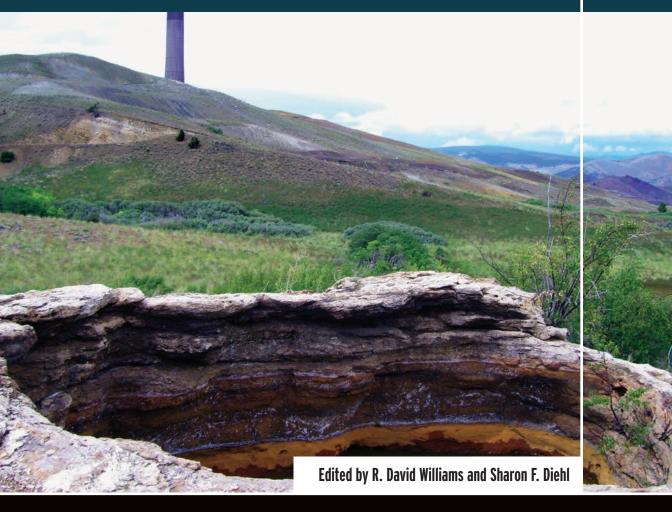
Management Technologies for Metal Mining Influenced Water

Techniques for Predicting Metal Mining Influenced Water

Volume 5





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Volume 5

Edited by R. David Williams and Sharon F. Diehl

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DISCLAIMER

This handbook is not intended to provide all of the information needed for all the possible variations for predicting metal mining influenced water that might be required at mining influenced sites. To the extent the document mentions or discusses statutory or regulatory authorities, it does so for informational purposes only. This handbook does not substitute for those statutes or regulations, and readers should consult the statutes or regulations themselves to learn what they require. The descriptions herein do not purport to address safety concerns, if any, and it is the responsibility of the reader to establish appropriate safety and health practices and to determine the applicability of regulatory limitations. Regulatory requirements may be more specific than the information included in this handbook. The use of firm, trade, and brand names or commercial products in this report is for identification purposes only and does not constitute endorsement or recommendation for use. The views expressed in this handbook are those of the authors and editors and do not necessarily represent the views of any federal agencies or the U.S. government.



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Preface

Techniques for Predicting Metal Mining Influenced Water is the fifth of six volumes in the Management Technologies for Metal Mining Influenced Water series of handbooks about technologies for management of metal mine and metallurgical process drainage. This is a technical handbook for an intended audience that includes planners, regulators, consultants, land managers, researchers, students, stakeholders, and anyone with an interest in mining influenced water. Other handbooks in the series are Basics of Metal Mining Influenced Water; Mitigation of Metal Mining Influenced Water; Mine Pit Lakes: Characteristics, Predictive Modeling, and Sustainability; Geochemical Modeling for Mine Site Characterization and Remediation; and Sampling and Monitoring for the Mine Life Cycle.

These handbooks are a volunteer project of the Acid Drainage Technology Initiative—Metal Mining Sector (ADTI-MMS). The Metal Mining Sector is part of the Acid Drainage Technology Initiative, which is the United States' regional member of the Global Alliance sponsored by the International Network for Acid Prevention. The mission of ADTI-MMS is to identify, evaluate, develop, and disseminate information about cost-effective and environmentally sound methods and technologies to manage mine wastes and related metallurgical materials for abandoned, inactive, active, and future mining and associated operations, and to promote understanding of these technologies.

Production of this volume was directed by the ADTI-MMS Prediction Committee, a technically focused consensus group of volunteer representatives from state and federal regulatory agencies and geological surveys, academia, the mining industry, consulting firms, and other interested parties who are involved in the environmentally sound management of metal mine wastes and drainage quality issues.

The chairman of the committee during the preparation of this document was R. David Williams (U.S. Department of the Interior, Bureau of Land Management [USDI, BLM]). Members of the committee at various times included Kim Lapakko (Minnesota Department of Natural Resources [MN DNR]), Charles Bucknam (Newmont Mining Corporation), Carol Russell (U.S. Environmental Protection Agency [USEPA]), Steve Wilson (U.S. Geological Survey [USGS]), Terry Chatwin (International Network for Acid Prevention), Sharon Diehl (USGS), Bill White (USDI, BLM, retired), Virginia McLemore (New Mexico Bureau of Geology and Mineral Resources), Steve Murphy (Shepard Miller), Dennis Turner (Arizona Department of Environmental Quality), and Christina Wilson (USEPA).

Information on the Sobek and modified Sobek methods; BC (British Columbia) research initial and confirmation tests; and Lapakko, siderite correction, and net acid generation tests (Appendixes A–E, G, and H) has been reprinted with the kind permission of Infomine. Information on the NCV test (Appendix F) has been reprinted with the kind permission of Charles Bucknam.

