

# CHARACTERIZATION AND STATIC TESTING OF TEN GOLD MINE TAILINGS<sup>1</sup>

by

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**Abstract.** Ten tailings from operating North American gold mines were characterized (particle size distribution, chemistry, mineralogy) and subjected to two static tests. The tailings contained 0.1% to 7.6% sulfur, most of which was associated with pyrite and pyrrhotite. The total calcium and magnesium carbonate content of the samples ranged from 1.4% to 21%. The Acid Production Potential (APP) and Neutralization Potential (NP) determined by Modified Acid Base Accounting (Coastech 1989) were more accurate than the corresponding values determined by Acid Base Accounting (Sobek et al. 1978). The APP values were slightly more accurate since they accounted for small amounts of non-acid producing sulfate present in the samples. The Modified ABA NP values more closely approximated the total calcium carbonate and magnesium carbonate content of the tailings, due to lower acid addition during the digestion and a higher endpoint in the titration. The static test Net NP values (NP - APP) indicated that two of the samples were acid producers, and that one or two samples had marginal potential for acid production. Using Modified ABA values, five of the samples had NP:APP ratios less than 3:1, a range proposed in California for identification of acid producing wastes.

**Additional Key Words:** Acid mine drainage, prediction, static tests, tailings, gold mine waste, mine waste characterization.

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## Introduction

Exploration for gold and other non-ferrous minerals is presently occurring in Minnesota. If an economic deposit is discovered, the mine wastes must be characterized and the mine waste drainage quality must be projected prior to mine development. This information will be used to identify the types of water quality control required to protect the water resources of the state. Since there has been little mining of base or precious metals in Minnesota, there is little information available on the characteristics of and drainage

quality from such mining wastes. The lack of such information will inhibit the effectiveness and efficiency of drainage quality projection, as well as the environmental review and permitting processes.

## Objective

The objective of this project is to identify potential water quality impacts and aid interpretation of drainage quality prediction tests for non-ferrous mines proposed in Minnesota. This will allow development of non-ferrous mineral resources while minimizing adverse impacts on water resources in the state. The objective was pursued by determining the physical, chemical, and mineralogical characteristics, as well as the dissolution behavior of a variety of rock samples which simulate potential mine wastes generated by future non-ferrous mineral development in Minnesota. The relationship between the solid phase composition and drainage quality will be examined to identify potential water quality impacts

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which may be produced by non-ferrous mining in Minnesota. This paper presents the results of solid phase characterization and static tests.

### Mine Waste Dissolution

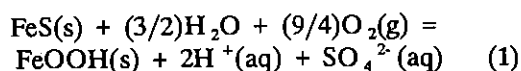
#### Introduction

Iron sulfide minerals, trace metal sulfide minerals, as well as calcium carbonate and magnesium carbonate minerals, play a dominant role in the release of acid and trace metals from mine wastes. The generation of acidic waters is the foremost concern for drainages from metallic mine wastes. In addition to high acidity, these drainages typically have elevated concentrations of the leachable trace metals present in the mine waste. Either condition can be toxic to aquatic organisms.

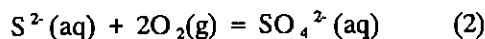
#### Acid Production

The stoichiometry of iron sulfide oxidation expresses the relative amounts of reactants required and products yielded in the oxidation of one mole of iron sulfide. Although there are numerous physical, chemical, and biological complexities to be considered, the basic reactions presented represent the fundamental process of acid generation as an oxidation of iron sulfide minerals to an assumed equilibrium state. Intermediate stages of the reaction are not considered, nor is the influence of other solid or dissolved components which may be present in a complex reaction environment.

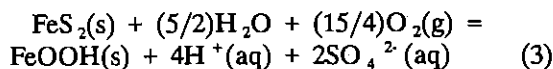
The dissolution of iron sulfide minerals leads to the majority of acid production by mine wastes. Although there are numerous iron sulfide minerals, the oxidation of a simple iron sulfide mineral is represented by reaction 1 (Nelson 1978). The overall reaction



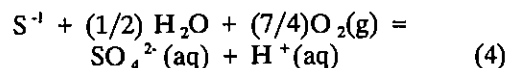
of FeS with water and oxygen releases two moles of acid and one mole of sulfate. The two moles of acid produced are the net result of the oxidation of ferrous iron and the subsequent precipitation of ferric iron, as lepidocrocite, in this example (Nelson 1978, Sung and Morgan 1980). The oxidation of sulfide to sulfate does not produce acid (reaction 2).



The most common iron sulfide associated with mine wastes is pyrite (FeS<sub>2</sub>). The stoichiometry of pyrite oxidation is presented in reaction 3 (Stumm and Morgan 1981). As in reaction 1, two moles of acid are produced by the oxidation and subsequent precipitation

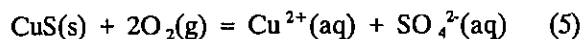


of iron. An additional two moles of acid are produced by the oxidation of the two moles of sulfur, which have an average oxidation state of -1, to sulfate (reaction 4).



#### Trace Metal Sulfide Oxidation

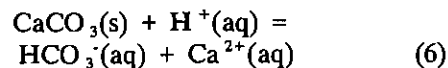
The oxidation of trace metal sulfide minerals releases trace metals but does not contribute acid (reaction 5). Concentrations of trace metals in solution tend to increase exponentially

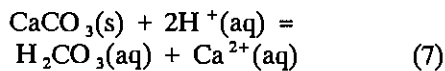


as solution pH decreases, therefore acidic drainages often contain elevated trace metal concentrations. However, circumneutral drainages can contain elevated concentrations of trace metals such as nickel (Eger and Lapakko 1985) and molybdenum (Brown 1989) which, compared to other trace metals, are relatively soluble in this pH range.

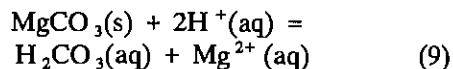
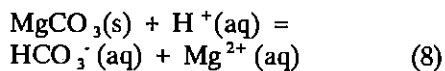
#### Acid Consumption

The balance between the rate of acid production by iron sulfide oxidation and the rate of acid consumption by host rock mineral dissolution, will determine the acidity of mine waste drainage. Drainage will remain neutral to basic as long as the rate of acid consumption equals or exceeds the rate of acid production. Calcium carbonate is the most effective mineral for neutralizing the acid produced by the oxidation of iron sulfides, and can be used to mitigate acid release. Reaction 6 is the dominant reaction above about pH 6.3, while reaction 7 is dominant at lower pH. Magnesium carbonate can also neutralize acid





(reactions 8, 9), but its rate of dissolution is reported to be slower than that of calcium carbonate (Rauch and White 1977). Similar to calcium carbonate dissolution, reaction 8 is dominant above pH 6.3 and reaction 9 dominates at lower pH. Iron carbonates will provide no net neutralization.



