PREDICTING ACID GENERATION – DO YOU KNOW WHICH TEST TO USE?
(A Review of Commonly Used Test Methods and Discussion of Test Limitations and Modifications Using Case Studies

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ABSTRACT

Static acid base accounting tests are simple chemical tests which are often relied upon to make decisions concerning the acid generation potential of various materials without supporting information. Several studies completed by experts in the field of acid rock drainage have shown that static tests can sometimes result in incorrect diagnosis of the field conditions. It is for this reason that there are several different static test methods currently available and researchers are still developing others. No one single test is adaptable enough to correctly predict the results for geologic materials in different environmental settings.

Existing static predictive methods including the Standard, and Modified EPA acid base accounting methods, the B.C. Research Initial Test and the Net Acid Generation test, are reviewed and compared in this paper. Kinetic humidity cell and column leach test methods are also discussed with respect to test methodology and test results. Case studies are used to demonstrate the advantages and shortcomings of the various static and kinetic test methods for (1) previously oxidized materials; (2) materials containing non-sulphur based acid generators; and (3) materials with complex mineralogy. Test design limitations and specific examples of modifications are also discussed.

1 INTRODUCTION

Acid generation from the oxidation of sulphides is one of the most significant and potentially most costly environmental concerns facing the mining industry today. Costs for the treatment of acidic mine effluents can threaten the viability of a mining operation. It is, therefore, important to have accurate and reliable methods for the prediction of Acid Rock Drainage (ARD) or Acid Mine Drainage (AMD). Several methods are currently available to predict the potential for acid generation. The selection of the most appropriate method depends on the geochemistry and mineralogy of the deposit, the environmental and regulatory setting of the site, as well as the proposed management and control methods being used or planned.

There are several tools available for use in ARD prediction, including laboratory testing, theoretical modelling and field-testing. Also, site-specific factors must be considered. This paper will discuss several of the laboratory test methods available with respect to their advantages and disadvantages. Both static and kinetic tests will be discussed and three case studies will be presented to illustrate how test modifications may be necessary to accurately predict ARD.

2 STATIC LABORATORY TEST METHODS

Static laboratory tests are popular because they are inexpensive and results can be obtained quickly. The lower cost can permit the analysis of a larger number of samples, thereby providing for the more thorough sampling necessary to characterize a site.

The majority of the static tests available compare the neutralization potential (NP) of the sample with the acid production potential (AP) to obtain the net potential (Net NP) of the sample. The AP is generally calculated using the concentration of total sulphur or sulphide sulphur in the sample. The NP can be determined using a number of different methods. Following is a summary of the most common static test methods used to determine the balance between the acid producing and acid consuming components of samples.

2.1 Standard EPA ABA Method

The most widely used static test method is the EPA ABA (Sobek Acid-Base Accounting) Method, which was developed by the Environmental Protection Agency in the United States (Sobek et al, 1978). The AP is calculated in this method using the total sulphur concentration. Since it is generally only the sulphides that will generate acid in the field, the use of the total sulphur concentration in the calculation may cause an overestimation of the AP of the sample. The NP is determined by digesting 2.0 g of sample in excess hydrochloric acid at near boiling temperature. The acid consumed during the digestion is determined by titration with NaOH to a pH of 7.0. This procedure may cause the overestimation of the NP of the sample because the aggressive digestion of the sample can release silicates and other buffering minerals that would not normally consume acid in the natural environment (White et al, 1997). Despite the problems with this test, it has been widely used for a long time, therefore, there is more data available on this test than any other static test. If the digestion is managed and maintained at the higher end of the pH range dictated by the test (i.e. around pH 2) the NP results have been shown to be comparable to those determined by the Modified EPA ABA test method described below (Price, 1999). Exercising control and regular monitoring during the digestion step can, therefore, increase the reliability and reproducibility of the standard ABA test results.

