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BEFORE THE WATER QUALITY CONTROL COMMISSION**

In the Matter of:)
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PROPOSED AMENDMENT)
TO 20.6.2 NMAC (Copper Rule))
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Chapter 15

STATIC-TEST METHODS MOST COMMONLY USED TO PREDICT ACID-MINE DRAINAGE: PRACTICAL GUIDELINES FOR USE AND INTERPRETATION

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INTRODUCTION

Acid mine drainage (AMD) is contaminated effluent from mines and mining wastes that results from the oxidation of iron-sulfide minerals exposed to air and water. The intensity and duration of AMD formation are complex functions of deposit geology, mineralogy, and hydrology, and the subsequent interaction of climatic conditions upon ore and waste when exposed by various mining methods. Because AMD can produce effluent containing acid- and heavy-metal concentrations that exceed water quality standards and is perceived as irreversible once started, it is one of the more vexing environmental problems facing land-managing agencies and the minerals industry today. Consequently, reliable prediction tools that quantify the risk for a particular mine waste to produce AMD are actively sought by the minerals industry and regulators.

Today numerous tools in the form of various laboratory "static"- and "kinetic"-predictive tests are available for fees that range from \$35 to as much as \$5,200 per sample. Static tests are short term (usually measured in hours or days) and relatively low cost per sample (from \$35 to \$135). Their objective is to provide an estimate of a mine waste's capacity to produce acid and its capacity to neutralize acid. One shortcoming of static tests is that they measure only the capacities for acid production and consumption and do not consider the differences between the respective dissolution rates of acid-producing and acid-consuming minerals. Another potential source of error inherent to static-test-data interpretation is the assumption that all acid-producing and acid-consuming minerals present will react completely, an assumption which ignores the influence of acid-producing and acid-consuming mineral particle-size and morphology.

Kinetic tests are long term (usually measured in months and sometimes years) and expensive per sample (from \$500 to as much as \$5,200). Their objectives are to confirm or reduce uncertainty in static-test classifications (i.e., the sample is either acid- or non-acid producing), identify dominant chemical-weathering reactions, and determine acid-generation rates and temporal variations in leachate water quality. This is accomplished by accelerating the natural weathering rate of a mine-waste sample under closely controlled laboratory conditions (Lapakko, 1988; Lawrence, 1990; White and Jeffers, 1994). One shortcoming of kinetic tests is the extended amount of time required to perform the tests, as it is not uncommon for these tests to continue for at least 20 weeks (e.g., Lapakko and Wessels, 1995).

As state and federal land-managing agencies have become aware of these predictive tests, they have required operators to subject larger populations of mine-waste samples to a variety of predictive tests as part of the permitting process. Because several different kinds of laboratory-predictive tests are now available, both operator and regulator have asked similar questions such as:

- 1) which tests are most commonly used today,
- 2) how are these tests performed, and
- 3) how accurate are these tests?

These questions are partially addressed by the objectives of this paper, which are to:

- 1) summarize mine-waste dissolution chemistry and its relationship to data produced by laboratory-predictive tests,
- 2) identify the static-test methods most commonly used in the United States and Canada,
- 3) summarize their protocols,
- 4) identify sources of error in neutralization potential (NP) determinations and quantify their influence on selected samples, and
- 5) suggest measures to improve NP determination accuracy.

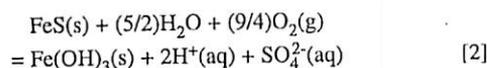
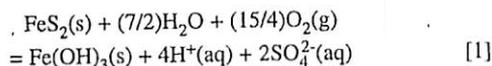
Previous analyses of predictive tests have been presented by Perry (1985), Ferguson (1985), Ferguson and Mehling (1986), Ferguson and Erickson (1988), Coastech Research Inc. (1989), Bradham and Caruccio (1991), and Lapakko (1992a, 1993, 1994a). This report includes relevant questions and conclusions from the more recent literature. It elucidates these points and draws additional conclusions based on data generated by selected static tests on mine-waste samples obtained from a variety of metal-mine settings. These tests were performed from 1991 through 1995 in the course of cooperative AMD-related studies by Minnesota Department of Natural Resources (MDNR) and the U.S. Bureau of Mines (USBM).

MINE WASTE DISSOLUTION

Sources of acid

The dissolution of iron sulfide minerals such as pyrite and pyrrhotite is responsible for the majority of mine-waste acid production (Stumm and Morgan, 1981). Equations [1] and [2] are commonly published reactions believed to represent typical pyrite and pyrrhotite chemical-weathering products (after Stumm and Morgan, 1981; Nelson, 1978):

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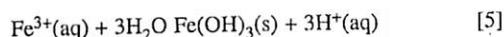
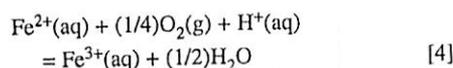
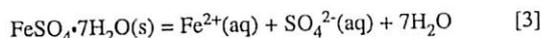
These weathering reactions produce acidic, iron- and sulfate-rich aqueous water which can

- 1) contact sulfide minerals and accelerate their oxidation,
- 2) evaporate partially or totally to precipitate hydrated iron-sulfate and other minerals and (or)
- 3) contact host rock minerals which react to neutralize some or all of the acid.

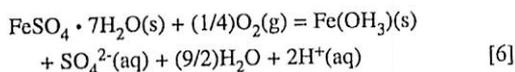
Acidic flow which migrates through the mine waste will exit as AMD.

Hydrated iron-sulfate minerals precipitate during the evaporation of acidic, iron- and sulfate-rich water within mine-waste materials and store (for potential subsequent release) acid generated by iron sulfide mineral oxidation. The more common hydrated iron-sulfate minerals that occur as efflorescent salts on the surfaces of weathering pyrite include melanterite, rozenite, szomolnokite, romerite and copiapite ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{FeSO}_4 \cdot 4\text{H}_2\text{O}$, $\text{FeSO}_4 \cdot \text{H}_2\text{O}$, $\text{Fe}^{2+}\text{Fe}_2^{3+}(\text{SO}_4)_4 \cdot 14\text{H}_2\text{O}$, and $\text{Fe}^{2+}\text{Fe}_4^{3+}(\text{SO}_4)_6(\text{OH})_2 \cdot 20\text{H}_2\text{O}$, respectively) (Alpers et al., 1994). According to Nordstrom (1982) and Cravotta (1994), these sulfate salts are highly soluble and provide an instantaneous source of acidic water upon dissolution and hydrolysis. They are partially responsible for increased acidity and metals loadings in the receiving environment during rainstorm events.

As an example, equations [3], [4], and [5] summarize the step-wise dissolution of melanterite.

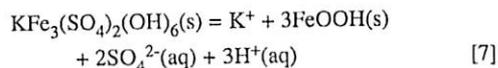


The net result of equations [3] through [5] is summarized in equation [6], which shows a net production of two moles, acid produced for each mole of melanterite dissolved.



Cravotta (1994) showed that a similar aqueous dissolution of romerite produced six moles of acid for each mole of romerite dissolved. The cumulative storage and incremental release of acid from these salts may help explain the lag from mine-waste placement to AMD-formation particularly in arid climates.

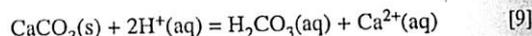
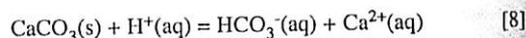
According to Nordstrom (1982), the formation of hydrated iron sulfates is an important intermediate step that precedes the precipitation of the more common insoluble iron minerals such as goethite and jarosite. However, jarosite is slightly soluble (Alpers et al., 1994) and can, therefore, contribute acid according to equation [7]. For example, recent preliminary leach studies



on natural and synthetic jarosites conducted by USBM showed a drop in pH from 6 in the deionized water leachate to 3 or 4 after contact with the jarosites. Because of its relatively low solubility, the acid contributed by jarosite dissolution is probably small relative to that by dissolution of more soluble hydrated iron sulfates.