In addition, thanks are extended to Lisa Kirk and Linda Figueroa, who assisted in refining the various comments in Chapters 4 and 5; Ron Schmiermund, who presented an improved write-up on column tests; and Bob Kleinmann, who provided some detailed editorial assistance. The ADTI-MMS Steering Committee also provided guidance and encouragement in completing

this handbook. Without the input of these individuals as well as the leadership, support, encouragement, and tenacity of Charles Bucknam, Linda Figueroa, and Doug Peters, and also the dedication and previous work by Kim Lapakko and Bill White, this volume would never have been completed.

Many of the participants' companies, institutions, and agencies provided in-kind funding, namely, U.S. Department of Interior, Bureau of Land Management; U.S. Geological Survey (Mineral Resources Program); Newmont Metallurgical Services and Technologies; the Minnesota Department of Natural Resources; and U.S. Environmental Protection Agency (Region 8 and the Office of Research and Development). Most importantly, we thank each individual's agency or company for the experience, time, and other resources devoted to the preparation of this handbook.

CHAPTER 1

Introduction

Identification of potential mine wastes, their characterization, and prediction of their drainage quality are critical aspects of mine site design, operations, and closure planning. Failure to effectively conduct these evaluations for a mine site can result in long-term environmental compliance issues that may result in concomitant long-term financial liabilities (Maest and Kuipers 2005). Ore in stockpiles and exposed pit walls can exhibit different physical and chemical characteristics from waste rock and mineral processed materials; therefore, these ore-grade materials also require evaluation by similar methods at a mine or processing site. General references to mine wastes in this volume will often be applicable to ore in stockpiles and pit walls as well as to waste rock and mineral processing wastes.

Mine and process wastes that generate acidic and metal-rich drainages may require robust and costly treatment measures. Effective and accurate characterization and prediction work should result in a mine-life waste management plan for the site that minimizes, to the extent practicable, the exposure of problematic wastes to the environment. This will limit generation of drainage that may require long-term treatment in order to meet regulatory requirements and operating company standards for protection of human health and the environment. In contrast, wastes that generate environmentally benign drainage may require no remediation whatsoever.

The quality of drainage from mine and process wastes is a function of waste composition, which is mineralogically and lithologically variable within a mine site and over the course of the mine life cycle. Furthermore, drainage quality can be variable with time. Drainage that appears innocuous during operation or at mine closure may acidify in the future, and drainage from materials that contains acidity initially may also become neutral or alkaline with time. Some of the test protocols covered herein can help evaluate the potential for future changes in drainage quality. Volume 4 of this series, *Geochemical Modeling for Mine Site Characterization and Remediation*, goes into considerable detail on the linkage between predictive tests and the various modeling programs that attempt to make specific predictions about anticipated water quality based on site-specific conditions.

Therefore, to effectively and efficiently manage mine wastes, and any potential metal mining influenced waters, the geochemical performance of the processed ore and wastes must be accurately assessed so they can be effectively managed throughout the active mine life and beyond. This handbook goes beyond some of the previous works devoted to prediction and characterization, discussing and comparing some of the relative merits of the various tests as well as discussing criteria for evaluating tests.

OBJECTIVES

The objectives of this handbook are to identify categories of tools available for characterizing mine and processing wastes and predicting drainage quality, and to catalog some of the specific tools within these categories. The analytical focus of these tools can range from screening large areas of mine wastes to determining compositions of individual mineral grains. This handbook

does not provide a complete catalog of tools nor does it describe those tools presented in detail. This volume explains some of the strengths and weaknesses of the various predictive techniques so that the user can better evaluate which methods might be most appropriate for a particular requirement.

Typically, there is no single best route to characterize and classify waste at a mine or processing site. Each of the various testing protocols has its own strengths and weaknesses, and it can be a continual challenge at a mine site to develop a sequential process using the analytical tools available that can adapt as the mine proceeds from exploration through closure. In some cases, the combination of tests that best characterize and classify mine waste may change during the life of a mine as the lithology, mineralogy, mining or processing techniques change through the life of the mine.

A reference list is provided in each chapter to identify more detailed information on topics of interest.

A brief summary of sulfide mineral geochemistry is provided in Chapter 2 to aid in understanding some of the complexities in mine-waste prediction and characterization. Chapters 3, 4, and 5 discuss the general background for predictive tests, commonly and less commonly used tests, and evaluation, respectively. Chapter 6 discusses the application of various tests.