2.2 Modified EPA ABA

A Modified Acid-Base Accounting test was developed by R. Lawrence (Coastech Research Inc., 1989, and described by Lawrence, 1990) to reduce the potential of the Sobek method to overestimate NP. In addition, the AP is calculated in this method using sulphide sulphur concentrations so that non-acid producing forms of sulphur are not included in the sulphuric acid generation calculations. The NP is determined by digesting the sample in excess hydrochloric acid at ambient temperature. As with the Sobek method, the acid consumed during the digestion is determined by titration with NaOH. However, the end point of the titration for the modified method is pH 8.3 while the end point for the original method was pH 7.0. This endpoint is based on the pH endpoint at which carbonic acid is neutralized to bicarbonate ion and results in a lower NP value than what would be determined using the standard method.

2.3 B.C. Research Initial Test

The B.C. Research Initial Test (BCRIT) (Duncan and Walden, 1975 and Bruynesteyn and Duncan, 1979) has been widely used for the determination of Net NP. The AP in this test is calculated using the total sulphur concentration. The NP is determined by titration a pulp of the sample with sulphuric acid to an end point of pH 3.5. The pH 3.5 endpoint was selected because it is argued that, at this point, active biochemical oxidation is not favoured (Coastech, 1990). This method takes at least 24 hours to complete, and can take several days, which makes it more expensive than the other tests discussed, however, the NP titration method does permit the neutralizing minerals present to react at their own rate, thereby permitting a more natural neutralization reaction. As with the standard EPA ABA test, however, the use of the total sulphur concentration in the calculation may cause an overestimation of the AP of the sample.

2.4 Net Acid Production Test

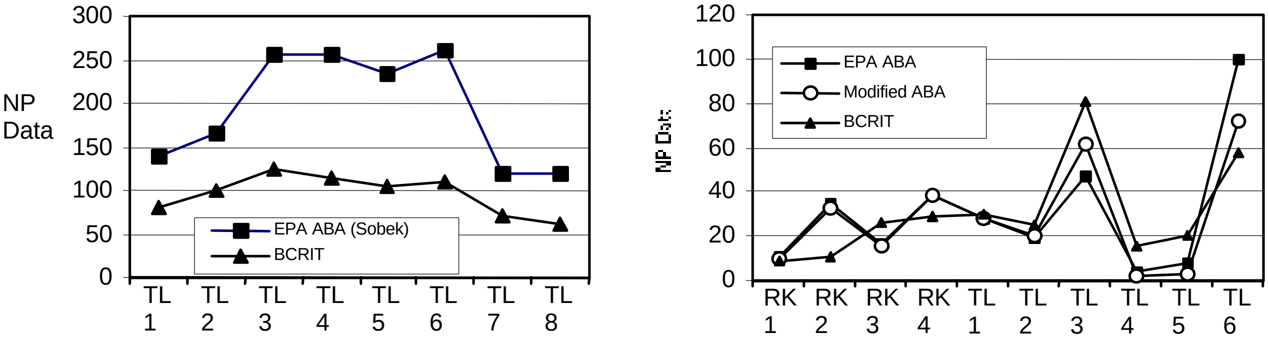
The Net Acid Production (NAP) test (Lapakko and Lawrence, 1993) and the Net Acid Generation (NAG) test (Miller et al, 1994) both expose a sample to excess hydrogen peroxide to induce complete oxidation of the sulphide minerals present. The resultant acidity produced reacts with the available NP. In the NAP test, the final solution pH and results from the titration of the solution to pH 7.0 are used to provide an indication of the NAP capacity of the material. This test removes the uncertainty related to the availability of sulphur species to generate acidity and provides an end result indication of net acid Production capability. It does not, however, provide a measure of the NP available.

Other static tests used, but not covered here, include the Carbonate NP determination (Lawrence and Day, 1997), and the Lapakko NP (pH 6.0) method (Lapakko, 1994).