#### Available Reactive Surface Area

The aforementioned reactions of iron sulfide, calcium carbonate, and magnesium carbonate minerals are dependent on the mineral surface area available for reaction. The oxidation rate of individual metal sulfide minerals has been reported to be directly proportional to the reactive sulfide mineral surface area available (Sato 1960a 1960b, Sato and Mooney 1960, Nelson 1978, Lapakko 1980). Similarly, the rate of calcium and magnesium carbonate minerals is proportional to their respective available surface areas. The reactive mineral surface area present is a function of the mineral grain size, the roughness of the grain surface, and the mode of mineral occurrence within the rock matrix. As particle size decreases, the specific surface area (surface area per unit mass) increases. Thus, a kilogram of small mineral particles will react more rapidly than a kilogram of larger particles, since the small particles have a larger total surface area. Similarly, a "rough" particle will have a greater surface area than a smooth particle of similar diameter and mass.

The mode of occurrence of sulfide and carbonate minerals within the host rock will influence the availability of the mineral surface for reaction. Minerals which are liberated (free from the matrix of the rock and its other minerals) will have their entire surface area available for reaction, while dissolution of sulfides and carbonates included within other minerals will be inhibited. As particle size decreases the extent of mineral liberation increases. In larger rocks, the sulfides and carbonates are more likely to occur within the rock matrix, as inclusions within other minerals or

between other minerals (interstitially). Over time these minerals may be gradually liberated by physical and/or chemical weathering of the encapsulating rock or mineral. The reactivity of the mineral surfaces may be reduced due to chemical precipitation on the mineral surface.

#### Static Tests

The objective of static tests is to quantify the maximum capacity of a mine waste to produce acid and its maximum capacity to consume acid. Assumptions on the extent of mineral reaction and relative reaction rates are necessary for these predictions, since they are based only on solid phase analyses. These tests do not take much time, are fairly inexpensive and simple, and are useful in predicting acid mine drainage "when the difference between acid producing and neutralizing mineral content is wide" (Ferguson and Erickson 1987). They have been proposed as an initial screen for the acid drainage potential of mine wastes (Ferguson and Erickson 1987, Lawrence et al. 1989).

Accepting assumptions on the extent and relative rates of reaction as necessary, static tests can be evaluated on the accuracy with which they quantify the maximum potentials for acid production and acid consumption. To accurately determine the capacity for acid production (or consumption), it is necessary to measure those and only those components which lead to acid production (or acid neutralization). Thus, the capacity for acid production should be calculated based on sulfur associated with iron sulfide minerals (reaction 1) and sulfate associated with acid producing sulfate minerals such as jarosite (Dollhopf 1989) and melanterite. Sulfate minerals such as gypsum ( $\text{CaSO}_4$ ) will not generate acid. Similarly, the capacity for acid consumption should measure the content of calcium carbonate, magnesium carbonate, and other minerals which will buffer drainage pH in an environmentally acceptable range.

Acid-Base Accounting (ABA) was developed by Smith et al. (1974) and subsequently modified by Sobek et al. (1978) to evaluate the acid producing capacity of coal mine wastes. The test determines the Acid Production Potential (APP) based on the assumption that two moles of acid will be produced for each mole of sulfur present in the mine waste (reactions 1, 3). The total sulfur content in percent is multiplied by 31.25 to yield the APP in units of tons acidity as  $\text{CaCO}_3$  per 1000 tons rock (or

equivalently, kg CaCO<sub>3</sub>/metric ton). The Neutralization Potential (NP) is determined by adding hydrochloric acid (the volume and strength of which is determined by the "fizz" test) to the sample, boiling, and then titrating to pH 7.0 with sodium hydroxide to determine the amount of acid consumed by the rock. The resultant value is expressed as kg CaCO<sub>3</sub>/t. Whether or not a waste will produce acid is determined by the Net Neutralization Potential, which is the difference between these values (Net NP = NP - APP). Sobek et al. (1978) indicated that waste would produce acid if and only if Net NP were less than -5 kg CaCO<sub>3</sub>/t.

The APP calculation assumes that two moles of acid will be produced for every mole of sulfur present. If sulfur compounds which do not produce acid (including sulfate minerals such as gypsum, CaSO<sub>4</sub>, and barite, BaSO<sub>4</sub>) are present in significant quantities, the use of total sulfur will overestimate the acid production potential. The sulfur present with gypsum will contribute to the total sulfur, but will not produce acid. An example of this shortcoming was presented by Lawrence et al. (1989).

The method of NP determination (Sobek et al. 1978) may overestimate the actual neutralization potential available to maintain mine drainage pH above 6.0. The strong acid used in this method may dissolve minerals which would not consume acid under the conditions typical of environmental dissolution (Lutwick 1986). For example, the pH of drainage from Duluth Complex rock decreased below 6.0 after an NP depletion of only 0.5 kg CaCO<sub>3</sub>/t, considerably less than the measured NP of 11 kg CaCO<sub>3</sub>/t (Lapakko and Antonson 1991). Apparently silicate minerals present in the rock neutralized acid at low pH in the NP "digestion" but not during actual dissolution at pH 6.0 (Lapakko and Antonson 1991). Iron carbonates (e.g., siderite), which provide no net neutralization, may also contribute to the measured neutralization potential in the boiling HCl digestion. Furthermore, the strength of acid addition is based on the qualitative interpretation of the "fizz" test. Adding an excessive amount of acid based on this qualitative interpretation would further compound the overestimation of actual neutralization potential.

Sobek et al. (1978) indicated that waste would produce acid if and only if Net NP were less than -5 kg CaCO<sub>3</sub>/t. [This indicates that mine waste containing less than 0.16% S will not produce acid

(5/31.25 = 0.16).] While results presented by Lawrence et al. (1989) were consistent with this "critical Net NP" value, Day (1989) suggested that a Net NP value of +10 kg CaCO<sub>3</sub>/t be used as the threshold for acid production. Morin (1989) observed values of -15, +10, and +20 kg CaCO<sub>3</sub>/t for a modified procedure on various sets of samples. Ferguson (1989) indicated that it was difficult to determine the acid generating character of samples with Net NP values in the range of -20 to +20 kg CaCO<sub>3</sub>/t because of poor agreement among methods.