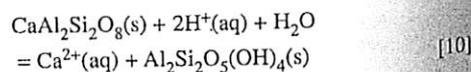
Sources of neutralization

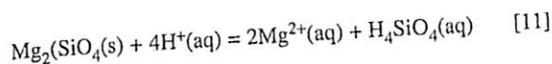
The balance between the rates of acid production by iron-sulfide mineral oxidation and host rock buffering will determine the acidity of mine-waste drainage. The most effective minerals for neutralizing acid are those containing calcium carbonate and magnesium carbonate. Examples include calcite, magnesite, dolomite, and ankerite (CaCO_3 , MgCO_3 , $\text{CaMg}(\text{CO}_3)_2$, and $\text{CaFe}(\text{CO}_3)_2$, respectively). Equation [8] represents the dominant dissolution reaction of calcite (CaCO_3) with iron-sulfide-generated acid (H^+) above pH 6.4, while equation [9] is the dominant reaction below pH 6.4 (Drever, 1988):



The dissolution rates for the calcite reactions shown in equations [8] and [9] are relatively rapid. However, dissolution rates are not the same for all carbonates; for example, Rauch and White (1977) and Busenberg and Plummer (1986) have reported that the rates of magnesium carbonate and calcium-magnesium carbonate (i.e., magnesite and dolomite) dissolution are substantially slower than that of calcium carbonate. Additionally, iron carbonates do not provide for net acid neutralization under oxidizing conditions, due to oxidation of the ferrous iron released, subsequent precipitation of ferric hydroxide, and the consequent acid production (reactions 4, 5).

Dissolution of silicates such as plagioclase-feldspar minerals (e.g., anorthite in equation [10], Busenberg and Clemency, 1976) and olivine minerals (e.g., forsterite in equation [11], Hem, 1970) can also neutralize acid under acidic conditions, but their dissolution rates (and subsequent acid neutralization) are slow relative to the carbonate minerals.





The effectiveness of silicate-mineral neutralization is thought to be optimized by these factors:

- 1) the acid-production rate is relatively slow,
- 2) feldspar minerals comprise a significant percentage of the overall mineralogy, and
- 3) the available silicate-mineral surface area is large (Morin and Hutt, 1994).

STATIC TEST METHODS

The most commonly-used static test is known as acid-base accounting (ABA; Ferguson and Erickson, 1988). Several variations of ABA are in use in the United States and Canada (Lapakko, 1992a); they include standard ABA (Sobek et al., 1978), modified ABA (Coastech Research Inc., 1989; Lawrence, 1990; Reclamation Research Unit and Schafer and Associates, 1987), the B.C. Research Initial Test (BCRI, Bruynesteyn and Duncan, 1979), and the NP(pH6) Test (Lapakko, 1994a). This report will focus on these tests.

The Net Acid Production (NAP) (Coastech Research Inc., 1989) and Net Acid Generation (NAG) (Miller et al., 1990) tests are based on the principle that hydrogen peroxide accelerates the oxidation of iron sulfide minerals. The acid consequently produced dissolves neutralizing minerals present, and the net result of the acid production and neutralization can be measured directly. This test does not require sulfur determinations and is, therefore, more readily conducted in a field laboratory than other static tests. Problems associated with this type of test and a modification of the protocol are presented elsewhere (Lapakko, 1992a, 1993; Lapakko and Lawrence, 1993). Hydrogen peroxide based tests are not discussed here. Likewise, the paste pH test (Sobek et al., 1978) and the acid concentration present test (Bucknam, personal commun., 1994), which measure the acid present on mine-waste particles, are not addressed in this report.

Acid-base accounting

Principles

The acid-base accounting (ABA) test was originally designed to evaluate the acid-producing capability of coal-mine wastes. It is now used to evaluate both coal- and metal-mine wastes. ABA measures the balance between the acid-producing potential (AP) and acid-neutralizing potential (NP) of each mine-waste sample. AP is determined by sulfur assay and represents the sulfur contained in acid-generating iron-sulfide minerals present in the sample. The AP value is calculated based on the assumption that two moles of acid will be produced for each mole of sulfur present (equations [1] and [2]). Because one mole of calcium carbonate will neutralize the two moles of acid (equation [9]), the percent sulfur obtained from the sulfur assay is multiplied by 31.25¹ to yield AP in units of tons calcium carbonate equivalent per thousand tons mine waste. NP is determined by subjecting the mine-waste sample to some form of acid digestion and represents the amount of acid-neutralizing carbonate minerals present in the sample. NP value is also reported in units of tons calcium carbonate per thou-

sand tons of mine waste. This report expresses AP and NP in equivalent and more concise units of kg calcium carbonate per ton of mine waste or kg/t CaCO₃.

Net-neutralizing potential (NNP), which is the difference between these values (NP - AP = NNP), is one of the measurements used to classify a mine-waste sample as potentially acid or non-acid producing. The NNP is often called the "acid-base account" of the sample. If NP is greater than AP, NNP is positive; conversely, if NP is less than AP, NNP is negative.

The question of how positive or negative the NNP must be for a sample to be considered safely non-acid producing (or conversely, definitely acid producing) has been a source of classification controversy. Recent ABA classifications for mine-waste sampling are based on both NNP and the NP/AP ratio (i.e., if NP > AP, then NP/AP > 1; conversely, if NP < AP, then NP/AP < 1). Three categories comprise the ABA classification ("high," "uncertain," and "low" acid-producing potential). The ranges of NNP and NP/AP values included in each of the three categories have been described by Brodie et al. (1991), Day (1989), Ferguson and Morin (1991), and Morin and Hutt (1994) and are shown in Table 15.1. Note the variability in the NNP values for each category as compared with the more consistent corresponding NP/AP values. Also note that NNP values have not been defined for the "non acid" category. Samples classified in the "uncertain" category are typically subjected to kinetic testing (Ferguson and Morin, 1991).

TABLE 15.1—Interpretation of acid-producing potential for samples subjected to ABA static tests, based on NNP (kg/t CaCO₃) and NP/AP ratio.

Acid-Base Account	Acid	Uncertain	Non-Acid
NNP:			
Appalachian coal-mine criterion ¹	< -5	ND	ND
B.C. metal-mine criterion ²	≤ 0	ND	ND
Ferguson & Morin, 1991, p. 86	ND	-20 < NNP < +20	ND
Day, 1989	< +10	ND	ND
NP/AP:			
Brodie et al., 1991, p. 121	< 1	1 < NP/AP < 3	> 3
Morin & Hutt, 1994, p. 148	< 1	1 < NP/AP < 1.3 to 4.0	> 1.3 to 4.0

¹Sobek, et al., 1978, p. 3.

²Ferguson and Morin, 1991, p. 86.

ND, not defined in reference.

K.A. Morin (1989, oral commun.) observed threshold NNP values for acid production of -15, +10, and +20 kg/t CaCO₃ for a modified procedure on various sets of samples.

The ABA classification that requires a mine-waste sample to meet or exceed an NP/AP ratio of 3 to 1 to be considered non-acid producing has interesting ramifications when sample sulfide content approaches 9%. If the sulfide is present as pyrite, the pyrite would represent about 17% of the sample mass; and, if the sulfide is present as pyrrhotite, the pyrrhotite would make up about 21% of the sample mass. In either case, all of the remaining sample

¹A 1 to 1 mole ratio of S to CaCO₃ (32 g/mole and 100 g/mole, respectively) is equal to a weight ratio of 1:3.125. Multiplying the weight-percent sulfur contained in the sample by 3.125 converts the sulfur to weight percent calcium carbonate required to neutralize the produced acid; multiplying by 31.25 yields the required amount of calcium carbonate in parts per thousand.

mass would have to be calcium carbonate to meet the 3 to 1 criterion. Therefore, mine-waste samples containing more than 9% sulfide would be classified as acid producers during the initial sample screening process (Lapakko, 1992a). Table 15.2 compares percent sulfide (converted to percent pyrite and pyrrhotite) with the corresponding percent of calcium carbonate required to be present in the sample to meet 3 to 1 criterion proposed by California and Montana, and 1.2 to 1 criterion required by Nevada (U.S. EPA, 1994, Table 7).

TABLE 15.2—Percent sulfide converted to percent pyrite and pyrrhotite, and compared with corresponding percent calcium carbonate required to meet NP/AP 3:1 and 1.2:1 criteria.

Sulfide (S ²⁻)	Weight Percent		NP/AP	
	Pyrite (FeS ₂)	Pyrrhotite (Fe ₅ S ₆ - Fe ₁₆ S ₁₇) ¹	3:1 ² % CaCO ₃	1.2:1 ³ (% CaCO ₃)
0.25	0.47	0.61 - 0.66	2.34	0.94
0.50	0.94	1.23 - 1.32	4.69	1.87
1.00	1.88	2.46 - 2.65	9.38	3.75
3.00	5.63	7.38 - 7.94	28.13	11.25
5.00	9.38	12.29 - 13.24	46.88	18.75
7.00	13.13	17.21 - 18.53	65.63	26.25
8.50	15.94	20.90 - 22.50	79.69	31.88
8.89	16.67	NAP	83.34	33.34

¹Range of compositional variation (Dana, 1963, p. 428).