3 COMPARISON OF RESULTS FROM DIFFERENT STATIC TESTS

The results of comparative tests on tailings samples using different static test methods and different sample types are presented in Figures 1 (Calow et al, 1995) and 2 (White et al, 1997). The test results show that the NP is affected by the test methodology. This is partly due to the differences in grain size used in each of the test methods. The standard method specifies a minus 60-mesh particle size. The modified method specifies a much finer particle size of 80 % minus-200 mesh. And the BCRIT uses samples pulverized to less than 400 mesh. It has been shown that the finer the particle size the greater the release of NP due to greater mineral liberalization (Lapakko and Antonson, 1991*).*

FIGURE 1. Comparison of Neutralization Potential (expressed as tonnes $CaCO_3$ equivalent/1000 tonnes material) calculated for EPA ABA and BCRIT data for samples of tailings from a Canadian gold mine.



Sample Identification Number: RK = Waste Rock, TL= Tailings

The differences in NP values are also partly due to the differences in titration and end point pH's with higher NP's being reported where lower end point pHs are used. Since the standard Sobek method does not define the pH end point range, the lower digestion pH's possible can result in typically unavailable silicate mineral neutralization capacities being reported in the NP values determined. The results also show that the differences between the tests are not consistent. This variability in NP determinations coupled with the potential of the overestimation of AP illustrates the complexities involved in interpreting static test results. For this reason, the use of other test methods, such as the NAP and kinetic testing, is often recommended.

4 KINETIC LABORATORY METHODS

Bench and pilot scale kinetic tests are often used to determine the weathering and leaching characteristics of a sample over time. In addition to verifying the results obtained from static tests, kinetic tests are also used to determine the rates of acid generation and neutralization depletion and to predict the water quality from the stockpiled materials. Misinterpretation and oversimplification of kinetic test results, however, has resulted in erroneous predictions of acid generation.

4.1 Humidity Cell Tests

The Humidity Cell Test (ASTM Method#D5744-96) is the most commonly used bench scale kinetic test for determining the potential for acidic leachate generation. According to the standard method, a minimum of 1 kg of sample is placed in the test cell and dry and humidified air is alternately pumped through the cell. The contents of the cell are periodically washed with de-ionized water and the wash water is then filtered and analyzed. The type of analyses conducted varies with the requirements of the test and the geochemistry of the material tested. Humidity cell tests may be modified to better answer specific questions. For example, altering the environmental conditions may accelerate the weathering of the material in the cell. An example of an approach to this could involve inoculating the cell with sulphur oxidizing bacteria. The strain(s) of bacteria used would be dependent upon the type of bacteria present at the site location and efforts should be made to collect natural bacteria populations from the site. The humidity cell design can also be modified to better simulate on-site weathering conditions, or to model a control or closure system design. (Elliott et. al., 1997, 1996).

4.2 Column Leach Tests

The standard tailings humidity cell set-up does not allow for the precipitation of secondary minerals and, therefore, the leachates generated by these cells should not be considered representative of ARD leachate quality. Agitated leachate tests, used more commonly in the past, also do not recognize the importance and effect of secondary precipitation on the quality of ARD. Both standard tests such as the State of Nevada Meteoric Water Mobility Procedure (better suited to waste rock) and specifically designed column leachate tests, can be used to determine the quality of leaching waters from various mine waste materials. The use, again, of representative samples is important to these tests.

Kinetic tests generally require a minimum of 20 weeks to complete and can produce useful information for up to 2 years. The duration of the test is a function of the mineralogy of the sample being tested and the weathering conditions to which it is exposed.

5 COMPARISON OF KINETIC TESTS TO FIELD DATA

Lawrence et al. (1989) assessed and compared the results of humidity cell tests with field data. Selected data from their study is presented in Table 1. The study identified two main concerns: 1) that the small test sample weight used may not be representative of the natural sample variability in the field situation and 2) laboratory tests are not always allowed to continue to full sulphide depletion.