The Modified ABA method (Lawrence et al. 1989) bases the APP on the sulfide-sulfur content, that is APP = 31.25 x (percent sulfide-sulfur). The Neutralization Potential is determined using a 24-hour ambient temperature digestion (Coastech 1989). The pH is checked at least once after six hours of reaction, and a pH in the range of 1.5 to 2.0 must be attained for the test to be valid. If the pH is not in the range of 1.5 to 2.0, the acid addition is modified and the digestion is repeated until the target pH range is attained. The solution is then titrated with NaOH to pH 8.3 to determine the amount of acid consumed by the sample. As with the standard ABA method, the Net NP is calculated as the difference between the NP and APP. The mine waste is identified as an acid producer if and only if the Net NP is less than zero.

The use of sulfide-sulfur content instead of total sulfur content, corrects for the presence of sulfate minerals which do not produce acid. This procedure may underestimate the APP if acid-producing sulfate minerals (e.g., jarosite) are present. Another procedure considers the potential contribution of both sulfides and acid-producing sulfate minerals (Reclamation Research Unit, Schafer and Associates 1987), although the accuracy of this procedure has not been verified (Schafer 1992). The oxidation of trace metal sulfides also release sulfate, as well as the associated trace metal, but does not necessarily contribute acid (reaction 5).

Requiring an NP "digestion" pH in the range of 1.5 to 2.0 quantifies the acid addition required for the digestion more accurately than the subjective evaluation of the "fizz" test in the standard ABA method. This improves the reproducibility of the NP values obtained. It should be noted that digestion for 24 hours in the pH range of 1.5 to 2.0 may also attack host rock components which would not be effective in maintaining drainage pH in an

acceptable range. This method will also be more time consuming than that of the standard ABA if repeated acid additions are required to attain a digestion pH in the target range.

Conducting the acid digestion at a low temperature, as opposed to boiling, may reduce contributions of iron carbonate dissolution to the measured NP. Coastech (1989) examined a 24-hour digestion at room temperature. Data presented by Frisbee and Hossner (1989) suggest that iron carbonate dissolution would decrease at the lower temperature. If so, this digestion may more accurately determine neutralization potential for samples containing siderite ( $\text{FeCO}_3$ ) and possibly rhodochrosite ( $\text{MnCO}_3$ ). However, the presence of these minerals was not reported in the samples tested by Coastech (1989), and the NP determined by this technique was generally higher than that by the standard technique. The pH 8.3 endpoint is used for the NaOH titration since the pH 7.0 endpoint has been reported as unstable (Coastech 1989).

Both the standard ABA and Modified ABA identify acid producing wastes based on the difference between NP and APP. This calculation suggests that the acid consumption by one mole of calcium carbonate or magnesium carbonate equals the acid production associated with oxidation of one mole of sulfur present in iron sulfide. That is, both methods assume an acid consumption of two moles  $\text{H}^+$  per mole of carbonate mineral dissolved. Under the low pH conditions of the NP digestion, each mole of calcium carbonate or magnesium carbonate present will consume two moles of acid (reactions 7, 9). While these reactions most likely contribute to acid consumption, it is possible that the stoichiometry is not precise. Above approximately pH 6.3, it is likely that some fraction of the calcium carbonate and magnesium carbonate dissolution neutralizes only one mole of acid per mole of carbonate mineral dissolved (reactions 6, 8). Indeed, acid consumption in the neighborhood of 1 mole  $\text{H}^+$ /mole  $\text{CaCO}_3$  dissolved was reported for laboratory dissolution of metal mine waste (Lapakko and Antonson 1991). This study further indicated that not all of the calcium carbonate present was effective in maintaining drainage pH above 6.0.

The consumption of two moles of  $\text{H}^+$  per mole of carbonate mineral dissolved implies that a 1:1 ratio of NP:APP will produce a drainage which is

not acidic. However, coal mine overburden with an APP:NP ratio less than 2.4 produced drainage with a negative alkalinity (diPretorio and Rauch 1988). Other field results from coal mines (Brady et al. 1990) and consideration of the aforementioned carbonate equilibria (Cravotta III et al. 1990) suggest that an NP:APP ratio in the neighborhood of 2:1 may be near the critical point for acid generation. However, caution must be exercised when applying results from coal mine waste to the dissolution of metal mine wastes.

Drafts of California regulations proposed a NP:APP ratio of 3:1 as the acid production threshold value for the initial mine waste screen (Krull 1988). Although this value appears to be conservative, no published reference for its establishment was found. In particular, the ratio provides a more conservative threshold value if the APP is high. For example, using a threshold Net NP value of 5 kg  $\text{CaCO}_3$ /t seems relatively safe if the APP is 1 kg  $\text{CaCO}_3$ /t (i.e., APP = 1, NP = 6 kg  $\text{CaCO}_3$ /t). However, if the APP is 150 kg  $\text{CaCO}_3$ /t, the 5 kg  $\text{CaCO}_3$ /t difference between APP and NP provides a less comfortable safety factor.

## Methods

### Tailings Analysis

Tailings and feed samples were received from eight North American gold mines. The ten samples selected for this study were analyzed for particle size distribution in the Hibbing DNR Minerals office and by Hanna Research. Hanna Research used a multi-acid digestion with hydrochloric, hydrofluoric, and 50% sulfuric acids for analysis of Ca, Mg, Na, K, Fe, Mn, Al, and P. The solid/acid mixture was baked dry and redissolved with a solution of distilled deionized water and hydrochloric acid. For analysis of Cr, Ti, and V, samples were digested with a mixture of sulfuric and phosphoric acid. Metals were analyzed by ICP (ARL Model 3410).