²California and Montana (% S²⁻ × 3.125 × 3 = % CaCO₃).

³% S²⁻ × 3.125 × 1.2 = % CaCO₃.

NAP, not applicable.

Protocols

Standard ABA Method—This method was developed by Smith et al. (1974) and later modified by Sobek et al. (1978); it is often referred to as the standard Sobek method. The AP is determined based on the total sulfur content obtained by sulfur assay and assumes that all sulfur is present as sulfide.

The first step in determining the NP is a "fizz" test which is designed to estimate the calcium carbonate and magnesium carbonate content of the sample. An acid solution comprised of one part concentrated hydrochloric acid to three parts water is applied dropwise to 0.5g of sample, and the extent of "fizzing" is observed (the "fizzing" is the result of the reaction of the acid and carbonate present in the sample—equation [9]). Based on the vigor of this reaction, the volume and concentration of hydrochloric acid to be added to 2g of minus 60-mesh mine-waste sample are determined ("no fizz," 20 ml 0.1N; "slight fizz," 40 ml 0.1 N; "moderate fizz," 40 ml 0.5 N; "strong fizz," 80 ml 0.5 N). The mixture of acid and mine-waste sample is then boiled until the reaction has ceased (as indicated by cessation of bubble production). After the boiling step, the mixture is cooled and titrated to pH 7.0 with sodium hydroxide (NaOH) to measure the amount of acid consumed during its reaction with the sample.

Modified ABA Method—This method, which is also known as the modified Sobek method was developed by Coastech Research Inc. (1989) and described by Lawrence (1990). While similar to the Standard ABA Method in sample mass and acid concentration and volume used, the modified ABA method bases the AP on sulfide content rather than total sulfur. This requires additional steps during analysis to speciate the dominant sulfur forms

present in the sample. This speciation can be accomplished with either a combustion-infrared spectrophotometer (e.g., a LECO furnace), or a sequential, wet-chemical leach (Sobek et al., 1978; see "Carbon-Sulfur Analysis" for details). Basing the AP on the sulfide-sulfur content assumes that sulfur present as sulfate is not acid producing (e.g., sulfate minerals such as gypsum and barite, which are common to some western U.S. metal mines). In place of a heated acid digestion, the modified ABA uses a 24-hour, ambient temperature, agitated acid digestion to determine the NP. At the completion of the 24-hour digestion, the mixture of acid and mine-waste sample is required to have a pH range of 1.5 to 2.0 (if not, the test is re-run with an acid addition that is adjusted based on the previous test's final pH). A titration endpoint of pH 8.3 is used rather than the reportedly unstable pH 7.0 value used in the standard ABA method (Coastech Research Inc., 1989).

B.C. Research Initial Method—(BCRI, Duncan and Walden, 1975; Bruynesteyn and Duncan, 1979). Although this method uses different terminology and different units of quantification, the terminology and units are translated to be consistent with those of the ABA methods for ease of presentation. As with the standard ABA, AP is based on total sulfur content. NP is determined by titrating, with 10N sulfuric acid, a stirred mixture of 10g mine waste (70% minus 325 mesh or, equivalently, 70% of the particles having diameter less than 0.044 mm) and 100 ml distilled water. The titration is continued until pH 3.5 is reached and less than 0.1 ml of acid is added over a period of 4 hours. More recently, a 1.0N sulfuric acid titrant has been used (O'Hearn, personal commun., 1996).

NP(pH6) Method—The NP(pH6) is the same as the B.C. Research Initial method except for the strength of acid and the endpoint used. Specifically, a slurry comprised of 10g solid and 100 ml deionized water is titrated with 1N sulfuric acid until pH 6.0 is reached, and less than 0.1 ml of acid is added over a period of 4 hours. The resulting volumes of acid for the respective endpoints are then converted to parts per thousand calcium carbonate equivalent (NP). The NP obtained at pH 6 is termed the "effective" NP, or the calcium carbonate equivalent available in the sample to maintain the pH above 6 (Lapakko, 1992b). The NP(pH6) method differs significantly from the previously mentioned tests in that the "digestion" occurs at pH 6 rather than in a more acidic environment.

Modified NP (pH6) Method—A modification of the NP (pH6) method was necessary to evaluate mine-waste samples that contained only traces of actual calcium- or magnesium-carbonate mineral NP, or whose carbonate minerals were mainly iron carbonate (e.g., siderite, ankerite, and ferroan dolomite). These conditions caused titration endpoints to be exceeded with addition of the first drop of titrant during BCRI and NP (pH6) titration methods. The problem was resolved by distributing six to eight 10-g aliquots of the solid sample to separate 400-ml beakers where each sample was slurried with 100 ml of deionized water. No sulfuric acid was added to the slurried sample in the first beaker which served as a control. Pre-selected, progressively-increasing volumes of acid were added to each of the sample slurries contained in their respective beakers (numbered 2 through 6 or 8). All of the sample beakers were then placed on an orbital shaker and agitated for a total of 4 hours. After the first and second 0.5-hour periods, and after three subsequent 1-hour increments, agitation was stopped and pH measurements were taken and recorded for each sample. Recorded pH obtained at specified time intervals from each sample was converted to corresponding hydrogen ion

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concentration. Hydrogen ion concentration representing pH 6 and its corresponding acid volume was estimated by interpolation for each sample. The estimated acid volume at pH 6 was converted to calcium-carbonate equivalent NP. The NP obtained at pH 6 is termed the "effective" NP, or the calcium carbonate equivalent available in the sample to maintain the pH above 6 (Lapakko, 1992b).

Carbon-Sulfur (C-S) Analysis—Bucknam (1995a) is developing a consensus-based ASTM-standard method for total carbon and sulfur determination to estimate carbonate, sulfide, and subsequent acid-base account in metal-mine ore, concentrate, tailings and waste-rock samples. The approach is predicated on the concept that all sulfide present is associated with iron (and therefore can be used to quantify acid production potential) and all carbonate is associated with calcium and magnesium (and therefore can be used to quantify acid neutralization potential). Total carbon and sulfur analyses for these samples are determined by a combustion-infrared spectrophotometer (e.g., a LECO furnace). Carbonate and sulfide are determined from sample duplicates through partial-decomposition procedures, followed by combustion-infrared spectrophotometer C-S analysis. The partial-decomposition procedures include pyrolysis (muffle roast at 550°C) and chemical decomposition (individual hydrochloric-acid, nitric-acid, and sodium-carbonate leaches).

The muffle-furnace roast at 550°C drives off sulfide sulfur as sulfur dioxide, and organic carbon as carbon dioxide. The pretreated sample is then analyzed for carbon and sulfur, and the results are subtracted from the total carbon and sulfur results of a non-pretreated sample split. The remaining sulfur determined in the pretreated split is assumed to be sulfate sulfur, and the remaining carbon is considered to be carbonate.

Chemical decomposition using hydrochloric acid is intended to solubilize carbonate minerals. Total carbon and sulfur values from the hydrochloric-acid pretreated sample represent organic carbon, pyritic sulfur, some trace-metal sulfides, and organic sulfur; carbonate carbon is estimated by difference. The purpose of nitric acid decomposition is to oxidize sulfides to sulfate. Sulfate reports to the acid leachate and consequently is separated from the solid phase. The sodium carbonate leach is intended to solubilize all sulfates (with the exception of barite—BaSO₄) while most sulfides are unaffected and remain in the solid phase. When samples are subjected to chemical decomposition, their sulfide-sulfur contents are estimated by subtracting the nitric-acid residual-sulfur value from the sodium-carbonate residual-sulfur value.

This proposed ASTM method for ABA by C-S analysis has been distributed for interlaboratory testing. It will be available

after tests are completed and the method has received the required ASTM-committee approvals (Bucknam, 1995b, written communication.).

An alternative sulfur-speciation method to the C-S analysis method is the sequential, wet-chemical-leach speciation method for sulfur forms (total, sulfide, and sulfate) described in Sobek et al. (1978).