Additional information on a variety of aspects related to acid rock drainage (ARD) is also available in the first three volumes of this series.

ACID DRAINAGE TECHNOLOGY INITIATIVE-METAL MINING SECTOR

The Acid Drainage Technology Initiative (ADTI) is the United States' regional member of the Global Alliance sponsored by the International Network for Acid Prevention (INAP) (McLemore 2008). The Metal Mining Sector (MMS) is part of the ADTI. The mission of ADTI-MMS is to identify, evaluate, develop, and disseminate information about cost-effective, environmentally sound methods and technologies to manage metal mine wastes and related metallurgical materials for abandoned, inactive, active, and future mining and associated operations, and to promote understanding of these technologies. ADTI-MMS is not a policy or regulatory initiative; it is a technically focused consensus group of volunteer representatives from state and federal government, academia, the mining industry, consulting firms, and other interested parties who are involved in the environmentally sound management of metal-mine wastes and drainage quality issues. More specifically, the goals of the ADTI-MMS include

- · Finding ways to reduce the extent and severity of adverse impacts of drainage from mine wastes and mining-related materials;
- · Developing consensus on environmental mine-waste management technologies for sampling and monitoring, drainage quality prediction, mitigation, and modeling;
- · Identifying technological needs and working to address these needs; and
- · Using sound scientific and technological fundamentals to interpret, assess, and summarize published literature and operational practices, and then communicating these findings to those responsible for environmental mine-waste management.

Overall direction for the ADTI-MMS is provided by the Steering Committee, which has representatives on the Operations Committee of ADTI. The Operations Committee also includes members from the Coal Mining Sector (ADTI-CMS). ADTI-MMS also includes several members from universities and schools with active environmental and mining programs.

INTRODUCTION 3

ACID ROCK DRAINAGE PREDICTIVE APPROACH

This volume is intended to give the reader an advanced overview of available prediction and characterization methodologies. Before digging into the toolbox, some general ideas on an overall approach to predicting ARD will be discussed. ARD is a natural environmental process that has been part of the normal geologic cycle of weathering and erosion of mineralized regions throughout geologic time. In fact, some of the historical geologic indicators of ARD—gossans, iron caps, ferricrete, iron staining, and impacted creeks—have been used by prospectors and miners for centuries as indicators of metal occurrences. Normal buffering processes in natural systems are often able to limit the impacts of natural ARD.

The process of mining and processing ores can accelerate the natural weathering process by comminuting metals and rock fragments and liberating minerals, thereby increasing the surface area of particles at rates that far exceed any natural processes. This accelerated availability of these reactive geologic materials to the weathering environment can have impacts on lands and receiving waters that can persist for decades or much longer. The prediction approach envisioned in this volume is that by understanding this accelerated weathering process and applying a detailed characterization and prediction approach throughout the mine life cycle, adverse environmental impacts can be limited to the maximum extent practicable. By recognizing problematic units through a detailed prediction program, these units can be managed to limit environmental impacts.

A synergistic effect of this process can be to reduce the potential long-term environmental liabilities and costs that can be incurred if acid-producing waste is managed inappropriately.

RELEVANCE OF ARD SOURCE

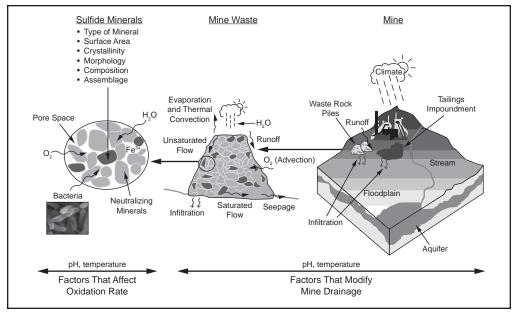
Although having an understanding of the weathering of sulfide minerals is important, comprehension of the geology, mineralogy, hydrology, and environmental setting of a potential economic mineral deposit can be equally significant. The geology of a site controls what rock types and structures are present, and it plays an important role in the environmental site setting, as well as in the surface and groundwater hydrology of a site, which are critical factors in designing waste facilities to minimize environmental impacts. Without a detailed evaluation of these factors, it is difficult to accurately interpret the results of predictive tests. Failure to accurately interpret predictive tests can result in waste management mistakes that may be difficult and expensive to remediate or can produce improper design of necessary containment facilities. Figure 1.1 illustrates the range of factors that relate to the possibility of acid drainage and potential impacts to the receiving environment.

