DETERMINATION OF NEUTRALIZATION POTENTIAL IN THE PREDICTION OF ACID ROCK DRAINAGE

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ABSTRACT

The prediction of acid rock drainage involves a large number of tests, analyses, assessments and interpretations to provide an effective contribution to waste management planning and environmental control. One of the most critical parameters measured in the initial stages of a prediction program is the neutralization potential, NP, which provides a measure of the ability of waste materials to consume the acidity produced when sulfide minerals oxidize. A test program has been carried out on 120 samples of waste rock and tailings from several mines in different parts of the world and has shown that NP values for a specific sample can vary significantly depending on the particular test procedure used and on variations within a specific procedure. Results show that the most widely used procedure, the acid base accounting (ABA) procedure of Sobek, overestimates NP for the majority of samples. Overestimation is often high enough to affect the classification of a sample as either non-acid producing or acid-producing when applying classification criteria such as the NP to AP (acid potential) ratio as used by many regulatory agencies. NP values obtained by the Sobek procedure are compared with those from Modified ABA and Carbonate NP procedures. Test procedures for NP determination and interpretation are recommended and implications for the long range prediction of acid rock drainage are discussed.

Keywords: acid rock drainage, waste characterization, prediction, neutralization potential, permitting

INTRODUCTION

Acid rock drainage (ARD) is the single most important environmental concern in the mining industry. Feasby and Jones (1994) indicate that the financial liability for the remediation of waste rock and tailings accumulations in Canada is in excess of $5 billion. Waste rock and tailings are the most important sources of ARD, although other mine components such as the walls in open pits, underground workings, ore stockpiles, and concentrate storage and loadout areas can contribute significant amounts. Proponents of new mines and operators of existing mines are required to provide evidence that waste materials can be stored in a manner that will prevent or mitigate the generation of ARD and the migration of heavy-metal- contaminated runoff and seepage into downstream watercourses both during operation and long after mine closure. Uncontrolled ARD emissions can result in significant ecological disruption in sensitive and productive receiving waters. To provide confident waste management plans that will allow permitting to proceed and acceptance of closure measures requires that proponents and operators characterize the wastes resulting from current or proposed mining activities in order to predict future performance of the materials when disposed of under environmental conditions.

Mining waste materials can be the source of acidic drainage, with associated heavy-metal contamination, due to natural oxidation reactions taking place when the waste is exposed to air and water, with sulfide oxidizing bacteria contributing a significant role in the process (Kleinmann et al., 1981). Acidic drainage can be neutralized and heavy metals attenuated, however, due to reaction of the drainage with alkaline components of the wastes. Almost without exception, wastes are initially characterized to determine whether they have the potential to produce acidic drainage using tests known as static prediction tests. Such tests attempt to determine the balance between the acid potential (AP) of the material, derived by quantifying the acid-producing components of the material, and the neutralization potential (NP), determined by measuring the quantities of acid-consuming components. Although there are several test methods available (Ferguson and Erikson, 1987; Lawrence and Marchant, 1990), the most widely used procedure is still the Acid Base Accounting Test of Sobek et al. (1978).

Acid base accounting and other static tests only provide an initial assessment of the potential of mining wastes to produce ARD. Subsequent assessments, particularly kinetic testing, should be carried out to provide a more comprehensive characterization to predict, for example, acid generation rates, neutralization rates and water quality. In practice, however, kinetic testing is often inconclusive and considerable weight is placed on the results of the static tests. This is evident by the fact that most jurisdictions responsible for the issuing of permits to develop and operate mines use the results of static testing as criteria in the classification of wastes for planning purposes. In British Columbia, for example, it is proposed that the NP to AP ratio be used to classify wastes either as safe for disposal, without specific control requirements, or as requiring controlled disposal, or as uncertain in their classification (Price and Errington, 1995). In B.C., the value of the NP to AP ratio (sometimes written as NPR) is not a fixed value but is established following careful review of both static testing, kinetic testing and other evaluations.

In a typical static test, the acid potential of a sample is determined by calculating the theoretical quantity of acid that could be produced if the total sulfur content of a sample is converted to sulfuric acid. In the standard acid base accounting test of Sobek et al. (1978), the neutralization potential is determined by boiling a small quantity of finely ground sample in excess hydrochloric acid and the acid consumption is measured by back-titrating the acid remaining after acid digestion with sodium hydroxide to a pH of 7.0. The volume and strength of acid added is determined by assigning a fizz rating for the sample based on the degree of reactivity observed when a few drops of strong hydrochloric acid are added to a sample. In the Sobek test, the fizz ratings are none, slight, moderate, or strong fizz.