Samples were analyzed by Bondar-Clegg for silicon, trace metals of regulatory interest (Ag, As, Ba, Be, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Zn), and a set of miscellaneous trace metals included in the ICP analytical package (Au, Bi, Ce, Co, Ga, La, Li, Nb, Rb, Sc, Sr, Ta, Te, V, W, Y, Zr). The analyses were conducted on a solution generated by digestion of the solids in a heated 3:1 mixture of hydrochloric

and nitric acid. Most metals were analyzed directly by ICP, with the following exceptions. Thallium was extracted with MIBK and analyzed by graphite furnace. Gold was analyzed using fire assay extraction with analysis by gravimetric finish and/or atomic absorption. Mercury was analyzed by graphite furnace atomic absorption. Silica was analyzed by peroxide fusion with gravimetric finish. Sulfur was analyzed by LECO induction furnace and sulfate was analyzed using a sodium carbonate leach. Carbon dioxide was analyzed using a Coolermetrics carbon dioxide analyzer.

Mineralogical analyses were conducted by Louis Mattson of the Hanna Research Center. X-ray diffraction (XRD) was used for mineral identification as well as for determination of the approximate modal composition. This analysis was applied to the bulk sample and to a heavy mineral concentrate. The heavy mineral concentrate was analyzed to more accurately identify the sulfides and other heavy minerals present. This was necessary since the sulfide content of the tailings was fairly low.

The chemical analyses were used in conjunction with XRD data to more accurately determine the mineral composition. The formula for dolomite used by Hanna was 30% CaO, 22% MgO, and 48% carbon dioxide, which represents equal molar amounts of calcium carbonate and magnesium carbonate. The corresponding contents for ankerite were 30%, 15%, and 45%, with the remaining 10% composed of iron oxide. The values for dolomite composition are theoretical while those for ankerite are "typical" for ankerite. The extent of sulfide and carbonate mineral liberation was determined by wet screening on 100, 270, and 500 mesh sieves, and using optical microscopy to examine the fractions separated.

#### Static Tests

The Acid-Base Accounting static test was conducted using the method of Sobek et al. (1978), with sulfur analysis by LECO furnace. For the Modified Acid Base Accounting test the APP was based on the sulfide-sulfur content (Coastech 1989). As with the ABA procedure, the "fizz" test was used to determine the acid addition. The mixture of acid and the 2.00 g, minus 60 mesh sample was digested for 24 hours at room temperature on a shaker table (25 to 30°C). The pH was checked at least once after six hours of reaction, and for the test to be valid, a pH in the range of 1.5 to 2.0 was required.

The solution was then titrated to pH 8.3 to determine the amount of acid consumed by the sample.

The Modified ABA Test requires that a pH in the range of 1.5 to 2.0 be attained following the acid addition for determination of Neutralization Potential (NP). Of the 10 tailings samples subjected to the test, only one of the samples met this requirement initially. The remaining samples were tested again using an increased or decreased acid addition. Although the target pH zone was attained in only one additional case, the pH values observed were adequate for reasonable extrapolation of the NP value for the target zone.

### Results and Discussion

#### Traditional Solid Phase Analysis

Analyses for particle size distribution, chemical and mineralogical composition, and the extent of mineral liberation were conducted on the ten tailings samples. The tailings samples were quite fine, with 47% to 97% of each sample occurring in the minus 270 mesh fraction and 26% to 91% in the minus 500 mesh fraction. Samples T5 and T6 were the coarsest, while sample T7 was the finest.

Sulfur content is an indicator of the potential presence of iron sulfide minerals and/or jarosite, the dissolution of which leads to acid production. The total sulfur contents of the samples ranged from 0.1% to 7.63% (table 1). With the exception of samples T2 (1% SO<sub>4</sub>), T9 (5.83% SO<sub>4</sub>), and T10 (0.57% SO<sub>4</sub>), the sulfate concentrations in the tailings were less than 0.2%, and typically less than 0.1%. Subtraction of the sulfate-sulfur from the total sulfur values yielded sulfide-sulfur values in the range of 0.08% to 7.3%.

The carbon dioxide concentrations in the samples ranged from 0.61% to 21.85%. These values indicate the presence of variable amounts of carbonate minerals. Trace metal analyses indicate elevated concentrations of arsenic in samples T6, T7, and T8. Sample T9 had the highest concentrations of molybdenum and antimony. Sample T2 had the highest zinc content.

The key parameters with respect to acid generation are the iron sulfide minerals. The iron sulfide content of the samples ranged from 0.6 percent for sample T7 to 13.6 percent for

Table 1. Sulfur, carbonate, and trace metal content of tailings.  
(Analysis by Bondar-Clegg.)

	Concentrations in PCT				Concentrations in PPM						
	S <sub>TOT</sub>	SO <sub>4</sub>	S <sup>2-</sup>	CO <sub>2</sub>	As	Cu	Mo	Ni	Pb	Sb	Zn
T1	0.51	0.06	0.49	9.86	38	26	<1	32	15	<5	339
T2	7.63	1.00	7.3	8.91	429	155	<1	125	122	<5	931
T3	1.03	.05	1.01	7.50	151	117	<1	118	22	<5	114
T4	1.15	.14	1.10	6.84	115	149	<1	110	19	<5	110
T5	0.67	0.04	0.66	2.94	47	39	2	27	55	<5	84
T6	2.12	0.16	2.07	7.54	1240	67	<1	18	21	<5	66
T7	0.10	0.05	0.08	10.23	1346	33	<1	126	14	18	60
T8	1.73	0.07	1.70	4.88	>2000	124	<1	140	106	41	85
T9	5.58	5.83	3.64	0.61	234	32	1130	19	26	288	152
T10	4.08	0.57	3.89	21.85	281	23	<1	26	28	<5	61

See Methods for complete list of parameters analyzed.

Table 2. Sulfide and carbonate mineralogy of tailings.  
(Analysis by Hanna Research Center.)