This handbook focuses on the sulfide minerals and mine-waste components and the tools that are available for predicting whether a specific sample is likely to produce acid or not. Other factors include the environmental setting, geology, mineralogy, hydrologic considerations, mine life cycle mining and processing considerations, and climate. These are relevant to a risk-based analysis of whether or not the sulfide minerals and mine waste being evaluated represent a risk to the receiving environment. The basis for modeling of potential environmental impacts often depends on the detailed prediction work that this volume will discuss.

ENVIRONMENTAL SETTING

The environmental setting of a site is a detailed evaluation of the general environmental site conditions, which can include the following:

Depth and quality of soil



Source: INAP 2009.

FIGURE 1.1 Range of factors related to acid drainage possibilities

- · Type, diversity, and density of vegetation
- · Types and diversity of wildlife
- · Elevation and topography
- Aspect and existing disturbance or impacts
- · Climate

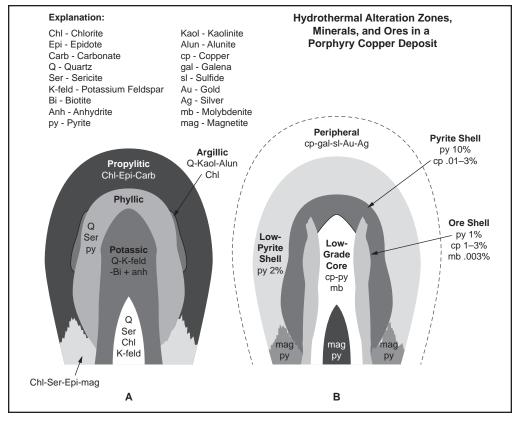
GEOLOGY

The geologic setting of a site includes a detailed evaluation of the rock types and structures present. Structures include deformation structures such as fractures, faults, and veining as well as sedimentary structures. This information should be noted in three dimensions. Geologic factors include detailed lithology and mineralogy (see the "Mineralogy" section that follows and Chapter 3), the nature and extent of weathering and alteration halos, the composition, extent and nature of veining, major structures, structural complexity, and geohydrology. Much of this information can be accumulated during the exploration phase of a project at little additional cost, compared to the considerable expense of going back and collecting it later should unanticipated ARD occur. See also the "Mine Life Cycle, and Mining and Processing Considerations" section later in this chapter.

MINERALOGY

A detailed evaluation of the mineralogy of all rock units to be involved in a project is essential to any comprehensive assessment of predictive tests. A detailed evaluation should include a description and analysis of all primary and secondary minerals; their grain size and distribution within the rock units; potential changes in mineral composition due to weathering or processing; and

INTRODUCTION 5



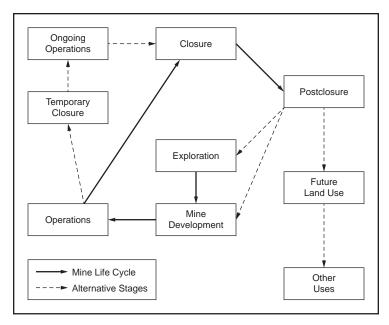
Source: Lowell and Guilbert 1970, courtesy of the Society of Economic Geologists.

FIGURE 1.2 Classic Lowell and Guilbert work on hydrothermal alteration zones

mineral variability in size, distribution, and occurrence laterally or vertically. Special attention should be made to the type, composition, and distribution of sulfide minerals, as these minerals are critical to any evaluation of predictive tests. The presence of carbonate minerals that can act to neutralize acid production is also critical information. This data should include a detailed description of the carbonate minerals, the presence of other potential acid-neutralizing silicate minerals and compositional variations in the minerals that may affect the ability to neutralize any produced acid. Figure 1.2 displays the classic Lowell and Guilbert (1970) work on hydrothermal alteration zones in a theoretical porphyry copper deposit. This figure highlights the importance of accurate three-dimensional geological and mineralogical work and also shows why this effort must continue throughout the project. As a mine expands and deepens, the ore and the waste mineralogies will change. Sulfide mineral percentages will also change as will the presence or absence of acid-neutralizing minerals. This information must be represented in three dimensions. Detailed methodology for evaluating minerals is discussed in Chapter 3.

HYDROLOGIC CONSIDERATIONS

Surface water, groundwater, and geologic structures at the project area should be evaluated to accurately determine the flow paths, as well as the water volumes and water quality, upstream and downstream of the project area by watershed. In the event the project area has preexisting



Source: McLemore 2008.

FIGURE 1.3 Mine life cycle stages

mine-impacted water present, a detailed evaluation should be made of the contaminants and their sources and flow paths. If the potential exists to degrade high-quality surface water or groundwater, it may be necessary to conduct a detailed survey to determine hydraulic parameters of all flow path materials and potential boundary conditions in order to develop an accurate conceptual model that can predict any anticipated impacts to groundwater and surface waters. Information on these parameters can be routinely recovered during the exploration phase of a project at considerably less expense than as a separate inventory later in the process.