Under environmental conditions, not all sulfur determined by analysis will actually oxidize to produce acid. Neither will all the neutralizing capability, as determined under the vigorous conditions of the Sobek test, be available for reaction. The value of NP of a sample, however, is significantly more sensitive to the test methodology used and the mineralogical characteristics of the sample than the value of AP. The value of the NP to AP ratio and the subsequent classification of the sample can change, therefore, depending on the NP value obtained. Discrepancies in results often arise due to mineralogical factors which affect the analysis. For low carbonate content materials, high neutralization potentials will be apparent in tests if very acidic digestions are utilized as part of the acid base accounting procedure. Under such conditions, some minerals, primarily silicates, will dissolve and contribute to the apparent NP value (Sherlock et al., 1995). Under environmental conditions, such high acidities are unlikely to he encountered and effective NP values will be much lower, restricted to the contribution of minerals which will dissolve or alter under conditions of lower acidities (higher pH). Although silicate alteration has been shown to contribute to ARD neutralization and heavy metal attenuation under field conditions (Lapakko, 1988); Alpers and Nordstrom, 1990; Blowes et al., 1992; Sherlock et al, 1995), reactions are relatively slow and their short and long term contribution to the behaviour of wastes needs further understanding.

Consideration of the test conditions used in ARD prediction procedures is essential for the interpretation of the results and for more confident ARD prediction and waste management planning. In a recently-completed study (Lawrence and Wang, 1996), test methodologies and factors affecting NP determination were evaluated. This paper provides a summary of some of the key results of the study, including a detailed examination of the standard Sobek acid base accounting procedure, variations in NP due to variations in the procedure, and its limitations. Comparison with other static test results and supplemental tests and analyses will provide the basis for recommendations for static testing.

EXPERIMENTAL OBJECTIVES AND METHODS

The objectives of this investigation were:

- To compare NP values obtained from the standard the Sobek test with values obtained using the Modified ABA procedure of Lawrence (1990) and Carbonate NP values, calculated from analyses of the inorganic carbon contents, for 120 samples of waste rock and tailings.
- To compare NP value variations in the Sobek procedure when different fizz ratings were used for each waste sample tested. This was done since the assigning of a fizz rating can be subjective. In addition, some test laboratories, for convenience do not carry out a fizz test in strict accordance with the Sobek procedure. Instead, acid additions are made according to a strong fizz rating.
- To provide recommendations on the use of static test procedures and their interpretation for acid rock drainage prediction.

Waste Rock and Tailings Samples

One hundred and twelve waste rock and tailings samples from 12 operating or proposed mines in Canada, the United States, the Philippines, Chile and Papua New Guinea, were obtained for this study. In addition, 8 certified reference standards (concentrates, ores and other metallurgical samples) obtained from CANMET were tested. One of the reference standard materials, NBM-1, has recently been produced as a reference material for static test calibration (Leaver et al., 1994).

Most of the samples were received as assay or ABA rejects and were therefore already pulverized, with a typical size range of 100% minus 60 mesh to 80% minus 200 mesh. No further sample preparation was required for these samples. Waste rock samples received in large sizes were jaw crushed, cone crushed, and pulverized as required. Tailings samples were air dried and tested at the as-received size.

Standard (Sobek) acid base accounting requires only the total sulfur analysis to be determined to calculate the Acid Potential of a sample. Several studies have shown that AP can be overestimated if significant quantities of non-sulfide sulfur species are present. Modified procedures address this shortcoming by requiring analysis of sulfur species, particularly sulfate, so that AP is calculated on the basis of sulfide sulfur. However, since the emphasis of this study was on the determination of neutralization potential, sulfur species analyses were not carried out. Total sulfur analyses were determined either by using a Leco induction furnace or by classical acid digestion/barium sulfate precipitation.

Procedures for NP Determination

Standard Sobek Method

The standard NP procedure of Sobek et al. (1978) was followed for all samples, with acid additions selected according to the actual fizz rating. Since the samples tested were already pulverized, some samples were finer than the minus 60 mesh size specified in the procedure but were typically no finer than 80% minus 200 mesh.