	Weight Percent Minerals									
	T1	T2	T3	T4	T5	T6	T7	T8	T9	T10
<u>Carbonates</u>										
Calcite	0.2	1.5	0.5	0.6	6.0	1.3	0.2	-	1.4	2.1
Dolomite	18.9	16.1	14.5	13.0	-	-	20.9	10.1	-	-
Ankerite	-	-	-	-	0.5	3.6	-	-	-	19.7
Siderite	1.9	1.3	0.9	1.1	0.2	14.0	0.2	0.1	-	31.4
<u>Sulfur-bearing minerals</u>										
Pyrite	0.86	13.58	1.82	1.99	1.09	1.29	0.04	2.43	6.57	7.32
Pyrrhotite	0.04	0.02	0.10	0.05	0.22	3.50	0.54	1.04	0.13	-
Barite <sup>1</sup>	-	-	-	-	-	-	-	-	14.22	-
Arsenopyrite	0.01	0.09	0.03	0.03	0.01	0.27	0.29	0.40	0.05	0.06
Chalcopyrite	0.01	0.04	0.03	0.04	0.01	0.02	0.01	0.04	0.01	0.01
Molybdenite	-	-	-	-	-	-	-	-	0.19	<0.01
Galena	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	-
Stibnite	-	-	-	-	-	-	<0.01	0.01	0.04	0.01
Sphalerite	0.05	0.14	0.02	0.02	0.01	0.01	0.01	0.01	0.02	-

<sup>1</sup> Barite was the only sulfate mineral detected. Sulfate in other samples is probably due to pyrite and/or pyrrhotite oxidation which often forms melanterite, FeSO<sub>4</sub>·7H<sub>2</sub>O.

sample T2 (table 2). Calcium carbonate and magnesium carbonate are the dominant minerals for acid neutralization. The major forms of these two minerals are calcite and dolomite [ $\text{CaMg}(\text{CO}_3)_2$ ], respectively, although both compounds are also present in ankerite. The typical calcite content of the samples ranged from 0.2 to 1.5 percent, with two samples beyond this range. The frequency distribution with respect to dolomite content was bimodal. Four of the samples contained virtually no dolomite while the remaining six contained 10 to 21 percent (table 2). Iron carbonate was present as siderite and/or ankerite in all samples except T9. Iron carbonate concentrations were particularly high in samples T6 and T10 (table 2). Since the tailings samples were very fine, the pyrite, pyrrhotite, and carbonate minerals were all well liberated. The major host rock components were quartz (16-43%), feldspar (1-37%), mica (4-24%), and chlorite (2-20%).

The release of trace metals from mine waste samples is largely a function of the trace metal content, the mineral form in which the metal is present, the chemistry of the individual metal, and the pH of the drainage. The trace elements of regulatory concern were present as sulfides (table 2). The arsenic in samples T6, T7, and T8 was present largely as arsenopyrite. The molybdenum and antimony in sample T9 occurred as molybdenite and stibnite, respectively. The zinc in sample T2 was present as sphalerite.

In summary, traditional analyses provide useful information on the dissolution of the tailings. Chemical analyses for sulfur and carbon dioxide provided an initial indication of the acid producing and acid consuming tendency of the tailings. The mineralogical analyses identified the sulfide minerals and carbonate minerals present. The presence of iron carbonates, which provide no neutralization, indicates that analysis for carbon dioxide alone will not provide an accurate quantification of neutralization potential. The presence of arsenic, antimony, molybdenum, and zinc as sulfides suggests that these may be readily released under oxidizing conditions. The fact that the sulfide and carbonate minerals are well liberated indicates that the mineral surfaces are available for acid producing and acid consuming reactions, as well as trace metal release.

### Static Tests

The tailings were subjected to the ABA and Modified ABA static tests. The static test results were evaluated by calculation of Net NP values and, for the Modified ABA, the NP:APP ratio. The determination of Acid Production Potential (APP) for the ABA method is based on the total sulfur content, while that for the Modified ABA technique is based on the sulfide-sulfur content. Since there was little difference between the total sulfur content and the sulfide-sulfur content of the samples (i.e., the samples contained little sulfate), the Modified ABA APP values were only slightly less than those determined by the ABA method. The sulfur contents reported for static test samples were different from those reported for the bulk chemical analyses (tables 3 and 1, respectively). The difference was slight except for the sulfate content of sample T9 and the total sulfur content of T10. The discrepancy in sulfate values was most likely due to the different sulfur speciation procedures of the two laboratories which analyzed the two sets of samples. The reason for the discrepancy on T10 is unclear and is under examination.

The NP values determined by the ABA method ranged from 18 kg  $\text{CaCO}_3/\text{t}$  for T9 to 373 kg  $\text{CaCO}_3/\text{t}$  for T10 (table 3). The NP determined by the ABA method was highly reproducible and tended to increase as the acid addition increased. Four of the samples were subjected to this test in duplicate. The difference from the mean for these pairs ranged from 0.65% to 7.6%, with an average value of 3.8%. Sample T8 was subjected to 40 mL acid additions of 0.1N and 0.5N HCl, with resultant NP values of 88 and 174 kg  $\text{CaCO}_3/\text{t}$ , respectively. This suggests that the NP value could vary by a factor of two, depending upon the laboratory technician's interpretation of the "fizz test."

The Modified ABA NP values ranged from 16 to 220 kg  $\text{CaCO}_3/\text{t}$ . The NP values determined by the Modified ABA digestion technique also tended to increase as the acid addition increased (table 4). Since this technique requires a pH in the range of 1.5 to 2.0 after the acid addition, more consistent readings would be expected. That is, the volume and concentration of the acid added are determined by the objective criterion of the target pH range rather than the subjective interpretation of the "fizz test."



Table 3. Summary of static test results.

Sample	S <sub>T</sub> <sup>1</sup>	S <sup>2</sup>	ABA			MODIFIED ABA			NP/APP <sup>7</sup>
			APP <sup>3</sup>	NP	NET NP <sup>4</sup>	APP <sup>5</sup>	NP	NET NP <sup>5</sup>	
T1	0.55	0.50	17	230 <sup>6</sup>	213	16	200	184	12
T2	8.19	7.87	256	230	* -26	246	180	* -66	* 0.73
T3	1.12	1.08	35	195 <sup>6</sup>	160	34	130	96	3.8
T4	1.23	1.18	38	184 <sup>6</sup>	146	37	130	93	3.5
T5	0.63	0.59	20	98 <sup>6</sup>	78	18	92	74	5.1
T6	2.18	2.10	68	69	M 1	66	64	M -2	* 0.97
T7	0.31	0.30	9.7	270	260	9.3	220	211	24
T8	1.86	1.79	58	174	116	56	120	64	* 2.1
T9	5.40	5.03	169	18	* -151	157	16	* -141	* 0.10
T10	6.51	6.30	203	373	170	197	200	M 3	* 1.0

<sup>1</sup> Total sulfur, percent. Analyzed by Lerch Brothers, Inc.

<sup>2</sup> Sulfide sulfur calculated as the difference between total sulfur and sulfate sulfur, percent.