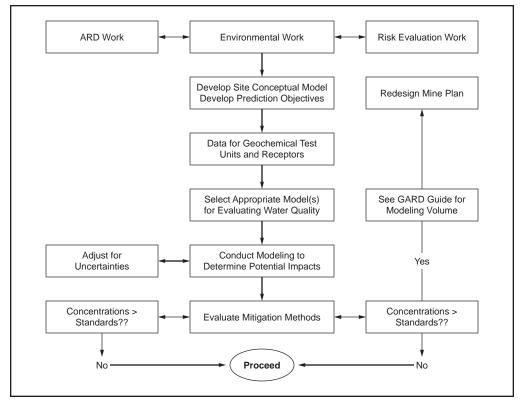
MINE LIFE CYCLE, AND MINING AND PROCESSING CONSIDERATIONS

Mines in general follow a relatively predictable life cycle, as shown in Figure 1.3. The collection of data that will be useful in acid drainage and rock characterization work should begin at the exploration phase. In some cases, field-scale observation such as extensive zones of weathering/oxidation or iron staining in stream channels can be an early tip-off that acidic drainage is likely to be a problem. Much of this information is routinely collected during the exploration phase, but in the past, it has repeatedly not been linked with detailed rock characterization work. This has often led to expensive secondary studies to reevaluate geologic material and information collected during the exploration and early development phases of a project.

Information that is routinely collected by drillers, project geologists, engineers, and metallurgists that can prove very useful includes the following:

· For drilling: drilling difficulty, rock competence, zones of poor or no recovery, quantity and quality of water

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Source: Adapted from INAP 2009; Maest and Kuipers 2005.

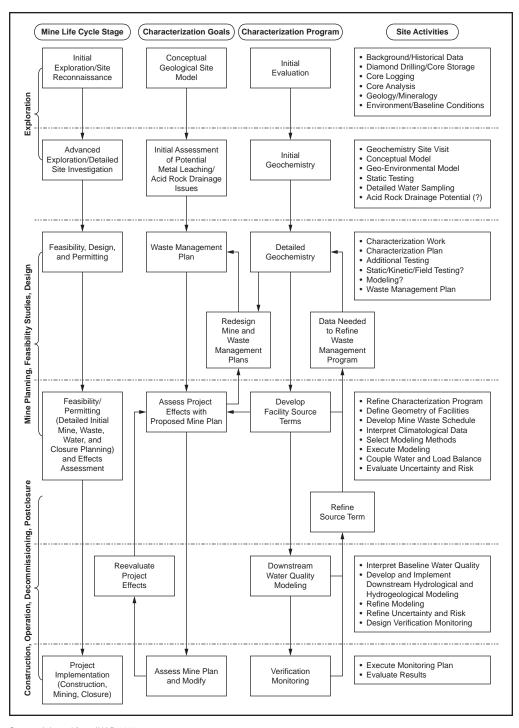
FIGURE 1.4 Steps for predictive modeling

- · For geology: detailed rock characterization of mineralogy and the presence of sulfides, carbonates, and other minerals such as sulfates, jarosites, barite, siderite, or undefined iron hydroxides, and trace elements
- · For hydrology: depth to water table; quality and quantity of water and structural zones that may control flow paths

Comprehensive core logging is necessary for quantitative information on the extent of weathering, mineralogy, structural complexity, rock hardness, and geology at a more detailed scale. The earlier that detailed information can be transferred to corporate environmental staff or consultants, the less likely it will need to be recreated at considerable expense later in the mine life cycle. Figures 1.4 and 1.5 show appropriate acid drainage program activities relative to the mine life cycle. A general representation of the timing and location of proposed geologic units to be mined is also useful. A detailed understanding of this information is often critical to understanding mineralogy, surface water and groundwater chemistry, surface water and groundwater flow volumes and paths, as displayed in Figures 1.4 and 1.5.

CLIMATE

Climatic factors can play a significant role in ARD, in terms of temperature and the type and timing of anticipated precipitation. Higher precipitation generally means that if acid drainage occurs, there is more likelihood of mining influenced water needing collection and treatment



Source: Adapted from INAP 2009.