Use of a Different Fizz Rating in the Standard Sobek Method

For every sample, two additional Sobek-type tests were carried out, with acid additions based on different fizz ratings than the standard Sobek procedure. For example, if a particular sample was judged to have a moderate fizz rating, the additional tests were carried out with acid additions corresponding to fizz ratings on either side of the moderate rating (i.e. slight and strong). For strong fizz samples, the additional tests were performed with acid additions corresponding to the two lower levels (moderate and slight). For no-fizz samples, the additional tests were carried out with acid additions corresponding to the two higher fizz ratings (slight and moderate). In all other ways, the tests were performed in strict adherence to the Sobek method. For a few samples, insufficient material was available to allow testing at both alternative fizz ratings.

For all tests at all fizz ratings, the quantities of base added in the back-titrations were recorded at intervals of 0.5 pH units so that the back-titration curves could be plotted. For selected samples, leachates from digestions were analyzed for Al, Ca, Mg, Na, K and Fe by atomic absorption spectrophotometry.

Modified Method

The neutralization potential of every sample was determined using a method based on the Modified Acid Base Accounting Procedure of Lawrence (1990). This test was originally developed to reduce the tendency to overestimate NP values obtained by the Sobek procedure. This is achieved by performing the hydrochloric acid digestion for 24 hours at lower temperatures (25-35°C) and by controlling the amount of acid added so that the pH of the pulp after digestion is in a specific range (2.0 to 2.5). In addition, the end point of the back-titration is

8.3, being the usual endpoint for acidity titrations, corresponding to the stoichiometric equivalence point for carbonate/bicarbonate in natural waters in which carbonic acid is the most dominant weak acid. The procedure is described in detail in Appendix I.

Carbonate NP

The inorganic carbon content of every sample was determined using a Coulimetrics Model 5030 Carbonate Carbon apparatus linked to a Coulimetrics Model 5010 CO2 Coulometer. The Carbonate NP was calculated from the carbon content as the stoichiometrically equivalent quantity of CaCO3 as follows:

$$\text{Carbonate NP (kg CaCO}_3\text{/t)} = \frac{\text{mg C in sample} \times 8.34}{\text{weight of sample (g)}}$$

The method assumes that all inorganic carbon is present as carbonate minerals capable of neutralizing acid. No corrections are made to account for the iron carbonate, siderite, $FeCO_3$, (Norecol, 1991) unless it is identified as being in significant abundance.

RESULTS AND DISCUSSION

Comparison of NP Values by Different Methods

The results of NP determinations by the Sobek, Modified and Carbonate NP methods were compared by determining the linear regression coefficients for plots of Modified vs. Sobek and Carbonate NP vs. Sobek. These results are shown for samples from each individual mine in Table 1 which also shows the regression coefficients for all 120 samples as a single group. The NP results for all samples can also be compared in Figures 1, 2 and 3 which show the relationships between NP values determined by Modified and Sobek, Carbonate NP and Sobek, and Carbonate NP and Modified respectively. Linear regression equations and correlation coefficients are indicated on the plots.

The data in Figures 1 and 2 show that, with the exception of 4 low-NP samples, NP values determined by the Sobek method are significantly higher than those obtained by either the Modified or Carbonate NP method. The regression equations indicate that, when taking all samples as a single population, NP values obtained by either the Modified or Carbonate NP method are approximately 50% of those obtained by the Sobek method. Inspection of the regression coefficients for comparisons of NP values for individual mines confirms that, in all cases, NP values determined by the Sobek method are higher. For some mines, however, Sobek values are considerable higher (e.g. Mines 1 and 2) whereas for others, the difference is less than the average for the whole population (e.g. Mines 3 and 11). For some mines, r values are low, indicating a poor correlation of NP values by the different methods.

Inspection of Figure 3 suggests a close correlation between NP values obtained by the Modified or Carbonate methods, with the Modified method giving slightly higher values based on the regression equation for the whole sample population. Correlations for individual mines shown in Table 1 show that for the majority of mines, the Modified method provides higher values than the Carbonate method. It should be noted, that the regression equations are not intended to provide a model to predict an NP value by one method given the experimental value from another. The relationship between NP values by different methods can vary significantly from one mine site to another or for different lithological groups at the same mine site.