Protocol modifications for this study—Three modifications of standard protocols were made in USBM methods used for the present study. First, a standard minus 150 mesh particle size was used for the standard ABA, modified ABA, and modified NP(pH6) tests. This was done to normalize for particle size effects. The minus 150 mesh fraction was selected since it was used by USBM to prepare samples for chemical analysis. Two additional changes were made to the standard ABA method:

- 1) the titration endpoint of pH 7.0 was changed to the more stable endpoint of pH 8.4, and
- 2) the qualitative "near boiling" temperature for the acid leach was modified to heat the sample pulp for 1 hour in a water bath maintained at 85°C.

RESULTS AND DISCUSSION

Sources of error from protocol variables

Static tests quantify the potential of mine waste samples to produce and neutralize acid. The commonly used static tests quantify acid producing potential (AP) using either total sulfur or sulfide-sulfur content to estimate the quantity of acid-producing minerals present (Table 15.3). The total sulfur content will overestimate the actual AP of samples containing substantial non acid producing sulfate minerals (e.g., barite or gypsum). On the other hand, the sulfide-sulfur measurement will underestimate the actual AP of samples containing substantial acid-producing sulfate minerals (e.g., melanterite or jarosite, reactions 6, 7). Knowledge of the mine waste sulfate mineralogy will indicate if the sulfate minerals present, if any, are acid producing and, thereby, allow selection of the more appropriate AP quantification. Existing techniques, such as those using a combustion furnace with subsequent quantification of the sulfur dioxide evolved (e.g., LECO furnace, as applied in ASTM E-1019, ASTM E-395, ASTM D-4239), are capable of accurately determining either total sulfur or sulfide-sulfur and, thereby, the AP.

Different static-test methods can produce markedly different NP values for the same sample (Lapakko, 1992a, 1992b, 1994a).

TABLE 15.3—Standard and modified ABA, BCRI, and NP(pH6) static test methods.

ABA method	Particle size (mesh) ¹	AP Sulfur form	(Acid, and N)	NP-Determination Acid Leach		Temperature (°C)
				pH (range)	Duration (hours)	
ABA, standard	-60	Total	HCl, 0.1–0.5	0.5–7.0 ²	1	85
				1.5–2.0 ³	24	25
ABA, modified	-200	Sulfide	HCl, 0.1–0.5	3.5	4+	25
BCRI	-100 to -400	Total	H ₂ SO ₄ , 1.0	6.0	4–120	25
NP(pH6)	-150 to -325	Sulfide	H ₂ SO ₄ , 0.1–1.0			

¹Particle size for the BCRI test was reported as -100 mesh by Bruynesteyn and Duncan (1979) and -400 mesh by Bruynesteyn and Hackl (1982). 75% passing 325 mesh has been used by industry laboratories (Lapakko 1994a).

²pH is not controlled with this method.

³This range was used by Lawrence Consulting LTD. (1991); a range of 1.0 to 1.6 was reported in an earlier publication (Lawrence 1990).

Protocol variables which may contribute to these differences include mine-waste particle size (tailings are typically run "as received"); "digestion" variables such as the acid used, amount of acid added (i.e., digestion pH), temperature, and duration; and the endpoint pH of the "back titration," if a back titration is used (Table 15.3). The most influential of the protocol variables are particle size, extent of acid addition, and the back titration endpoint. The extent to which protocol variables will affect the measured NP is dependent on the sample mineralogy.

Particle size—To examine the effect of particle size on NP, the standard ABA method was used to determine the NP of the -1/4 inch, -60 mesh, -150 mesh, and -325 mesh fractions of three rock samples (Table 15.4). The minus 1/4-inch size was included because it is commonly used in humidity-cell accelerated-weathering tests, such as those described in White and Jeffers (1994). For each of the rock types, NP increased as particle size decreased. This trend reflects increasing dissolution of acid-neutralizing minerals, due to increasing mineral surface area with decreasing particle size (e.g., Lapakko and Antonson, 1991). Thus, the reduction of particle size in neutralization potential determinations increases the dissolution of acid-neutralizing minerals which, in turn, leads to overestimation of the NP of the larger particles. The extent of this overestimation would tend to be greater for the BCRI and NP(pH6) tests, in which finer particle size fractions are used (Table 15.3).

TABLE 15.4—Standard-ABA NP increased as particle size decreased.

Sample size (mesh)	Sample NP (kg/t CaCO ₃)		
	1-C	MN-4	3-A
-325 ¹	8	15	943
-150 ¹	6	12	915
-60 ¹	6	11	900
-1/4 inch ²	0.09	2	ND

¹2-g sample mass.
²20-g sample mass.
 ND, not determined.

Amount of acid added—The amount of acid added during the digestion can have a substantial influence on the NP value determined. As the acid addition increases, pH tends to decrease. This, in turn, leads to increased dissolution of acid-neutralizing minerals and a consequent increase in the NP measured. Indeed, the general approach for determining NP has been criticized since the low pH in the strongly acidic digestions may dissolve minerals, with consequent acid neutralization, which would not dissolve (and neutralize acid) at circumneutral pH (Lutwick, 1986). That is, minerals which dissolve to neutralize acid at low pH in NP digestions may not dissolve fast enough (at higher pH) to maintain neutral pH conditions in the environment. Consequently, NP digestions have been reported to generally overestimate the environmentally practical capacity of mine wastes to neutralize acid (Lapakko, 1994a). One example of such overestimation is reported for Duluth Complex rock, in which silicate mineral dissolution contributed to the standard ABA NP. In both laboratory and field tests, this dissolution neutralized acid produced by oxidation of sulfide minerals present in the rock. However, the rate of dissolution was not fast enough to maintain drainage pH above 6.0 even for rock of moderate sulfide mineral content (Lapakko, 1988, 1990; Lapakko and Antonson, 1994; Lapakko, 1994b).

To illustrate this phenomenon, the standard ABA NP of 14 samples was compared to the amount of acid neutralized by the samples prior to drainage pH decreasing below 6.0 in laboratory tests. This observed neutralization potential has been referred to as the "empirical NP" (Lapakko, 1994b). In all 14 cases the standard ABA NP overestimated the empirical NP by 5 to 21 kg/t CaCO₃ (Table 15.5). Even after drainage from these rocks had acidified (decreased below pH 6.0), indicating that no neutralization potential remained, the standard ABA tests conducted on the leached solids indicated a residual NP. Examination of the unleached, empirical, and leached NP values clearly indicates that the standard ABA NP overestimates the actual capacity of these mine wastes to neutralize acid while maintaining drainage pH above 6.0.

TABLE 15.5—Standard ABA NP (kg/t CaCO₃) of fresh and leached Duluth Complex drill core samples.

Sample	PCT S	Unleached NP	Empirical NP ¹	Minimum pH	Leached NP	Change in NP
G	0.92	21.8	0.40	4.20	13.9	-7.9
H	0.92	21.8	0.41	4.55	20.8	-1.0
K	1.24	12.6	0.61	4.20	11.8	-0.8
L	1.24	12.6	0.58	4.15	12.9	+0.3
C	1.35	10.5	0	3.75	10.2	-0.3
Q	1.74	9.1	0	4.05	5.9	-3.2
R	1.74	9.1	0	4.00	7.2	-1.9
S	1.87	23.7 ²	2.0	4.50	8.3	-15.4
T	1.87	23.7 ²	1.8	4.20	7.8	-15.9
B	2.01	7.9	0	3.70	8.3	+0.4
U	2.17	7.9	0.22	3.90	35.1 ³	+27.2
V	2.17	7.9	0	4.55	35.1 ³	+27.2
W	2.57	5.1	0	3.95	2.4	-2.7
X	2.57	5.1	0	3.95	3.7	-1.4

¹Neutralization potential above pH 6 observed in laboratory dissolution test.

²Average of duplicate values: 20.4, 27.1 kg/t CaCO₃.

³One sample of U, V combined. The reason for the increase in NP after leaching is unknown.

This error can be exacerbated and reproducibility hindered due to the potential for variable acid addition in the standard ABA and modified ABA NP digestions. Acid addition can vary with the standard ABA and modified ABA (although to a lesser degree) but not with the BCRI "titration" of a mine waste sample, during which mineral dissolution and neutralization occurs at pH greater than or equal to 3.5. This is higher than the typical pH range in the ABA test and the prescribed modified ABA test range (1.5–2.0) and, consequently, acid neutralization due to the dissolution of relatively unreactive minerals would contribute less "false NP" in the BCRI test. Nonetheless, mineral dissolution and acid neutralization can occur in the BCRI test at lower pH values, and may not be rapid enough to maintain mine waste drainage pH above 6.0. Contributions from low pH mineral dissolution and acid neutralization are eliminated in the NP(pH6) test.