FIGURE 1.5 Acid drainage program activities relative to the mine life cycle

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before release to the environment. Less precipitation can mean that there is commensurately less water available to transport contaminants off-site and thus less water that might need treatment. In general, it is unlikely there will be insufficient moisture or microbial activity to eliminate the oxidation reaction, as it takes very little moisture for the weathering reaction to proceed on the surfaces of minerals (Ritchie 1994). Cold temperatures can play an important role in limiting reaction rates, microbial activity, and transport of metals or contaminants. Water that is frozen of course will limit transport. The oxidation reaction is an exothermic response, so cold temperatures will not necessarily limit the oxidation reaction (Elberling et al. 1999). Conversely, warm temperatures tend to enhance the reaction process by generally providing a more favorable environment for bacteria that tend to catalyze the oxidation reaction.

The depth of weathering by oxidation within a mine-waste pile is mainly dependent on the depth of the water table. In general, water tables are high in humid climates and will be deep in semiarid to arid climates. Because of a high precipitation rate, erosion and leaching of metals from mine-waste piles in a humid climate can be more extreme in the short term than erosion of mine waste in an arid environment. However, oxygenated surface waters can infiltrate to a greater depth in a waste pile in a dry climate, which may result in a greater thickness of highly altered rock.

Climate changes that result in a local increase or decrease in precipitation will affect flushing of metals from mine-waste piles into receiving streams. Decreasing precipitation may affect the ability of streams to dilute metal concentrations. Increasing precipitation may result in episodes of rapid runoff and erosion, which increases sediment and metal loads in streams. Changes in precipitation will affect streamflow behavior, such as an earlier spring runoff and lower flow rates in the late summer and early fall.

Warmer average temperatures may affect internal conditions in mine-waste piles, such as increasing the potential for capillary rise of metal-bearing waters, which leads to the precipitation of evaporative efflorescent metal-bearing salts on the surfaces of waste material (Palumbo-Roe et al. 2007). Evaporative secondary minerals at the surfaces of mining wastes are highly soluble and dissolve with every storm event, flushing metals into drainages (Diehl et al. 2007).

Tailings storage facilities, which hold fine-grained milled material commonly suspended in water for transport to the tailings facility from the mill, may dry and be more susceptible to wind erosion in a dryer, warmer climate. The fine particle size, moisture retention between grains in impoundments above the water table, and evaporation can be controlling factors for the rate of oxygen diffusion and consequent sulfide oxidation. Wind-blown, metal-bearing dust is a potential human health hazard in active, closed, and abandoned waste sites in the arid southwestern United States if improperly managed (Hayes et al. 2009).

Static and kinetic tests are typically run at predetermined temperatures and water volumes (if applicable) as specified in the test procedures, and thus in general do not provide information on local climatic effects of waste sites. It may be necessary to extrapolate climatic effects based on site-specific measurements, test results, modeling, and professional judgment. It can be useful to have ongoing site-specific column tests or test piles on-site that can be directly correlated to static or kinetic tests.

Geochemical modeling must be adjusted for the site-specific temperature, precipitation, and evaporation. Climatic effects can be estimated based on meteorological and hydrologic records, but semiquantitative to quantitative predictions can be very difficult, particularly in the current context of a changing climate (Williams 2012).

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CHAPTER 2

Acid Rock Drainage: Sulfide Mineral Geochemistry Affecting Drainage

This chapter, taken largely from Lapakko et al. (2004), focuses on mineral dissolution reactions that influence mine-waste drainage quality. The reactions presented result in acid production, acid neutralization, and trace metal release. Within the chapter, soluble and insoluble ferrous and ferric solid phases of interest in mine and process waste characterization are identified. Detailed discussion of aqueous geochemistry fundamentals and geochemistry of acid mine drainage is presented by Nordstrom (1999), Nordstrom and Alpers (1999), Smith (1997), and Smith and Huyck (1999).

ACID GENERATION

Three general types of acid release reactions from mine wastes are iron sulfide oxidation, dissolution of soluble iron sulfate minerals, and the dissolution of less soluble sulfate minerals of the alunite/jarosite series. The oxidation of iron sulfide minerals such as pyrite (FeS₂), marcasite (FeS₂), and pyrrhotite (Fe_{1-x}S, 0.7 < x < 1.0) is responsible for the majority of acid production by mine wastes (Stumm and Morgan 1981). Equations 2.1 and 2.2 represent pyrite and pyrrhotite oxidation by oxygen (Stumm and Morgan 1981; Nelson 1978). In addition to acid (H⁺), sulfate (SO₄²⁻) is also released to solution, and the presence of sulfate or acid in mine-waste drainage is typically the first indicator of sulfide mineral oxidation. The iron product presented is ferric hydroxide, although formation of intermediate aqueous ferric hydroxyl species (e.g., Fe(OH)₂⁺(aq), Fe(OH)₃(aq)) will also result in acid production.