Mine	n	Modified NP *vs* Sobek NP			Carbonate NP *vs* Sobek NP		
		β	α	r	β	α	r
1	5	0.19	7.0	0.84	0.10	2.8	0.60
2	11	0.13	15.7	0.48	0.11	8.3	0.42
3	9	0.61	-6.0	0.79	0.55	-8.3	0.85
4	8	0.47	-0.3	0.98	0.53	-2.4	0.94
5	5	0.32	-1.2	0.90	0.0	0.0	n/a
6	13	0.42	5.1	0.94	0.45	2.6	0.95
7	10	0.47	-1.8	0.48	0.22	-0.4	0.41
8	5	0.53	0.74	0.99	0.47	-2.9	0.99
9	30	0.47	-3.7	0.97	0.43	-8.0	0.95
10	4	0.36	13.5	0.99	0.38	0.8	0.95
11	4	0.64	1.3	0.98	0.78	-4.3	0.97
12	6	0.58	-1.5	0.86	0.65	-16.6	0.86
All	120	0.51	-1.7	0.93	0.49	-5.0	0.89

Table 1. Comparison of the linear regressions [$y = \alpha + \beta x$] between Modified NP [y] and Sobek NP [x] and between Carbonate NP [y] and Sobek NP [x] for individual mines with n samples