The acid added in the standard ABA digestion varies from 20 ml 0.1N to 80 ml 0.5 N hydrochloric acid, a twenty-fold variation based on a subjective interpretation of the "fizz" test. This range of acid addition can produce a digestion pH ranging from 0.5 to 7. In the modified ABA the amount of acid added is based on the "fizz" test, but is also limited by requiring a pH range of 1.5 to 2.0 at the end of the digestion. During the BCRI and NP(pH6) "titrations" of a mine waste sample, mineral dissolution and neutraliza-

tion occurs at pH greater than or equal to 3.5 and 6, respectively. Lapakko (1992b) reported that increasing H⁺ additions for the modified ABA digestion by factors of 1.25 to 5 increased the resultant NP by an average of 27 kg/t CaCO₃, with a maximum increase of 76 kg/t CaCO₃ (Table 15.6). These values demonstrate graphically the influence of the digestion acid strength on NP. However, the actual impact on modified ABA values is limited since the protocol requires a digestion pH in the range of 1.5 to 2.0. Thus, the potential range of the acid addition is limited by the objective criterion of the target pH range rather than the subjective interpretation of the "fizz" test (Lapakko, 1992b).

TABLE 15.6—Effect of acid addition on NP (pH 8.3 titration endpoint) determined by modified ABA method (Lapakko, 1992b). pH₀ = pH at beginning of titration. NP in kg/t CaCO₃.

Sample	ml HCl	N HCl	H ⁺ equivalents	pH ₀	NP
T1	40	0.100	0.004	4.83	200
	40	0.300	0.012	1.37	187
	40	0.500	0.020	0.65	205
T2	40	0.300	0.009	2.09	170
	30	0.300	0.020	0.64	200
	40	0.500	0.004	4.39	78
T3	40	0.100	0.010	1.20	146
	20	0.527	0.020	0.81	148
	40	0.500	0.004	4.39	76
T4	40	0.100	0.004	4.39	76
	40	0.100	0.004	4.39	76
	40	0.100	0.004	4.39	76
T4	40	0.100	0.004	4.39	76
	20	0.527	0.010	1.14	144
	40	0.500	0.020	0.62	152
T5	40	0.100	0.004	2.14	69
	40	0.100	0.004	2.14	69
	60	0.105	0.006	1.65	92
T6	40	0.105	0.006	1.65	92
	40	0.500	0.020	0.51	88
	40	0.100	0.004	2.14	41
T7	40	0.100	0.004	2.14	41
	60	0.105	0.006	1.76	62
	60	0.105	0.006	1.76	62
T7	30	0.527	0.016	1.28	219
	40	0.500	0.020	0.74	226
	40	0.500	0.020	0.74	226
T8	40	0.100	0.004	4.59	69
	20	0.527	0.010	2.18	109
	20	0.527	0.010	2.18	109
T9	20	0.105	0.002	1.85	22
	40	0.100	0.004	0.51	16
	40	0.100	0.004	0.51	16
T10	35	0.527	0.018	1.22	204
	40	0.500	0.020	1.00	210
	40	0.500	0.020	1.00	210

In contrast, the impact in the standard ABA test can be substantial, since the acid addition is based strictly on a subjective evaluation of the "fizz" test by the laboratory technician. Differing evaluations of this test lead to different acid additions and, consequently, variations in the NP determined. Lapakko (1991) reported that increasing the acid addition to a hydrothermal-quartz-carbonate gold tailing sample from 40 ml 0.1N HCL to 40 ml 0.5N HCL, increased the resultant ABA NP from 80 to 174 kg/t CaCO₃. The initial digestion pH values for the two acid additions were 4.87 and 1.17, respectively.

Back titration endpoint—The ABA and modified ABA tests use back titrations, to respective endpoints of 7.0 and 8.3, following the digestion step to determine the amount of acid remaining in solution. With hydrothermal quartz-carbonate hosted gold tailings, modified ABA NP values using a pH 7.0 endpoint were typically 20 to 30 kg/t CaCO₃ higher than those for the pH 8.3 endpoint, with a maximum increase of 165 kg/t CaCO₃ (Table 15.7). This suggests that the pH 7.0 titration endpoint did not account for all of the acidity in solution. In particular, the lower titration end-

point did not allow for the oxidation of ferrous iron (released from iron carbonate minerals, for example) and subsequent precipitation of iron hydroxide (Lapakko, 1992b). The contribution of iron and other metals in solution can be accounted for by peroxide addition, as described in methods for determination of mineral acidity (ASTM Method D1067 or American Public Health Association et al., 1992). Since the BCRI and NP(pH6) methods do not include a back titration, acidity released during the digestion step (e.g., iron) is not accounted for.

TABLE 15.7—Comparison of ABA NP values determined at pH endpoints of 7.0 and 8.3. Acid addition for ABA was 40 ml of 0.5 N HCl unless otherwise noted (Lapakko, 1992b).

Sample	Standard ABA NP, kg/t CaCO ₃	
	pH 7.0 endpoint	pH 8.3 endpoint
T1	231	205
	228	208
T2	202	173
	202	173
T3	188	163
	190	162
T4	178	156
	106	98
T5	91	85
	69	48
T6 ¹	270	241
	174	142
T7	80	74
T8	18	17
T8 ¹	18	17
T9	373	208
T9 ¹	373	208
T10		

¹Acid addition 40 ml of 0.1 N HCl.

Acid type—Except for the influence of digestion duration on the NP(pH6), there is little evidence to indicate the influences of the remaining digestion variables (acid type, temperature, duration) are substantial. Standard and modified ABA digestions use hydrochloric acid, while the BCRI and NP(pH6) tests use sulfuric acid. Pyrrhotite is more soluble in hydrochloric than sulfuric acid and, therefore, might be expected to contribute more false NP in the standard and modified ABA tests than the BCRI and NP(pH 6) tests. Jennings and Dollhopf (1995) reported an average of 80% of three pyrrhotite samples was solubilized by treatment with 4.91 M HCl (approximately 150 ml HCl/g -100-mesh pyrrhotite).

However, solubilization of pyrrhotite under the conditions of the ABA NP determination appears to be slight. No increase in ABA NP values was observed as the pyrrhotite content of Duluth Complex rocks increased from 1.2% to 6.5% (Lapakko, 1995). To further examine this phenomenon, the USBM subjected pyrrhotite-bearing samples of Duluth Complex rock to a procedure similar to the ABA neutralization-potential determination. Samples of 80% passing 150 mesh were subjected to 40 ml 0.1 N HCl (plus 60 ml E-pure water) for one hour at 80°C. The pyrrhotite dissolution during this digestion was minimal. The average sulfur content (LECO furnace) of the three samples was 1.50±0.10% after the test as compared to 1.60±0.03% before the digestion. Thus, it is concluded that little false NP would be contributed by pyrrhotite dissolution in the presence of hydrochloric acid in the standard and modified ABA static tests. Apparently either

1) pyrrhotite was solubilized by the more concentrated hydrochloric acid used by Jennings and Dollhopf (1995) but not by lower

- concentrations in the standard ABA, or
 2) there was some difference between the pyrrhotite present in the samples examined in the two studies.

Digestion temperature and duration—Relative to the modified ABA, the elevated temperature used in the standard ABA digestion may enhance the dissolution of host rock minerals. On the other hand, a greater degree of neutralizing mineral dissolution would be expected with the longer digestion duration of the modified ABA test. However, previous research indicated that, within the ranges typical of standard and modified ABA testing, the influence of elevated temperature and test duration on 12 mine waste samples of variable composition (Coastech Research Inc., 1989) and 10 hydrothermal-quartz-carbonate-hosted-gold tailings was negligible (Lapakko, 1992b).

To examine the effect of modified NP(pH6) digestion duration on NP results, USBM subjected five samples to this test. Sample 3-A is nearly 100% calcite; 3-B is comprised of 60% carbonate as dolomite/ankerite; sample 3-D contains approximately 5% carbonate as ankerite and dolomite; and the final two samples were purchased specimens of calcite and siderite. With this digestion the NP values after 120 hours of digestion were typically 1.1 to 2.3 times those after a 4-hour digestion (Table 15.8). The increase for samples dominated by calcite (3-A and calcite sample) was small relative to that for samples dominated by magnesium and iron carbonates, reflecting the relatively rapid dissolution of calcite. That is, the most of the calcite dissolved within the first four hours of digestion, while the majority of magnesium carbonate dissolved between 4 and 120 hours. Thus, for this test, measurement of NP contributions from magnesium carbonates requires a longer duration than those from calcium carbonate.