<sup>3</sup> Acid Production Potential in kg CaCO<sub>3</sub>/metric ton = 31.25 x total sulfur

<sup>4</sup> Net Neutralization Potential in kg CaCO<sub>3</sub>/metric ton = NP-APP

<sup>5</sup> Acid Production Potential in kg CaCO<sub>3</sub>/metric ton = 31.25 x sulfide sulfur

<sup>6</sup> Average of duplicate values.

<sup>7</sup> Neutralization Potential to Acid Production Potential Ratio for Modified ABA Method

\* Indicates samples identified by the test as a potential acid producer.

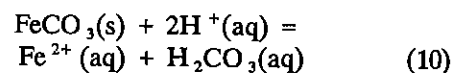
M Indicates moderate potential for acid production, with Net NP in the range of -20 to +20 kg CaCO<sub>3</sub>/metric ton.

The Modified ABA NP values were 2 to 173 kg CaCO<sub>3</sub>/t lower than the corresponding values for ABA NP (table 3). The difference between the two methods was typically in the range of 30 to 65 kg CaCO<sub>3</sub>/t. The lower values are due largely to the higher titration endpoint pH and a lower acid addition for the digestion.

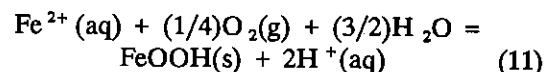
The Modified ABA procedure uses a titration pH endpoint of 8.3 rather than the 7.0 endpoint used by the ABA procedure. This higher endpoint will always yield a higher NP value. To demonstrate this point, samples digested by the ABA technique were titrated to endpoints of 7.0 and 8.3. Compared to the NP values at the pH 7.0 endpoint, those for the pH 8.3 endpoint were lower by 1 to 165 kg CaCO<sub>3</sub>/t, with most values lower by 20 to 30 kg CaCO<sub>3</sub>/t (table 5).

The major factor in the lower NP at the higher pH endpoint was apparently the precipitation of ferric iron released during the digestion. The difference was largest for sample T10, which also contained the most iron carbonate. During the ABA digestion the pH was in the neighborhood of

1.0, and some of the iron carbonate minerals (e.g. siderite, ankerite) present in the tailings dissolved while neutralizing acid (reaction 10). The H<sub>2</sub>CO<sub>3</sub> subsequently reacts to form water and release carbon dioxide gas to the atmosphere.



Under oxidizing conditions in the neutral pH range, the one mole of ferrous iron released will oxidize to ferric iron which will precipitate as ferric oxyhydroxide (reaction 11). The oxidation and precipitation reaction will yield two moles of acid. Thus, iron carbonate will not contribute to acid consumption.



Apparently the oxidation and precipitation of iron released from iron carbonates was incomplete at the pH 7.0 endpoint and, consequently, the NP values were excessive. At the pH 8.3 endpoint the extent of iron oxidation, which is the slower

Table 4. Effect of acid addition on NP determined by Modified ABA method.

	mL HCl	N HCl	Equiv. H <sup>+</sup>	pH <sub>o</sub> <sup>1</sup>	NP <sup>2</sup>
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	30	0.300	0.009	2.09	170
	40	0.500	0.020	0.64	200
T3	40	0.100	0.004	4.39	78
	20	0.527	0.010	1.20	146
	40	0.500	0.020	0.81	148
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
	60	0.105	0.006	1.65	92
	40	0.500	0.020	0.51	88
T6	40	0.100	0.004	2.14	41
	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
T8	40	0.100	0.004	4.59	69
	20	0.527	0.010	2.18	109
T9	20	0.105	0.002	1.85	22
	40	0.100	0.004	1.51	16
T10	35	0.527	0.018	1.22	204
	40	0.500	0.020	1.00	210

<sup>1</sup> pH<sub>o</sub>: pH at beginning of titration.

<sup>2</sup> pH 8.3 titration endpoint.

reaction, and subsequent precipitation is greater. This is likely due to the more rapid oxidation of ferrous iron at the higher pH value. This oxidation is reported to be second-order with respect to (OH<sup>-</sup>) (Sung and Morgan 1980, Eary and Schramke 1990) and would, therefore, be roughly 100 times faster at the higher titration pH.

Second, the Modified ABA NP values were lower since the acid addition was lower than that for the ABA digestion, except for samples T6, T8 (on the second titration), and T9. As discussed

Table 5. Comparison of ABA NP values determined at pH endpoints of 7.0 and 8.3, and Modified ABA NP. (Acid addition for ABA was 40 mL of 0.5 N HCl unless otherwise noted.)

	Initial ABA pH	ABA pH 7.0	NP pH 8.3	Mod ABA NP
T1	1.18	231	205	200
	1.14	228	208	
T2	1.17	202	173	180
T3	1.17	202	173	130
	1.21	188	163	
T4	1.15	190	162	130
	1.17	178	156	
T5	.98	106	98	92
	1.05	91	85	
T6 <sup>1</sup>	2.76	69	48	64
T7	1.35	270	241	220
T8	1.17	174	142	120
T8 <sup>1</sup>	4.87	80	74	
T9 <sup>1</sup>	1.62	18	17	16
T10	1.03	373	208	200

<sup>1</sup> Acid addition 40 mL of 0.1 N HCl.

above, the lower acid addition tends to yield lower NP values (table 4). The initial pH for the ABA titration of samples T6, T8 (at the lower acid addition), and T9 were 2.76, 4.87, and 1.62, respectively. Using values determined for the pH 8.3 endpoint (to eliminate differences due to the titration), the ABA NP values for these three samples were either roughly equal to or less than those for the Modified ABA values. For the remaining seven samples, the initial pH for the ABA titration was below the pH 1.5-2.0 range prescribed for the Modified ABA digestion. For these samples, the ABA NP (pH 8.3 endpoint) was lower than the Modified ABA NP on only one sample (T2), and this difference was slight.

Differences in the temperature and time of digestion (as opposed to magnitude of acid addition) only slightly influenced the extent of non-carbonate mineral dissolution. Each sample was subjected to both tests using the same acid addition (40-mL HCl of either 0.1 or 0.5 N HCl) and titrated to the pH 8.3 endpoint. Although the Modified ABA NP values were typically lower, the difference was slight. The ABA digestion is of shorter duration and, therefore, limits the time available for dissolution. However, the high temperature of this digestion may enhance the dissolution of some host rock components. For the samples examined, the influence of these variables was negligible.

