what is occurring in the NAG solution as a result of boiling, it is necessary to understand the carbonate buffer system in detail. Unlike a number of other naturally occurring buffer systems, such as borate, the carbonate buffer system has a gaseous component, and therefore the pH behaviour of the buffer is dependent on interaction between the solution phase and the gaseous phases (Stumm & Morgan, 1996). Furthermore, the nature of the NAG test, in which acidity rapidly dissolves carbonate minerals, means conditions are suitable for gas phase disequilibrium to occur in accordance with the following stoichiometry:



Owing to the sensitivity of gas solubility to solution temperature (in accordance with Henry's Law), any changes in solution temperature may have a marked effect on solution pH. Furthermore, the relatively shallow concentration gradients involved in CO_{2(g)} equilibrium with a solution phase due largely to the relatively low atmospheric concentration, means the rates of CO₂ gas transfer between gaseous and solution phases (as described by Fick's law) is slow, requiring hours or days to attain.

The use of the typical NAG test method involving a heating step on NAF samples with elevated carbonate content can therefore produce excessively basic NAG pH as an artefact of test conditions. The basicity of the NAG solution pH reported in this study immediately following cooling after the boiling step may lead to the following potential issues:

- Increased mobility of certain elements, especially the oxyanions potentially including arsenic, selenium antimony, and chromium
- Decreased mobility of certain elements, especially metals whose minimum hydroxide solubility is above pH 9 such as zinc, nickel and cadmium.
- Potential misinterpretation of pH conditions from mine-waste rock drainage leading to incorrect classification of acid generation characteristics.

CONCLUSION

NAG testing has emerged as a useful tool in the assessment of ARDML at mine sites. However, the results of this study and previous work undertaken utilising NAG tests have identified that there is still much to understand about the nature of these tests, and the chemical mechanisms that control the stability and behaviour of the H₂O₂ solution. This study has focused on methodological variations in NAG test pH conditions, resulting from carbonate disequilibrium, resulting from the

particular prescribed methodology. The results suggest that the pH variations seen in the test may be attributed to CO₂ disequilibrium during the heating stage of the NAG test. Therefore NAG pH according to the standard test methodology (for certain net alkaline solutions) cannot be fully relied on as an indication of mine water pH, due largely to the gas phase disequilibrium created.

In order to address this, it is recommended that the pH of a NAG solution be recorded pre- and post-boiling in order to identify the discrepancy in pH disequilibrium. In addition, if the intention is to analyse the NAG solution to give an indication of the potential for mobilization of problematic elements, it is advised that the solution is collected prior to solution heating stage to avoid under- and/or over-reporting metal mobility.

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