TABLE 15.8—Successive NP results from 4-, 24-, and 120-h modified NP(pH6) leaches of two mineral specimens and three carbonate-rock samples.

Sample	Neutralization potential (kg/t CaCO ₃)		
	4 hours	24 hours	120 hours
3-A ¹	737	790	864
3-B ²	105	204	250
3-D ³	10	19	32
Calcite	859	ND	945
Siderite	2	ND	9

ND, not determined.

¹Approximately 100% calcite.

²60% carbonate as dolomite and ankerite.

³Approximately 5% dolomite.

Effect of sample mineralogy

The extent to which protocol variables will affect the NP determined by various tests is dependent upon the mineralogy of the sample under examination. Tests were conducted on carbonate and feldspar minerals to assess their contribution to measured NP.

Carbonate minerals—Two relatively pure carbonate minerals ("Iceland-spar" calcite and siderite) were purchased from a commercial supplier. Their X-ray-diffraction (XRD) patterns were nearly perfect matches with respective patterns of their corresponding XRD-reference samples. XRD analysis detected some MnCO₃ in the calcite sample, and some mixed carbonates of cal-

cium, magnesium, iron, and manganese in the siderite sample. X-ray fluorescence analysis of the samples indicated traces of manganese, iron, and strontium in the calcite sample and traces of manganese and calcium in the siderite sample. The purchased samples were pulverized to 80% passing 150 mesh and, along with mixtures of the two samples, subjected to four different NP procedures (C-S analysis, standard ABA, modified ABA, modified NP(pH6)).

The C-S method determined the "calcite" sample was 57% carbonate (CO₃) which, assuming none of the carbonate was associated with iron or manganese, implies an NP of 950 tons CaCO₃/1000 tons rock. This value yields an upper bound for the NP present with calcium and magnesium carbonates, assuming the technique accurately determined the carbonate content of the sample. Furthermore, it indicates that approximately five percent of the sample was comprised of noncarbonate minerals. The agreement among the three remaining static tests was good, and the values determined were roughly 90% of the maximum calcium/magnesium carbonate content indicated by the C-S method (Table 15.9). The difference between the results of these three tests and the C-S determination may be due to (1) the presence of some iron and (or) manganese carbonate (approximately 10% of the entire sample) or (2) incomplete digestion of the sample by the standard and modified ABA and modified NP(pH6) methods.

TABLE 15.9—Neutralization potential (NP) of calcite and siderite using four different techniques (kg/t CaCO₃).

Sample	Expected value ¹	NP-determination methods			
		C-S analysis	ABA, standard	ABA, modified	NP(pH6), modified ²
Calcite	1000	948	863	858	859
Siderite	0	772	763	632	2
5:5:90 ³	50	86 ⁴	89.5	NA	35.2
10:10:80 ³	100	172 ⁴	165	NA	86.5
10:20:70 ³	100	249 ⁴	241	NA	94.9
20:10:70 ³	200	267 ⁴	254	NA	178

NA, not analyzed.

¹Expected NP = percent calcite x 10.

²4-h test duration.

³Ratio of calcite: siderite: quartz.

⁴Calculated based on values from "pure" samples.

Underestimation of the total calcium/magnesium carbonate content is atypical of static test NP determinations in general, but may have resulted from the extremely high calcite content of the sample. The amount of acid added in the standard and modified ABA was precisely that required to dissolve a sample of pure calcite. In other words the acid added in these two tests was only slightly in excess of the carbonate mineral content and, consequently, the digestion of the calcite may have been incomplete due to slow reaction at higher pH. Similarly, the modified NP(pH6) digestion of calcite may have been incomplete due to the near neutral conditions of this test.

For the siderite sample the C-S method yielded a carbonate (CO₃) content of 46.3% which implies an NP of 772 tons CaCO₃/1000 tons rock, assuming none of the carbonate was associated with iron. Based on the XRD analysis of the sample it is more reasonable to assume the carbonate was associated with iron, which yields a siderite content of 89% and a noncarbonate mineral content of about 11% for the sample. This further implies a cal-

cium/magnesium carbonate NP of 0. The standard and modified ABA techniques both yielded NP values well above the expected value of 0 kg/t CaCO₃ (Table 15.9). This indicates that the digestions dissolved the siderite, but that the oxidation of ferrous iron and subsequent precipitation of ferric oxyhydroxide in the back titration was incomplete (see also Lapakko, 1992b, 1994a). The modified ABA technique yielded a lower NP, indicating a lesser attack of the siderite during the digestion (Table 15.9).

The modified NP(pH6) was in excellent agreement with the expected value, and indicated that the dissolution of siderite was minimal at the higher digestion pH of this test. That is, only a small amount of acid could be added to the mixture of water and siderite sample and still maintain a pH of 6. Apparently the dissolution of siderite was minimal in the less acidic NP(pH6) digestion. If significant siderite dissolution did occur, the ferrous iron released was oxidized and precipitated as ferric hydroxide with the consequent acid production (equations [4] and [5]). Thus, this acidity would be accounted for in this test rather than being neglected due to its presence as aqueous ferrous iron, which can occur in other static tests.

The trends observed for the calcite: siderite: quartz mixtures semi-quantitatively reflected the analyses of the "pure" samples. The standard ABA NP values exceeded the expected values by an average of almost 80%, further supporting a substantial contribution of "false" NP by siderite in the standard ABA. In contrast, the modified NP(pH6) values were an average of 14% below the expected values, suggesting an incomplete digestion of the calcite. These errors were calculated assuming both the "calcite" and "siderite" samples were pure mineral phases. Since the C-S analysis indicates the "calcite" sample was less than pure, it is likely that the actual errors in the ABA NP values were slightly higher, while those for the modified NP(pH6) method were slightly lower.

Feldspar minerals—To determine the extent to which various feldspar minerals contribute to the NP determined by standard ABA, three different specimen-grade feldspars were subjected to a standard-ABA NP digestion. Acid additions for this test produce a calculated pH range of 0.5 to 1.8, prior to mine waste sample addition. Under these low pH conditions, a reaction similar to equation [10] occurs. Potassic feldspar (microcline), and the calcic and sodic end members of the plagioclase feldspars (bytownite-AN70-90 and oligoclase-AN10-30, respectively) were selected because they are common mineral constituents of rock types common to mine waste from metal mines. After completion of the acid leach, the leach mixture was filtered and the filtrate analyzed for potential feldspar-dissolution products (Al, Ca, K, Na, Si).

Table 15.10 shows that bytownite is solubilized to a much greater extent than either oligoclase or microcline. Concentrations of selected cations from bytownite dissolution are one to two orders of magnitude greater than corresponding cation concentrations for oligoclase and microcline. Cation concentrations for oligoclase and microcline were nearly identical. Additionally, bytownite dissolution has a buffering effect on the acid leach that is not demonstrated by either oligoclase or microcline (pH 3.5 versus pH 2.0).

A second standard-ABA NP-determination acid leach was conducted on splits of the same bytownite and oligoclase samples to determine if measurable NP would result from the rigorous leach conditions. Because the previous microcline and oligoclase acid-leach results were nearly identical (Table 15.10), NP was not determined for microcline. Resulting NPs are listed in Table 15.11. Results are consistent with the acid-leach results summa-

rized in Table 15.10; bytownite NP ranged from 12 to 24, while oligoclase NP ranged from 0.0 to 0.4. Although calcium carbonate was totally absent from the specimen-grade feldspars, bytownite dissolution resulting from the standard-ABA acid leach produced measurable NP and erroneously suggested the presence of more than 2% calcium carbonate. The significance of the tests summarized in Tables 15.4, 15.10, and 15.11 is that NP can be overestimated by tens of parts per thousand due to differences in sample-size reduction and unanticipated dissolution of silicate gangue minerals.

TABLE 15.10—Filtrate analyses¹ after acid leaches (standard ABA method) of three non-carbonate-bearing specimen-grade feldspars

Sample	Oligoclase (sodic)	Bytownite (calcic)	Microcline (potassic)
pH	2.02	3.46	1.99
Na	4.30	31.40	4.30
Ca	5.19	66.10	5.26
K	1.40	4.60	2.10
Si	1.50	128.00	<1
Al	2.04	99.40	2.04

¹Cation concentrations in mg/l (parts per million)

TABLE 15.11—Comparison of average pH and range of NP values determined from acid leaches (standard ABA method) of specimen-grade bytownite and oligoclase feldspars.