Assessment of the accuracy of NP values is complicated by the fact that acid consumption is a function of solution pH. Calcium carbonate will dissolve and neutralize acid not only under acidic conditions, but also when solution pH is above 6.0. For other minerals this dissolution and neutralization will be significant only at lower pH values. Since the ABA and Modified ABA Tests subject mine waste samples to solution pH values ranging from less than 1 to 2, they measure contributions to NP which occur below pH 6.0. Whereas these reactions do neutralize acid, they may not maintain drainage pH in an environmentally acceptable range. Thus, some of the NP measured by these techniques may not be "effective" NP with respect to maintaining acceptable drainage pH.

For example, the NP (Sobek et al. 1978) of a Duluth Complex rock sample was measured as 11 kg CaCO<sub>3</sub>/t, and the sample was subjected to a laboratory dissolution test (Lapakko 1990). After the depletion of only 0.5 kg CaCO<sub>3</sub>/t the drainage pH decreased below 6.0. In this lower pH range, host rock minerals such as plagioclase, pyroxenes, and/or olivine dissolved and consumed acid, but the rate of dissolution was inadequate to maintain a drainage pH above 6.0.

To provide a meaningful value for prediction of acid drainage, tests must quantify the NP available to maintain a drainage pH of at least 6.0. In order to assess the accuracy of the static test NP values, the NP available to maintain a pH above 6.0 was calculated based on sample mineralogy. The mineralogical calculation assumed that the calcium carbonate and magnesium carbonate present in the mine waste samples, and only these minerals, would buffer pH to at least pH 6.0 (table 2). These

minerals will dissolve and neutralize acid most readily when in contact with a solution of pH above 6.0.

The ABA NP values were consistently higher than the NP available as calcium and magnesium carbonates (figure 1), while the Modified ABA NP values closely approximated these values (figure 2). As discussed above, the elevated ABA NP values were due to the lower endpoint pH and higher acid additions for the ABA method. The NP for sample T10 was particularly high. This sample had the highest iron carbonate content of the samples. The higher acid addition in the ABA digestion apparently attacked the iron carbonate to a greater extent than the lower acid addition in the Modified ABA digestion. Furthermore, at the pH 7.0 titration endpoint the oxidation of dissolved ferrous iron and the subsequent precipitation of ferric oxyhydroxide was incomplete. Thus, based on the calcium carbonate and magnesium carbonate content of the samples, the ABA NP overestimated the NP available to maintain pH above 6.0. The Modified ABA NP was in good agreement with the calcium/magnesium carbonate NP of the samples examined.

The Net Neutralization Potential is the difference between the Neutralization Potential and the Acid Production Potential (Net NP = NP - APP). As indicated in the review of the literature, there is presently no agreement on the critical value of Net NP, below which drainage will be acidic and above which drainage will be neutral or alkaline. For simplification of data presentation, a critical Net NP value of zero will be used. Thus, if Net NP exceeds zero it has an excess of NP, and implies that the waste will not produce acid. This value is not endorsed, but rather used as a tool for data presentation.

The ABA technique identified samples T9 and T2 as acid producers, based on Net NP values of -151 and -26 kg CaCO<sub>3</sub>/t, respectively. Sample T6 yielded a Net NP of 1 kg CaCO<sub>3</sub>/t, slightly above the boundary for acid production. The Net NP of the remaining samples, using the ABA method, ranged from 78 to 260 kg CaCO<sub>3</sub>/t (table 3).

The Modified ABA technique also identified samples T9 and T2 as acid producers, based on respective Net NP values of -141 and -66 kg CaCO<sub>3</sub>/t. The Net NP values for samples T6 and T10 were in -2 and +3 kg CaCO<sub>3</sub>/t, respectively,

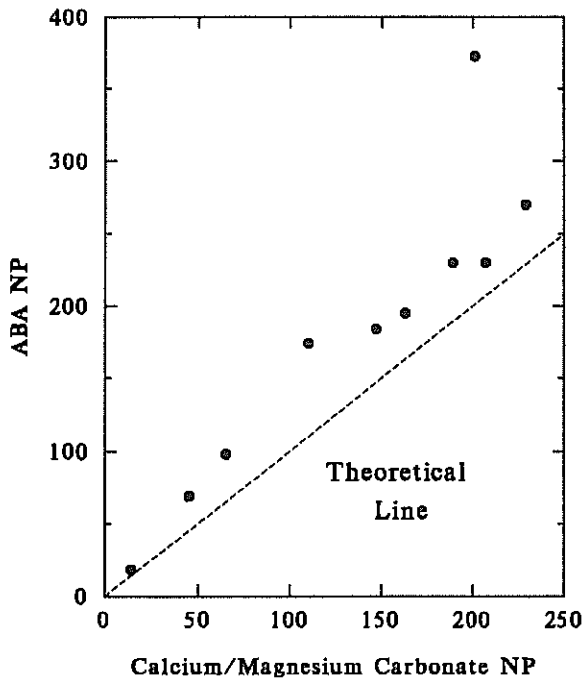


Figure 1. ABA NP v. NP based on calcium carbonate and magnesium carbonate content.

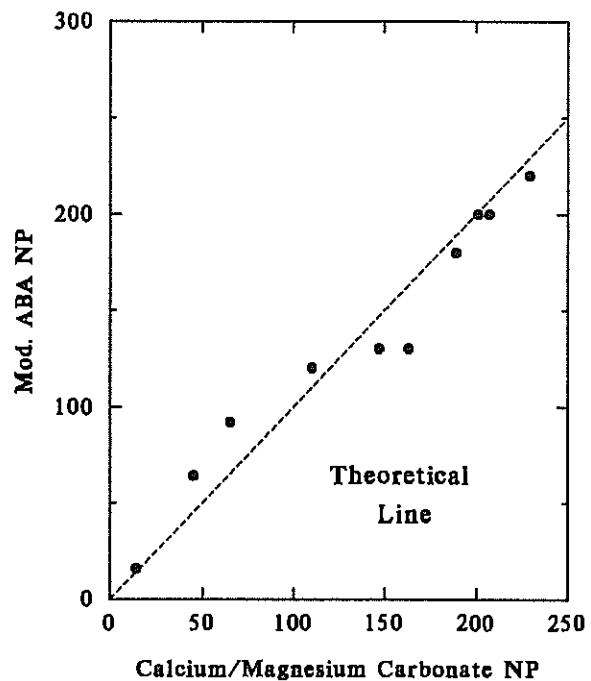


Figure 2. Modified ABA NP vs. NP based on calcium carbonate and magnesium carbonate content.

near the boundary between acid producing and non-acid producing. The Net NP of the remaining samples ranged from 64 to 211 kg CaCO<sub>3</sub>/t (table 3).