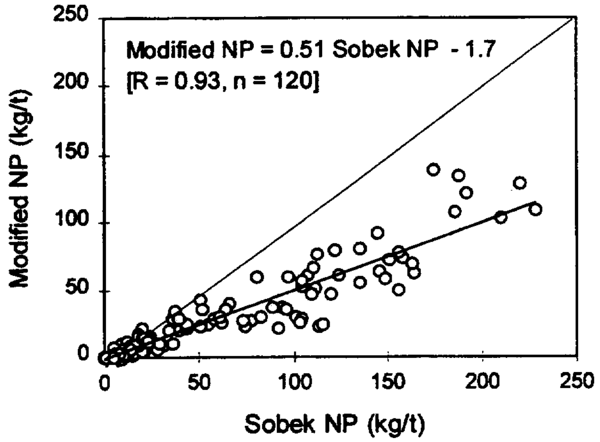

Figure 1. Comparison of Modified and Sobek NP

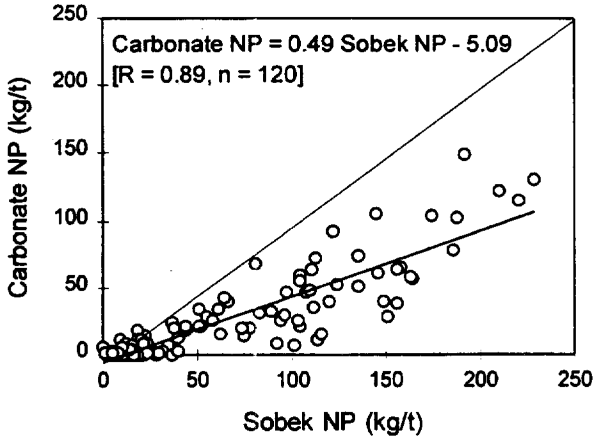

Figure 2. Comparison of Carbonate and Sobek NP

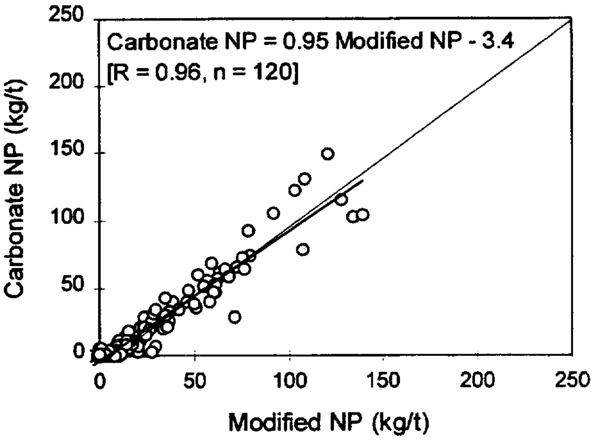

Figure 3. Comparison of Carbonate and Modified NP

Differences in the NP values obtained by the three methods can be understood by consideration of the mineralogy of the samples and the conditions of each test. As previously discussed, the mineralogical composition of a samples will control the NP value obtained in a particular test and how much acidity will actually be neutralized under the environmental conditions of waste disposal. In principal, Carbonate NP values represent the neutralizing capability of the carbonate minerals, such as calcite and dolomite. Since the Sobek method is carried out in conditions of temperature and acidity in which minerals other than the carbonates could react, the higher values obtained by the Sobek test are not surprising. Although there some notable exceptions, such extreme conditions do not usually occur under the environmental conditions found in waste dumps and tailings impoundments. Although silicate dissolution might occur under lower pH conditions, the rate and degree of dissolution and/or alteration will be significantly lower than in the Sobek test. The Sobek test clearly, therefore, overestimates NP availability under actual field conditions. The test conditions of the Modified method are such that, in addition to NP from carbonate, NP from the most reactive of the non-carbonate minerals which are likely to contribute to acid neutralization under environmental conditions is also measured.

The difference in mineral behaviour in the environment has been discussed by Goldich (1938) who ranked silicates in order of decreasing reactivity. In a similar manner, Kwong (1993) classified minerals into groups of different reactivity based on the work of Sverdrup (1990) and assigned relative reactivity values to each group. Using these classifications, Figure 2 provides a simple, non-quantitative, schematic which illustrates differences in NP values by showing some typical rock forming minerals in order of reactivity and an indication of the approximate extent of reaction achieved in each of the three test methods.

Empirical demonstration of the presence of specific mineral species in waste samples and their contribution to NP is not straightforward in most cases. Standard mineralogical analytical techniques such as X-ray diffraction cannot quantify low (<5%) concentrations of a specific mineral. if the mineral is calcite, for example, 5% is equivalent to a NP value of 50 kg/t which is not a small value. Many rock units which can prove to be significant neutralizers might have lower NP values. This problem is compounded by the fact that most samples will contain more than one acid consuming mineral, making mineral detection even more difficult when the total

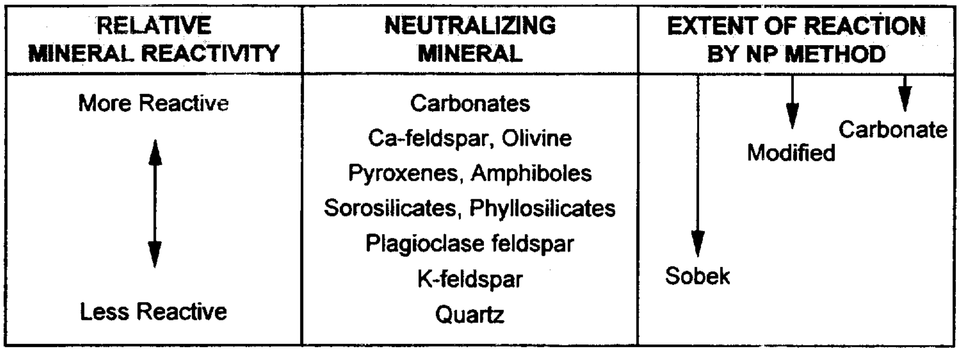

Figure 2. Relationship between mineral reactivity and method of NP determination

NP value is relatively low. Microscopic techniques, such as thin section or reflected light, together with visual examination by experienced geologists are probably of much greater value as an aid to the interpretation of NP data. Given the importance of understanding the mineralogy of the lithological groups and waste management units of a specific orebody, the input of the geologist in the initial sampling program for a waste characterization study and in the interpretation of results is strongly recommended.

A method to calculate NP based on mineralogical composition and relative reactivities has been proposed by Lawrence and Scheske (1997) and highlights the critical importance of mineralogy in the determination of NP.

Effect of Acid Addition (Fizz Rating) on NP Determination in the Sobek Test

The application of the fizz test in the Sobek procedure is very subjective. Different ratings can be assigned for the same sample by different investigators. Therefore, each of the samples tested for NP by the three methods were also tested by the Sobek method in which acid additions according to two different fizz ratings were used in addition to the standard fizz rating. For a complete discussion of all the results of this series of tests, the reader is referred to the report of Lawrence and Wang (1996). In summary, the results in Table 2 show that the degree of excess acidity, indicated by the pH, at the end of the digestion stage was greater as the fizz rating was increased from "none" to "strong". In almost all cases, this resulted in a higher NP value with a higher fizz rating. The extent of reaction, as implied in Figure 2, is therefore increased if more severe conditions are imposed on a sample, either as required by the particular test procedure, or through the assignment of a different fizz rating within one specific test procedure.