Sample	pH	NP ¹
Bytownite (calcium feldspar)	2.6	12-24
Oligoclase (sodium feldspar)	1.9	0.0-0.4

¹Expressed as kg/t CaCO₃.

It should, however, be noted that feldspars can provide practical acid neutralization. Morin and Hutt (1994) reported that for more than a decade of weathering in the field, the 50% calcium feldspar present in tailings neutralized the acid produced by the oxidation of pyrite present in a concentration of 1.9%. Factors which enhanced the effectiveness of feldspar neutralization in this case were

- 1) the relatively fine particles and consequent high specific surface area of the feldspar,
- 2) the feldspar was largely calcium feldspar, which is more reactive than sodium or potassium feldspar (Table 15.10), and
- 3) possible subaqueous conditions which would limit the rate of pyrite oxidation and consequent acid production.

Comparison of NP determinations on various mine waste samples

The extent to which various NP methods will differ for a given mine waste sample is dependent upon the sample composition. Lapakko (1992b, 1994a) compared various static test NP values to each other and to the total calcium carbonate and magnesium carbonate content, or mineralogic NP (equation [12]). The mineralogic NP was used to estimate the actual neutralization potential of mine waste samples, assuming that calcium carbonate and

magnesium carbonate present in the mine waste samples, and only these minerals, would dissolve to maintain a pH of at least 6.0. The mineralogic NP allows estimation of Static-test NP accuracy in the absence of adequate laboratory or field data required to determine the empirical NP (see definition of "empirical NP" on page 330 under the heading "Amount of acid added").

$$\text{Mineralogic NP} = 10 \times (\% \text{CaCO}_3) + 11.9 \times (\% \text{MgCO}_3) \quad [12]$$

For 10 hydrothermal quartz-carbonate tailings Lapakko (1992b) reported that, relative to the standard ABA method, the NP determined by the modified ABA method was lower, and more closely approximated the mineralogic NP (Table 15.12). The combined quartz and carbonate mineral content of the tailings averaged about 50%, with feldspar, chlorite, and mica constituting an average of 42%. The partial dissolution of some or all of these silicate minerals during standard and modified ABA NP-test digestions resulted in test NP values that exceeded corresponding mineralogic NP values. Modified ABA NP values that were less than mineralogic NP values suggest incomplete dissolution of the calcium and magnesium carbonates during the 24-hour digestion period.

TABLE 15.12—Comparison of neutralization potentials of hydrothermal quartz-carbonate hosted gold tailings as determined by standard ABA, modified ABA, and calcium carbonate plus magnesium carbonate content.

Sample	Neutralization potential, kg/t CaCO ₃		
	ABA, standard	ABA, modified	CaCO ₃ + MgCO ₃
T1	230	200	207
T2	230	180	189
T3	195	130	163
T4	184	130	147
T5	98	92	65
T6	69	64	45
T7	270	220	229
T8	174	120	110
T9	18	16	14
T10	373	200	200

Lapakko (1994a) used a similar approach to evaluate standard ABA, modified ABA, and B.C. Research Initial neutralization potentials of ten mine waste samples of varying mineralogy. The standard ABA and modified ABA produced similar NP values on most samples. For four of the samples the standard ABA and modified ABA NP values were not significantly different from the mineralogic NP of four samples, and were within 10 kg/t CaCO₃ of the mineralogic NP of an additional three samples (Table 15.13). The major minerals in these seven samples were quartz, potassium feldspar, and mica. However, for the remaining three samples NP values from these two static tests exceeded the mineralogic NP by 13 to 47 kg/t CaCO₃. The mineralogic components contributing to the excessive static test values for these three samples were calcium feldspar (and perhaps pyroxene and olivine), clinopyroxene, and iron carbonates.

The B.C. Research Initial NP values were slightly higher than those for the standard and modified ABA, perhaps due to the smaller particle size used in the BCRI. Nonetheless, the BCRI NP

values for eight of the samples were within 10 kg/t CaCO₃ of the corresponding mineralogic NP. The BCRI NP values for the remaining two samples were 15 and 49 kg/t CaCO₃ higher than the corresponding mineralogic neutralization potentials. Both of these samples contained iron carbonates, which were identified as responsible for the excessively high BCRI NP values. The NP(pH6), similar to the BCRI but using an endpoint of pH 6.0, was proposed as an alternative for NP determination. This method was within 3 kg/t CaCO₃ of the mineralogic NP of all ten samples.

TABLE 15.13—Neutralization potential (kg/t CaCO₃) of mine waste samples from various rock types.

Sample	Standard ABA	Modified ABA	B.C. Research	NP(pH6)	Mineralogic NP
RK1	12	9.6	7.7	3.0	0 (0-2.9) ¹
RK2	35	33	11	2.8	1 (0-2.3)
RK3	15	14	25	3.3	5 (1.9-9.7)
RK4	28	28	33	28	32 (28-37)
TL1	27	27	30	24	19 (17-23)
TL2	18	20	25	16	16 (13-21)
TL3	46	61	82	30	19 (8.1-33)
TL4	3.8	2.9	15	3.8	5 (3.7-6.6)
TL5	7.5	3.2	20	15	12 (9.8-17)
TL6	99	72	58	55	46 (35-52)

¹Error bar in parenthesis.

To further illustrate the difference among static test neutralization potentials, the USBM subjected samples from four different rock-type groups to four different methods of NP determination (C-S analysis, standard ABA, modified ABA, and modified NP(pH6)). The four rock-type groups tested were latite porphyry, mudstone, carbonate, and gabbro. Major mineral constituents for each rock-type group and a range of corresponding weight-percent estimates for each mineral are listed in Table 15.14. Mineral weight-percent ranges for latite porphyry, mudstone, and carbonate were estimated using X-ray diffraction (XRD), while estimates for gabbro were made using point counts from thin sections. Table 15.15 shows the response of the four rock-type groups (as influenced by their respective mineralogies) to the four NP-determination methods.

General gangue mineralogy for the latite porphyry and mudstone samples are similar. More importantly, XRD-determined carbonate content for both rock types is low (3 to 5%, and 4%, respectively), and the carbonate mineralogy is dominated by iron-carbonate species such as ankerite, siderite, and ferroan dolomite/magnesite (Table 15.14). C-S analysis of the latite porphyry suggests that less than 1% (expressed as CaCO₃) is actually present, whereas C-S analysis results of 4 to 6% for the mudstone samples are in good agreement with the XRD estimate of about 4%.

Insufficient acid addition to the latite porphyry and mudstone samples was first thought to be a plausible explanation for underestimated NP by the standard and modified ABA methods. However, when ABA acid-leach solutions from latite porphyry and mudstone samples were titrated (to a pH 8.4 endpoint) at the end of their respective leaches, between 85 and 100% of the original acid volume had not reacted with the rock samples. It was concluded instead that the carbonate present was predominantly associated with iron and, for these solids, the standard and modified ABA methods did not greatly overestimate the calcium and

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TABLE 15.14—Range of weight-percent estimates for major mineral constituents comprising the four rock-type groups.

Major Mineral Constituents	Rock-Type Group			
	Latite porphyry	Mudstone	Carbonate	Gabbro
Iron sulfide	Py (6)	Py (3-24), Po (0-5)	ND	Po (0.6-3)
Base-metal sulfide	ND	Tetra (tr.)	ND	Cp (1.5-3)
Carbonate	Ankerite ¹ (3-5)	Siderite ² (1-2) Kutnohorite ³ (1-2) Dolomite, ferroan ⁴ (tr.) Magnesite, ferroan ⁵ (tr.) Ankerite ¹ (tr.)	Calcite (10-90) Dolomite (5-20) Ankerite ¹ (3-4)	ND
Silicate	Microcline (20) Albite (15) Quartz (50)	Microcline (5-10) Albite (5-10) Quartz (45-50)	Quartz	Labradorite (60-65) Pyrox/amphib (14-19) Olivine (5-7)

NOTE: values in parentheses represent percent of minerals present
 ND, Not detected
 Po, pyrrothite
 Py, pyrite
 Cp, chalcopyrite
 Tetra, tetrahedrite

¹Ca(Fe,Mg)(CO₃)₂
²FeCO₃
³Ca(Mn,Mg)(CO₃)₂
⁴Ca(Mg,Fe)(CO₃)₂
⁵(Mg,Fe)CO₃

magnesium carbonate content. That is, the iron carbonate present did not contribute NP at the low pH digestions in these tests. A second explanation is that calcium and magnesium carbonates were present and of low reactivity (due perhaps, to iron oxyhydroxide coatings on their surface) and did not dissolve in the ABA digestions. Preliminary USBM scanning-electron microscopy (SEM) studies of related mudstone samples documented presence of rhombohedral carbonate occurrences rimmed with higher-density iron-calcium-manganese carbonate (Chirban, unpub. laboratory notes, 1991).