$$FeS_2(s) + (15/4)O_2(aq) + (7/2)H_2O = Fe(OH)_3(s) + 2SO_4^{2-}(aq) + 4H^+(aq)$$
 (EQ 2.1)

$$Fe_{1-x}S(s) + [(9-3x)/4]O_2(aq) + [(5-3x)/2]H_2O$$

$$= (1-x)Fe(OH)_3(s) + SO_4^{2-}(aq) + 2H^+(aq)$$
(EQ 2.2)

Ferric iron (Fe³⁺) can also oxidize iron sulfide minerals (Reactions 2.3 and 2.4) (Lapakko et al. 2006). The amount of acid generated as a result of the iron sulfide (ferric) oxidation is the same as that for oxidation by oxygen. The additional acid generated by ferric oxidation is due to precipitation of the aqueous ferric iron (left side of Equation 2.4) as ferric hydroxide (right side of Equation 2.4). This reaction is important to note because the ability of the ferric oxidation reaction to proceed in the absence of oxygen is pH dependent, which has important implications for potential mitigation strategies, as discussed in *Mitigation of Metal Mining Influenced Water* (Gusek and Figueroa 2009). At a pH of less than 3, ferric hydroxide will dissociate into ferric iron and water; at pH greater than 3, ferric iron and water combine to form ferric hydroxide and generate acid (Equation 2.4).

$$FeS_{2}(s) + 14Fe^{3+}(aq) + (91/2)H_{2}O + 15/4O_{2}(aq)$$

$$= 15Fe(OH)_{3}(s) + 2SO_{4}^{2-}(aq) + 46H^{+}(aq)$$
(EQ 2.3)

$$Fe^{3+}(aq) + 3H_2O = Fe(OH)_3(s) + 3H^+(aq)$$
 (EQ 2.4)

The rate of iron sulfide oxidation and attendant acid production is dependent on solid-phase compositional variables and microbial activity, as well as the availability of oxygen and water. Additional discussion of oxidation by pyrite with dissolved oxygen and ferric iron can be found in Chapter 12 of *Mine Pit Lakes: Characteristics, Predictive Modeling, and Sustainability* (Castendyk and Eary 2009).

Oxidation rates vary among iron sulfide minerals, and it is often reported that reactivity decreases in the order marcasite > pyrrhotite > pyrite (e.g., Kwong and Ferguson 1990). However, different reactivity rankings have been reported by other authors and may be a function of reaction conditions, trace element concentrations of the minerals, and crystal-morphology characteristics, among other factors (Jambor 1994; Plumlee 1999). For a given sulfide mineral, the oxidation rate increases with the available surface area. For example, the oxidation of framboidal pyrite, with a high associated surface area, is reported to be much more rapid than that of euhedral pyrite (Pugh et al. 1984; White and Jeffers 1994).

Whereas the abiotic rate of pyrite oxidation by oxygen decreases slightly as pH decreases, the overall abiotic rate increases as pH decreases into a range where ferric iron becomes the dominant oxidant (Williamson and Rimstidt 1994). Nordstrom (1982) reported that as "pH decreases to 4.5, ferric iron becomes more soluble and begins to act as an oxidizing agent." As pH further decreases, bacterial oxidation of ferrous iron becomes the rate-limiting step in the oxidation of pyrite by ferric iron (Singer and Stumm 1970), which is the only significant oxidizing agent in this pH range (Nordstrom 1982; Singer and Stumm 1970; Kleinmann et al. 1981). In laboratory tests, the pyrrhotite oxidation rate in the pH range of 3.5 to 4.05 was roughly six to seven times that in the range of 5.35 to 6.1, and the higher rate was attributed to bacterially mediated oxidation (Lapakko and Antonson 1994). Data presented by Nordstrom and Alpers (1999) suggest that the bacterially mediated rate of pyrite oxidation by ferric iron is roughly 2 to 3 orders of magnitude faster than the abiotic oxidation by oxygen at pH 2.

These weathering reactions produce acidic, and iron- and sulfate-rich waters that can react with metal sulfide minerals and accelerate their oxidation, evaporate partially or totally to precipitate hydrated iron-sulfates and other minerals, and/or contact host rock minerals, which react to neutralize some or all of the acid. Acidic flow, which is not neutralized within the mine waste, will exit as acid rock drainage (ARD). Drainage that is neutralized by this process can exit as neutral mine drainage and may still contain elevated levels of sulfate and metals such as cadmium or zinc that require higher pH values before precipitating.