Fizz Category	Typical End pH	pH Range
No fizz	2.0 to 2.5	1.6 to 5.2
Slight fizz	1.5	0.76 to 2.1
Moderate fizz	1.0	0.77 to 1.83
Strong fizz	0.8	0.35 to 1.07

Table 2. pH values at end of digestion stage in Sobek tests conducted at different fizz ratings

Analysis of selected leachates obtained at the end of the digestion stage of the Sobek test were analyzed for Al, Ca, Na, K, Mg and Fe to provide an estimation of the degree to which neutralizing minerals had reacted in the digestion. In all cases, the increased acid additions corresponding to a change in fizz rating resulted in increased concentrations of cations in the leachates. Table 3 provides typical results for three samples showing concentrations of aluminum in the leachates at each fizz rating for each sample. In these and all other cases, the increasing amounts of the leached silicate mineral cation, Al, are directly related to an increase in the NP values. Modified and Carbonate NP values are shown for comparison and it can be seen that Sobek tests conducted under lower conditions of acidity give NP values more closely related to the Modified NP values.

Sample	NP Method	NP (kg/t)	Al in Leachate (mg/L)
5	Sobek (slight fizz)	29	13.7
	Sobek (moderate fizz)	105	67.6
	Sobek (strong fizz)	153	88.9
	Modified	28	
	Carbonate	21	
111	Sobek (slight fizz)	20	8.5
	Sobek (moderate fizz)	30	17.0
	Sobek (strong fizz)	80	48.6
	Modified	21	
	Carbonate	3	
120	Sobek (slight fizz)	53	9.6
	Sobek (moderate fizz)	151	92.0
	Sobek (strong fizz)	179	95.0
	Modified	67	
	Carbonate	39	

Table 3. Analysis of three selected digestion leachates from Sobek tests

A simply determined indicator of the degree of overestimation of NP in the Sobek test can be obtained by considering the profiles of the back-titration curves obtained following the digestion stage (Lawrence and Wang, 1997). Since a lesser or greater quantity of the minerals in a sample will be dissolved under the digestion conditions of the NP procedure depending on the amount of acid added, the constituent elements in those minerals will be in solution in lesser or greater amounts at the end of digestion. During the back-titration, these metal ions will precipitate within a characteristic pH range for the individual ions present. As precipitation occurs, the solution is temporarily buffered within that pH range and this will be evident from the shape of the titration curve. In the pH range of interest in the Sobek test (from the digestion pH up to pH 7), aluminum is the major silicate cation that will precipitate (pH range 4.0 to 5.0). If an inflection point in the curve in this pH range is obtained, the dissolution of alumino-silicate minerals would be indicated and a potential overestimation of NP suggested.

Figure 4 shows back-titration curves for the Sobek tests on sample 5 (Table 3) using different acid additions. The difference in the shape of the curves, particularly with respect to the presence and size of an inflection in the curve in the pH range 4 to 5 is evident with a change in the fizz category. At the standard fizz category for this sample (moderate), the presence of aluminum in the leachate is clearly indicated, whereas at the lower fizz category, only a very slight inflection can be noted. A higher amount of aluminum leached under strong-fizz conditions is indicated. The NP values corresponding to these curves can be seen in Table 3.

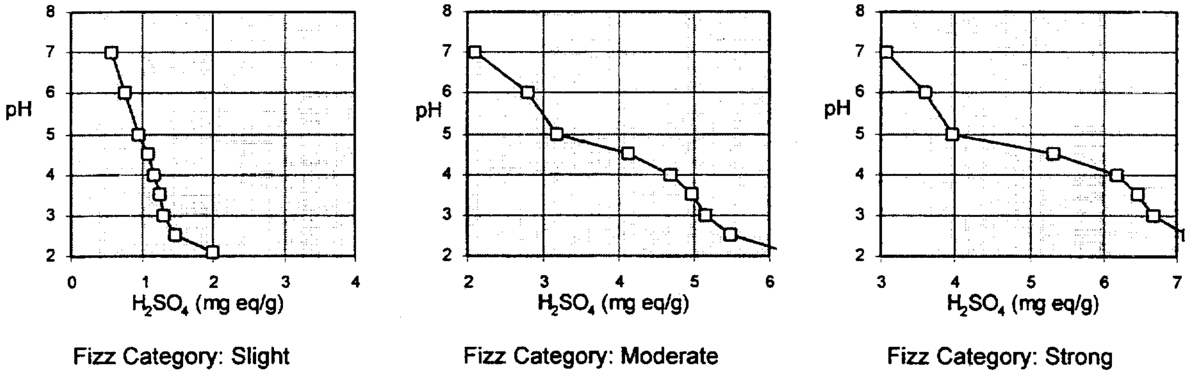

Figure 4. Back-titration curves for Sobek tests conducted on a waste rock sample with different acid additions