TABLE 1. Comparison of Humidity Cell Test Results With Field Data (Lawrence et. al, 1989)

Waste Rock	Total Sulphur %	Sulphide Sulphur %	Minus 16 Mesh %	Was ARD Generated?	
				Humidity Cell	Field
Curragh	8.66	8.19	65	No	Yes
Equity Silver	19.1	19.73	51	Very low	Yes
Heath Steele	6.12	5.89	55	Very minor	Yes
INCO	0.69	0.20	72	Very minor	Yes

A more recent study of humidity cell data has shown the importance of running duplicate cells and of preparing true duplicate samples for use in these tests. Poor reproducibility between some duplicate humidity cell tests is believed to be due to variations in the grain size of the duplicate samples (Mills, C. (undated) and Downing & Mills, undated)*)*. The results of the test indicated that significantly different generation rates were predicted for the two “duplicate” samples of material tested.

6 IMPORTANCE OF MINERALOGY IN ARD PREDICTIVE TESTING

Brief references to the importance of mineralogical testing and examinations for confirming the results of ABA tests have been made in the preceding sections. Various factors may interfere with the determinations of total sulphur and sulphide sulphur concentrations and the NP of samples. As the sulphur determinations are used to calculate the AP and the carbonate content effectively determines the NP of samples, any errors in these determinations will negatively influence the results of the ABA tests. Mineralogical examinations may also be used to determine the type, nature and availability of acid generating and neutralizing minerals, with each of these factors having a strong influence on the Net NP of a sample. A companion paper by S. Downing in the Environmental Advances Session 1 of the CIM Calgary 99 conference discusses the importance of mineralogy in detail.

7 CASE STUDIES

7.1 Case Study 1: Acid Generation Prediction for Previously Oxidized Materials

The determination of acid generation potential in previously oxidized materials can be difficult using the standard test methods because of the presence of stored acidity. Stored oxidation products, which may result in the release of ARD from direct leaching of the material, is not directly related to the strict definition of the acid generation potential of the material.

Because of the dependence of most static acid base accounting methods on fizz ratings and their approach to the NP determination through the addition of a standard volume and strength of acid based on fizz test ratings, stored acidity often results in negative, or “false” NP values. These cannot be accurately interpreted in terms of available NP. For this reason, where stored acidity is potentially of concern, Lakefield Research has developed the Wash Step Procedure for use in the characterization of the two forms of ARD related to these materials; the stored ARD product and the ARD potential remaining. The Wash Step Procedure is completed prior to the ABA test to minimize the effects of stored acidity on the static test results.

The Wash Step Procedure is recommended for materials with a paste pH value of less than 5.5. The washing of the sample is completed in a series of steps using deionized water. The number of steps necessary is determined by conducting analysis of the wash solution for the presence of sulphate ions. The purpose of the wash is to remove stored acidity and other oxidation products from the material. When no further sulphate ion concentrations are noted within the wash solution, the Wash Step Procedure is considered complete, i.e. all readily soluble sulphate is removed. Washing generally takes three to four steps for completion. The wash solution(s) collected from each step are combined, the total volume is recorded and the composite is analyzed for pH, acidity, sulphate and metals of interest. The washed solids are then submitted for static ABA testing.

The case study presented below involved the testing of eleven waste rock samples from Chile, S.A. submitted for modified EPA ABA testing. The modified EPA ABA tests were conducted on both the as-received solids and on subsamples that had been subjected to the Wash Step Procedure. The results of S assays completed on the wash solution, and on the solids before and after washing, indicated that the calculated total S, as determined by the summation of the total S in the wash water and the total S in the washed solids, was reasonably comparable to the measured total S in the unwashed solids. Therefore, performance of the wash step procedure did not appear to cause significant S losses from the material balance.

Table 2 shows the modified EPA ABA tests results on the washed and unwashed solids. The unwashed solids of samples WR-2, WR-3, WR-8, WR-9, and WR-10 produced negative NP values, suggesting no neutralization potential was present in the samples. However, the washed solids of these samples showed NP values ranging from 7.4 to 10.6 t $CaCO_3$/1000 t materials. Using these NP values it is possible to determine the Net AP capability of the material by calculating the AP related to the stored acidity and then simply adding the two separate sources of AP and subtracting the NP.