The NP:APP ratio is generally a more conservative quantifier for potential acid producers. The NP and APP from the Modified ABA technique were used to calculate this ratio. As with the previous classifications, this approach identified sample T9 as having the greatest potential to produce acid. This is indicated by the lowest NP:APP ratio of 0.10. In accord with the previous classifications, samples T2 and T6, respectively, exhibited the next highest potential for acid production. In addition, samples T8 and T10 also had NP:APP ratios less than three (table 3).

Thus, there was general, but not precise, agreement among the ABA Net NP, Modified ABA Net NP, and Modified ABA NP:APP ratio. All methods identified samples T9 and T2 as acid producers. Assuming all of the Neutralization Potential in sample T2 were available to neutralize

the acid produced by oxidation of the iron sulfides, a long time would be required before this sample would produce acid drainage. In contrast, sample T9 has one-tenth the NP and would acidify much sooner. With the exception of the ABA technique, samples T6 and T10 were identified as having some potential for acid production, while the potential for acid production by sample T8 was slight. The remaining seven samples (T1, T3, T4, T5, T7) exhibited very little potential for acid production.

### Summary

The generation of acidic drainage is the major regulatory concern associated with mine waste. Solid phase characteristics of a mine waste have a major influence on the quality of drainage generated. Oxidation of iron sulfides leads to production of acid, while dissolution of calcium carbonate and magnesium carbonate minerals present in a mine waste will neutralize acid. As long as the rate of acid neutralization exceeds the rate of acid production, the drainage pH will remain

neutral or alkaline. The rates of sulfide mineral oxidation (acid production), as well as calcium carbonate and magnesium carbonate mineral dissolution, increase with the mineral surface area available for reaction ("exposed" mineral surface).

Static tests, such as ABA (Sobek et al. 1978) and Modified ABA (Coastech 1989), are quick and inexpensive tests which measure the maximum potential of a mine waste to produce acid (APP) and neutralize acid (NP). These simplified tests do not measure the relative rates of acid production and acid consumption, nor do they quantify the extent to which the acid producing and acid consuming minerals will dissolve. In this study, ten gold mine tailings samples were subjected to the ABA and Modified ABA static tests to determine their potential for acid production. The accuracy of the APP and NP measurements was examined by comparing static test values with chemical and mineralogical characteristics of ten gold mine tailings. Characterization of particle size, mineralogy, and mineral liberation was used to qualitatively assess relative rates of acid production and acid consumption, as well as the extent of reaction. The potential for trace metal release was also assessed based on the chemistry and mineralogy of the tailings.

The results of the static tests indicated that from two to five of the ten samples would be classified as acid producing. The ABA method identified two samples as acid producers based on Net NP values of -151 and -26 kg CaCO<sub>3</sub>/t, and one sample as marginal (Net NP = 1 kg CaCO<sub>3</sub>/t). The Modified ABA was in agreement with the two acid producing samples and one marginal sample identified by the ABA method, although the Net NP values were slightly different. The Modified ABA identified one additional sample as marginal, based on a Net NP of 3 kg CaCO<sub>3</sub>/t. Using values determined by the Modified ABA method, four samples had NP:APP ratios less than or equal to one, and for a fifth sample this ratio was 2.1:1. These five samples would be identified as acid producers based on criteria used in California for mine waste screening.

The Modified ABA method was more accurate than the ABA method in quantifying both the APP and NP. The Modified ABA APP accounted for sulfur present in non-acid producing sulfate minerals such as barite and, therefore, was slightly lower and more accurately reflected the acid-

producing sulfide mineral content. The Modified ABA NP was fairly accurate in quantifying the neutralization potential available in calcium carbonate and magnesium carbonate minerals in the tailings. The ABA NP values exceeded this carbonate mineral NP due to excessive acid addition for the sample digestion and the lower titration endpoint. The excessive acid addition dissolved silicate and iron carbonate minerals present. The lower titration endpoint did not allow for the oxidation of ferrous iron (released from iron carbonate minerals, for example) and subsequent precipitation of iron oxyhydroxide.

The ABA NP was reproducible and the NP determined by either method increased with the strength of acid added for the digestion. For the Modified ABA NP, this point is academic since the volume and concentration of acid added are determined by a required pH range of 1.5-2.0 after acid addition. The degree to which the static test digestions attack the host rock components of other mine wastes, and consequently overestimate the available NP, will depend upon the mineral composition of the waste. These digestions may more readily dissolve non-carbonate host rock components in other samples, since the quartz (16-43%), feldspar (1-37%), mica (4-24%), and chlorite (2-20%) present in these samples are relatively unreactive even at low pH.

Chemical and mineralogical analyses indicated that most of the sulfur was present as pyrite and pyrrhotite, all samples contained calcium carbonate, and all but one contained magnesium carbonate and iron carbonate. These analyses further indicated that some samples contained elevated levels of arsenic, molybdenum, and antimony, which were present as sulfides. The degree of sulfide and carbonate mineral liberation ranged from 86 to 99%, indicating that the vast majority of the mineral surfaces present were available for reaction.

Since the sulfide minerals are liberated and present as small particles they will oxidize if exposed to water and oxygen, and the oxidation rate will be rapid relative to that for larger particles. Since the calcium and magnesium carbonates are similarly liberated and present as small particles, it is reasonable that they will initially neutralize the acid produced as a result of iron sulfide oxidation. The quality of drainage generated by the tailings over the long-term can not be predicted based only on static test results and other waste characteristics.

Predictions of long-term drainage quality require quantification of the relative rates of acid production and consumption, as well as the extent of mineral dissolution. Kinetic tests can be used to quantify the relative rates of iron sulfide and carbonate mineral dissolution (Lapakko 1990) and, if conducted for an extended period, can determine the extent of mineral dissolution (Lapakko and Antonson 1991). The extent of dissolution is important since, as dissolution progresses, the carbonate mineral surfaces (or sulfide mineral surfaces) may become coated with reaction products. Such coatings reduce the available neutralization (or acid production) below the level indicated by the maximum values determined by the solid phase characterization. Little field information on the relative rates acid production and acid consumption or the extent of dissolution is presently available for metal mine wastes.

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