Experience with back-titration curves shows that the apparent increasing silicate dissolution can often be matched to the magnitude of the deviation in NP values obtained when different fizz rating are used. Of course, this is a simplistic approach since many samples exhibit complex mineralogy for which clear interpretation of the shape of the curve is not easily performed. However, the analysis can be attempted with little extra effort over the standard procedures and can provide some useful insight to the minerals contributing to the NP value of a particular sample. In addition, it should be standard practice that the fizz rating and the pH at the end of the digestion are included with the test results. The practice of eliminating the fizz test and/or only adding the highest acid quantity specified by the Sobek test, a procedure followed in some laboratories for expediency, should be avoided.

CONCLUSIONS AND IMPLICATIONS FOR ARD PREDICTION

The results of this study have shown that the value of NP obtained in static testing is greatly influenced by the test procedure used. Values obtained by the Sobek method were significantly higher than those obtained by the Modified and Carbonate NP methods for almost all 120 samples tested. Dissolution of minerals other than carbonates and the most reactive of the silicate minerals in the Sobek test is the cause of the higher NP values. The results also show that significant variations in NP values can be obtained in a Sobek test if a sample is assigned a different fizz rating (acid addition). Since the assignment of a fizz rating can be very subjective, variations in NP values for a specific sample are likely to occur frequently An indication of the potential overestimation of NP in the Sobek procedure can be gained by plotting and inspecting the shape of the back-titration curve. The presence in the curve of a significant inflection point in the pH range 4.0 to 5.0 indicates aluminum dissolution from alumino-silicate minerals and might suggest an overestimation of NP.

NP values obtained by the Modified method were higher than values obtained by the Carbonate NP method due to the contribution of the reactive silicate minerals. Either method is considered to give much more practical values of NP than the Sobek method conducted under conditions of higher temperature and acidity. Under environmental conditions such as found in waste dumps and tailings impoundments, such extreme conditions do not usually occur and although silicate dissolution might occur under lower pH conditions, the rate and degree of dissolution and/or alteration will be lower than in the Sobek test. Since the Modified method accounts for the reactive silicate minerals which have been shown by other workers to contribute to NP under field conditions, this method of NP determination is recommended for standard use in waste characterization programs. NP values obtained by the Sobek test can, however, be used if due consideration is given the possibility of overestimation. Inspection of the form of the back titration curve can be particularly useful in the interpretation of a particular result.

It can often be observed that the reporting of the results of NP determinations in many reports and mine permit submissions often do not include a full description of methods used or a complete set of the data measured. Consequently, proper interpretation of the results and the possible degree of overestimation, if it exists, cannot be assessed if variations in procedure are inadvertently used or if the test laboratory carries out undocumented variations in a particular procedure. Whatever test is used for NP determinations, therefore, it is very important that the actual conditions used are properly documented to allow a proper interpretation of the results. This should include the reporting of fizz-ratings and pH values at the end of the digestion stage.

The significance of the dependence of a NP value on the test method used, or on procedural variations within one test method, is particularly important when considering how the data are used to classify wastes for disposal and in the development of control methods to prevent acidic drainage for potentially acid generating materials. Regulatory agencies who are responsible for issuing permits for mining projects with respect to waste management typically use criteria such as the ratio of NP to AP to assist them in determining which wastes can be disposed of safely with little or no ARD control measures implemented, and which wastes will require special provision for their disposal. The wide variations in NP values that can be obtained for the same sample, as shown in this study, can significantly affect the value of the NP:AP ratio to the extent that the sample could be classified as either "safe" or '"potentially acid generating" depending on the NP value used. The implication of this can be very significant. If a sample is classified as "safe" but might not be, the environment is at risk. Conversely, if a sample is classified as "potentially acid generating" but is not, then project viability might be at risk due to the additional, and often very costly, ARD control provisions that a company will have to employ. For example, in the Lawrence and Wang (1996) report, nearly half the samples which gave a NP:AP ratio greater than 3 when using the Sobek method were found to have a ratio less than 3 if the Modified or Carbonate NP values were used. Considerable variations of the NP:AP ratio were also evident for many samples if NP values for Sobek tests conducted with different fizz ratings were used.