TABLE 2. Acid Base Accounting Test Results for Unwashed and Washed Solids

Sa	**Unwashed Solids**					**Washed Solids**				
	$S^{=}$ %	**NP**	**AP**	**Net NP**	**NP/AP**	**$S^{=}$ %**	**NP**	**AP**	**Net NP**	**NP/AP**
WR-1	< 0.01	3.93	0.31	3.62	12.7	0.02	10.2	0.63	9.57	16.2
WR-2	0.02	-2.44	0.63	-3.07	-3.87	0.02	8.4	0.63	7.77	13.3
WR-3	0.02	-1.46	0.63	-2.09	-2.32	0.01	10.0	0.31	9.69	32.3
WR-4	0.02	14.5	0.63	13.9	23.0	0.02	11.2	0.63	10.6	17.8
WR-5	0.99	17.4	30.9	-13.5	0.56	0.56	8.7	17.5	-8.80	0.50
WR-6	0.02	10.3	0.63	9.67	16.3	0.02	10.2	0.63	9.57	16.2
WR-7	< 0.01	13.7	0.31	13.4	44.2	<0.01	9.4	0.31	9.09	30.3
WR-8	3.13	-0.97	97.8	-98.8	-0.01	1.69	7.4	52.8	-45.4	0.14
WR-9	0.45	-28.7	14.1	-42.8	-2.04	0.47	9.2	14.7	-5.5	0.63
WR-10	0.19	-7.59	5.94	-13.5	-1.28	0.21	10.6	6.56	4.0	1.61
WR-11	0.59	10.1	18.4	-8.3	0.55	0.51	8.6	15.9	-7.3	0.54

NP, AP and Net NP are expressed in tonnes $CaCO_3$ equivalent/1000 tonnes material

The final result of the static test program showed how stored oxidation products can affect the determination of acid generation potential using standard test methods. The presence of stored acidity in the samples, however, indicated that the materials may leach acidity and other stored products, such as metals. If the purpose of the static test program were to identify the potential environmental impacts related to these materials, the test program would have been considered a failure due to the selection of the wrong test design. A column leachate test would be better suited to that purpose.

7.2 Case Study 2: Materials Containing Non-Sulphide Mineral Based Acid Generators

In the majority of the static acid base accounting tests, the AP is calculated according to either total sulphur or sulphide sulphur content present in the materials, assuming (1) total conversion of sulphur to sulphate, and (2) production of 4 moles of H^+ per mole of pyrite oxidized. Acidity, however, can be generated by other means and, therefore, the prediction of acid generation potential is best determined from the use of a combination of several different test methods.

A development project required that the sediments from a delta area be dredged and stockpiled at surface. Initial testing had indicated that the sediment samples contained relatively low sulphur contents and were acid generating. Because the ferric-ferrous iron transformation can also generate significant acidity, several composite samples were analyzed for Fe^{2+} in addition to analyses for total S, and sulphide S. The acid consuming ability of the samples was determined using B.C. Research Initial test. NAP tests were also completed to assess the net potential for acid generation. The test results are presented in Table 3.

The results in Table 3 indicate that the sediment samples are acid generating, however, the total acid production potential by Fe^{2+} and S only accounted for 13-34% of the net acid produced by hydrogen peroxide oxidation. Other components in the samples that may contribute significantly to the acid production include organic matter, aluminum and other acid producing metals. As these sediment samples were high in organic matter, the generation of organic acids may be responsible for the balance of the acidity generated. Unfortunately, the scope of the test program did not allow for the testing of this hypothesis.