TABLE 15.15—Neutralization potential (NP) of four different waste-rock groups by NP-determination method (NP expressed as kg/t CaCO₃).

Waste-rock group	Sample	NP-determination methods			
		C-S analysis	ABA, standard	ABA, modified	NP(pH6), modified ¹
Latite porphyry	1-B	5.8 ²	0.0	0.0	0.1
Mudstone	1-C	59 ²	4.4 ²	3.9 ²	1.9
	1-D	40 ²	6.9 ²	2.9 ²	2.3
Carbonate	3-A	919 ²	885	872 ²	737
	3-B	369 ²	263 ²	294 ²	105
	3-D	36	40 ²	59	10
Gabbro	MN-2	7.9	29	10	2.8
	MN-4	8.3	28	7.8	0.2
	MN-6	6.5	21	16	7.4

¹4-hour test duration
²Average of two tests

Carbonate sample NP-determination test results are in good agreement with respective sample mineralogy. According to C-S analysis, sample 3-A contains 90% calcium carbonate equivalent which has been identified by XRD as calcite. Standard and modified ABA NP values were 95% of the C-S NP value, and modified NP(pH6) was 80% of C-S NP. These results are consistent with calcite's rapid reaction rate and subsequent nearly-complete digestion when subjected to excess acid. Sample 3-B contains about 40% calcium carbonate equivalent which has been identified by XRD as dolomite. Standard and modified ABA NP values

for sample 3-B were 70 and 80% of the C-S value, respectively. The underestimated ABA NPs may be due to insufficient acid addition; minimal titrant was required during the back titration to reach a pH 8.4 endpoint because 80 to 90% of the acid was consumed during its neutralization by the sample. The modified NP(pH6) value was only 28% of C-S NP, and is believed to be a result of insufficient leach time (4 hours). Dolomite has a slower dissolution rate compared with that of calcite; when sample 3-B was allowed to leach for a 120-hour period, an NP of 250 or 68% of C-S NP was achieved, which was more than twice the NP obtained by the 4-hour leach (see Table 15.8). Sample 3-D contains only about 4% calcium carbonate equivalent which has been identified by XRD as ankerite. Standard and modified ABA NP values are 100+% of C-S NP. This is consistent with the amount of excess acid present at the completion of the respective leaches (about 50 to 80% of the original volume was neutralized during the back titration). However, modified NP(pH6) value was only 28% of C-S NP, but again this was believed to be a function of insufficient leach time (4 hours). When sample 3-D was leached for a 120-hour period, an NP(pH6) value of 32 or 89% of C-S NP resulted.

Gabbro sample mineralogy is dominated by calcium, magnesium, and iron-magnesium silicates, and contains less than 1% carbonate (identified by C-S analysis as calcium-carbonate equivalent). Standard ABA NP values for all three gabbro samples were three times larger than their respective C-S NPs, while modified ABA NP values were only slightly higher for two of three samples. Overestimation of gabbro-sample NP by the standard ABA method is most likely caused by dissolution of calcic plagioclase which is the dominant gangue mineral in each of the three samples (MN-2, 4, and 6). Standard ABA NP values obtained for the gabbro samples are consistent with observed dissolution of pure calcic plagioclase samples when subjected to the same test (see Table 15.10). Modified NP(pH6) results are one and two orders of magnitude lower for gabbro samples MN-2 and MN-4, compared with their corresponding standard ABA NPs. These results are consistent with the higher pH conditions of the modified NP(pH6) test (pH > 6.0), which would be less likely to solubilize much calcic plagioclase.

SUGGESTIONS FOR IMPROVING STATIC-TEST ACCURACY

The following suggestions are offered to help solve specific problems with NP-determination tests that were identified in the previous section.

- The method of determining acid production potential should be based on the sulfur-bearing minerals present. If only iron sulfide is present, the AP can be based on total sulfur determination. If other sulfur-bearing minerals are present in substantial quantities, it may be necessary to determine their abundance to accurately quantify the AP.
- Know the carbonate mineralogy and major host rock minerals present in samples being tested.
- If iron carbonates are present, care should be taken to allow for iron precipitation in the back titration of the standard and modified ABA tests. This precipitation can be enhanced by use of an endpoint of 8.3 rather than 7.0 and allowing additional time for endpoint pH stabilization. It can be essentially eliminated by the addition of hydrogen peroxide, as used for mineral acidity determination (ASTM method D-1067 or American Public Health Association et al., 1992).
- If no iron carbonates are present, measurement of carbon dioxide evolved from the sample will accurately quantify the calcium/magnesium carbonate content of the mine waste. Errors from commonly used static tests will be limited to those introduced by dissolution of noncarbonate host rock minerals during the NP digestion and the attendant low-pH acid neutralization.
- The presence of minerals such as calcium-rich plagioclase should be noted. The possible contribution of false NP by dissolution of these minerals in low-pH digestions must be considered. Only if the rate of acid production is quite slow, will these minerals neutralize acid while maintaining a neutral pH in the environment. Their practical neutralization potential is severely limited if they are present in waste rock.
- The NP(pH6) test will probably be more accurate than the standard ABA, modified ABA, and BCRI initial tests in quantifying the NP present as calcium carbonate and magnesium carbonate for samples containing elevated levels of siderite (iron carbonate) or calcic plagioclase (e.g., labradorite). This test, however, is often more time consuming than the more commonly used NP determinations.
- For waste rock samples, NP digestions should be conducted on larger size fractions in addition to the recommended size reduction, in order to better quantify the influence of particle size on the available NP. Testing minus 1/4-inch samples will further benefit interpretation of humidity cell data from waste rock samples.

SUMMARY

Static tests conducted on a variety of metal-mine-waste samples showed that neutralization potential (NP) variability for a given sample was (1) most strongly influenced by differences in sample particle size, amount of acid addition, back-titration endpoint, and sample mineralogy; (2) influenced in one test by digestion duration; and (3) virtually unaffected by acid type and temperature of digestion:

- Reducing particle size of three samples from minus 1/4 inch to

minus 325 mesh increased NP by 8- to 43-kg/t CaCO_3 (increases of 98% and 5%, respectively).

- Increasing acid strength by up to five times increased the NP of ten samples by 5 to 76 kg/t CaCO_3 (increases of 3 to 50%, respectively).
- Changing the back-titration endpoint from pH 7 to pH 8.3 decreased NP by 20 to 30 kg/t CaCO_3 (decreases of 11 to 15%, respectively).
- The influences of temperature (25 vs 85°C) and digestion duration (1 vs 24 hours) on standard and modified ABA NP values for 12 mine-waste samples of variable composition and for 10 hydrothermal-quartz-carbonate-hosted gold-tailings samples were negligible.
- When digestion duration of the modified NP(pH6) test was increased from 4 to 120 hours for 5 carbonate-rock samples, NP increased by 1.1 to 2.3 times.

The extent to which protocol variables affect NP is a function of sample mineralogy:

- Standard ABA digestions performed on specimen-grade sodic- and calcic-end member feldspars produced NP ranges of 0.0-0.4 and 12-24 kg/t CaCO_3 , respectively. Although carbonate was not present in the feldspar samples, resulting calcic feldspar dissolution by the acidic ABA digestion produced an NP that is equivalent to the presence of as much as 2.4% CaCO_3 .
- For mudstone and carbonate rocks, standard ABA, modified ABA, and modified NP(pH6) determinations yielded lower NP values than that indicated by the total carbonate content of the rock (carbonate content was determined by speciating sample total-carbon content into carbonate- and organic-carbon components). The difference was attributed to:
 - the presence of some iron or manganese carbonate and (or),
 - the inability of static-test digestions to dissolve calcium and magnesium carbonate which were coated with iron and manganese precipitates.
- For Duluth Complex rock the standard and modified ABA NP values were typically two to three times those estimated based on the carbonate content of the rock, apparently due to acid neutralization by calcic plagioclase dissolution in the static test digestions. In contrast, the modified NP(pH6) values were less than or equal to estimates based on the carbonate content of the rock, reflecting the less acidic (and more conservative) conditions of this digestion.

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