The impact of test method on NP and the NP:AP ratio was found to be particularly significant for the sample NBM-1, a reference standard material developed specifically for laboratories to calibrate or check on NP determination procedures (Leaver et al., 1994). Values of NP ranged from a high of 95.2 kg/t for the Sobek test conducted under high fizz conditions to a low of 15.8 kg/t for a method proposed by Lapakko (1994) (Lawrence and Wang, 1996). With an AP value of 9.3 kg/t, the corresponding range of NP:AP ratios can be calculated to be 10.2 to 1.7. The use of a definitive value of NP for reference materials is, therefore not recommended. However, the value of NP for a reference material obtained by a laboratory using a specific procedure to determine the NP values of a sample set could provide a very useful tool to assist in the interpretation of the values, even if the exact procedure used is not clearly specified.

The significance of obtaining different NP:AP ratios for the same sample by using different test procedures should be considered appropriate by regulatory agencies wishing to apply a specific criterion for classifying wastes as acid producing or non-acid producing. In many, if not most, cases, NP:AP criteria have been established on the basis of static tests carried out by the Sobek method. In these cases, some conservatism in setting the criteria might be justified given the tendency for the overestimation of NP. However, in waste characterization programs in which more practical NP values have been determined, the NP:AP criteria for classification can be set at a lower value. In some cases, mining companies using a more realistic NP method, such as the Modified Procedure, could be penalized for reporting lower NP:AP ratios than would be obtained had they used the Sobek method if they are required to conform to a more conservative classification criterion.

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APPENDIX I

The **Modified Acid Base Accounting Procedure** for neutralization potential used in this study was as follows:

1. Add a few drops of 25% HCl to 1 to 2 g of pulverized sample on a watch glass or piece of aluminum foil. Observe the degree of reaction and assign a fizz rating as "none, slight, moderate, or strong fizz".
2. Weigh approximately 2.00 g of pulverized sample into a 250 mL conical flask and add approximately 90 mL of distilled water.
3. At the beginning of the test (time = 0), add a volume of certified or standardized 1.0 N HCl according to the fizz rating as follows:

Fizz Rating	Volume of 1.0 N HCl (mL) at time = 0 h	 at time = 2 h
None	1.0	1.0
Slight	2.0	1.0
Moderate	2.0	2.0
Strong	3.0	2.0

3. Place the flask on a shaking apparatus such a reciprocating shaker, maintained at room temperature. After approximately 2 hours, add the second acid quantity as indicated in the above table.
4. After approximately 22 hours, check the pH of the pulp. If it is greater than 2.5, add a measured volume of 1.0 N HCl to bring the pH into the range 2.0 to 2.5. If the pH is less than 2.0, too much acid was added in steps 2 and 3. In this case, repeat the test adding a reduced volume of HCl.
5. After 24 hours, terminate the test and add distilled water to the flask to bring volume to approximately 125 mL. Measure and record the pH, making sure it is in the required range of 2.O to 2.5.
6. Titrate the contents of the flask to a pH of 8.3 using certified or standardized 0.5 N or 0.1 N NaOH.
7. Calculate the NP of the sample as follows:

$$\text{Modified NP (kg CaCO}_3\text{/t)} = \frac{(\text{N x vol (mL) of HCl}) - (\text{N x vol (mL) NaOH})}{\text{weight of sample (g)}} \text{ x } 50$$

The acid generating potential of the sample should be calculated on the basis of the sulfide-sulfur content ($AP = S^{=} \times 31.25$). Sulfide-sulfur is typically determined as the difference between total sulfur and sulfate-sulfur, although analysis of other sulfur species such as elemental sulfur and barite-sulfur is sometimes justified. Caution should be exercised for certain samples in interpreting sulfate-sulfur analyses as this form can be either inert (eg. gypsum) or essentially stored products of acid drainage that could become mobilized if conditions within a waste change. Reference to the MEND Report 1.16.1c is suggested (Norecol Environmental Consultants, 1991) for a discussion of sulfur species.