TABLE 3. Acid Consuming Ability and Acid Production Potential of the Sediments

Sample	Acid Consuming Ability	Acid generation by Fe^{2+} and S						NAP
		AP by Fe^{2+}		AP by S		Total AP by Fe^{2+} and S	% of NAP	
		Fe^{2+} %	AP	S	AP			
CS-1	4.6	1.05	18.8	1.21	37.8	56.6	18.4	308
CS-2	10	0.72	12.9	1.38	43.1	56.0	25.9	216
CS-3	10	1.4	25	1.58	49.4	74.4	33.7	221
CS-4	10	0.55	9.8	0.47	14.7	24.5	13.5	182
CS-5	10	0.70	12.5	1.01	31.6	44.1	19.4	227
CS-6	4.3	0.64	11.4	0.09	2.8	14.2	25.0	56.9

Note:
1. The units for acid consuming ability, acid potential and NAP are kg $CaCO_3$/tonne material
2. AP by Fe^{2+} is calculated assuming 1 mole of Fe^{2+} produces 2 moles of H^+ that will be neutralized by 1 mole of $CaCO_3$
3. AP by S = %S x 31.25

7.3 Case Study 3: Acid Base Accounting for Materials with Complex Mineralogy

A heap leach residue sample was submitted for modified ABA testing. The test results showed that sample contained 4.44% total S and 2.9% sulphide S, with a NP value of 6.6 t $CaCO_3$/1000 t material. While a heap leach residue would not be expected to contain significant sulphide mineralization, repeated static testing confirmed the initial test results. These results indicated that the sample had a potential for acid production and that the sample was partially oxidized. To determine whether the residue would generate further acidity, a standard humidity cell test was conducted. The humidity cell test indicated that after 40 weeks of exposure to oxidizing conditions, the sample exhibited a very low rate of sulphate release and still exhibited the capability to buffer any acidity produced with a leachate pH around 8.5. Because of the observed slow rate of reaction, mineralogical examinations and the NAP test were recommended to provide additional information useful to determining whether or not the sample would eventually turn acid.

The mineralogical examination included X-ray diffraction and polished section and polished thin section. The mineralogical examinations identified the presence of alunite (15-20%) and jarosite (2-4%) which correlated well with previously reported aluminum (4.4% Al) and total S assays (4.44%). Only trace sulphide concentrations, estimated around 0.5% by volume, consisting predominantly of liberated pyrite with rare covelite and chalcopyrite, were identified in the heap leach residue. The observed sulphide concentration did not correlate well with the sulphide S assay (2.9% $S^{=}$). The presence of alunite, a sulphate mineral with low solubility that cannot be removed by the sulphate wash step in sulphide determinations, had interfered with the analytical determination of the sulphide concentrations. While less sulphide was present than initially believed, the mineralogical identification also confirmed that only rare carbonate particles (<0.2%) were observed in the sample. The sulphide:carbonate ratio for this sample (>2), as well as the availability / liberation of the sulphide particles still indicated, however, that the heap leach residue may be a mild acid producer.

The NAP test on the heap leach residue showed that the final pH at the end of hydrogen peroxide oxidation was recorded at 6.12 and the NAP value was calculated to be 0.18 kg $CaCO_3$/t material. The test results were within the range of values considered representative of a non-acid generating material. At the time of completion of the NAP test, the humidity cell test had run for a total of 53 weeks, and was still not generating sulphate concentrations indicative of sulphide oxidation and was maintaining above neutral pH levels in the leachates. By considering the combination of results from the humidity cell, NAP test and mineralogical examinations, the heap leach residue sample was determined to be unlikely to produce acid.

8 DISCUSSION

Small-scale tests for determining acid generation from tailings and waste rock are valuable tools for defining ore bodies and for initial scoping of storage and remediation plans. To ensure that valid data are obtained, it is important to choose the correct tests and recognize the limitation of each test method. It is also important to correlate the test results with chemical and mineralogical characterization of each sample. The data collected during bench scale testing may be influenced by the sampling error introduced with the small sample volumes used. The information obtained from static tests is also limited because it provides no data on the rate and extent of potential sulphide oxidation and neutralization that would occur in the field. The use of more than one single static or kinetic ARD predictive tool can provide confirmation of test results and the certainty necessary when making planning and operational decisions to deal with ARD issues.

The case studies in this paper illustrate the importance of the following factors in selecting ARD predictive tools:

- site geology and the extent of weathering
- site waste management practices
- site climate
- mineralogy
    - presence of interfering minerals
    - availability of AP & NP minerals
- the use of representative samples of sufficient number.

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