Hydrated iron-sulfate and trace-metal sulfate minerals (Table 2.1) precipitate during the evaporation of acidic, and metal- and sulfate-rich water within mine-waste materials. These secondary efflorescent minerals store acid and metals that can subsequently be released by additional flow through the mine waste (e.g., precipitation events, rain, snow melt). 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 (Alpers et al. 1994). According to Nordstrom (1982) and Cravotta (1994), these efflorescent salts are highly soluble and provide an instantaneous source of acidic water upon dissolution and hydrolysis. They are partially responsible for increased acidity and metal loadings in the receiving environment during rain and snow-melt events. Metals such as cadmium, cobalt, copper, nickel, and/or zinc can be

Selected Soluble Iron-Sulfate Minerals		Some Soluble Sulfate Minerals		Less Soluble Sulfates: Some Minerals of the Alunite Group	
Mineral	Formula	Mineral	Formula	Mineral	Formula
Fell		Epsomite	MgSO₄·7H₂O	Jarosite	KFeIII ₃ (SO ₄) ₂ (OH) ₆
Melanterite	FellSO ₄ ·7H ₂ O	Hexahydrite	MgSO₄-6H₂O	Natrojarosite	$NaFeIII_3(SO_4)_2(OH)_6$
Ferrohexahydrite	FellSO ₄ ·6H ₂ O	Goslarite	$ZnSO_4 \cdot 7H_2O$	Hydronium jarosite	$(H_3O)FeIII_3(SO_4)_2(OH)_6$
Siderotil	FellSO ₄ ·5H ₂ O	Bianchite	ZnSO ₄ ·6H ₂ O	Ammoniojarosite	$(NH_4)FeIII_3(SO_4)_2(OH)_6$
Rozenite	FellSO ₄ ·4H ₂ O	Gunningite	$ZnSO_4 \cdot H_2O$	Argentojarosite	$AgFeIII_{\scriptscriptstyle{3}}(SO_{\scriptscriptstyle{4}})_{\scriptscriptstyle{2}}\!(OH)_{\scriptscriptstyle{6}}$
Szomolnokite	$FellSO_4{\cdot}H_2O$	Gypsum	CaSO ₄ ·2H ₂ O	Plumbojarosite	$Pb_{0.5}FeIII_{3}(SO_{4})_{2}(OH)_{6}$
Halotrichite	$(FeII)Al_2(SO_4)_4 \cdot 22H_2O$	Anhydrite	CaSO ₄	Alunite	$KAI_3(SO_4)_2(OH)_6$
Mixed Fell-FellI		Retgersite	NiSO ₄ ·6H ₂ O	Natroalunite	$NaAl_3(SO_4)_2(OH)_6$
Copiapite	$FellFellI_4(SO_4)_6(OH)_2{\cdot}20H_2O$	Chalcanthite	CuSO₄·5H₂O	Ammonioalunite	$(NH_4)Al_3(SO_4)_2(OH)_6$
Bilinite	$FellFellI_2(SO_4)_4 {\cdot} 22 H_2 O$	Alunogen	Al ₂ (SO ₄) ₃ ·117H ₂ O	Osarizawaite	$PbCuAl_2(SO_4)_2(OH)_6$
Romerite	FellFelll ₂ (SO ₄) ₄ ·14H ₂ O	Mirabilite	Na ₂ (SO ₄)·10H ₂ O	Beaverite	PbCuFeIII ₂ (SO ₄) ₂ (OH) ₆
Voltaite	$\mathrm{K_{2}FeII_{5}FeIII_{4}(SO_{4})}12{\cdot}18\mathrm{H_{2}O}$	Thenardite	Na ₂ (SO ₄)		
FeIII					
Coquimbite	Fe ₂ III(SO ₄) ₃ ·9H ₂ O				
Kornelite	Fe ₂ III(SO ₄) ₃ ·7H ₂ O				
Rhomboclase	HFeIII(SO ₄) ₂ ·4H ₂ O				
Ferricopiapite	$\text{Fe}_{5}\text{III}(\text{SO}_{4})_{6}\text{O}(\text{OH})\cdot20\text{H}_{2}\text{O}$				

TABLE 2.1 Selected sulfate minerals

Source: Alpers et al. 1994.

stored in these soluble salts. Their cumulative storage and incremental release may help explain the lag from mine-waste placement to ARD formation, particularly in arid to semiarid climates.

As an example, Equations 2.5–2.7 summarize the step-wise dissolution of melanterite. The net result of Equations 2.5–2.7 is summarized in Equation 2.8, which shows a net production of two moles of acid for each mole of melanterite dissolved. Cravotta (1994) showed that a similar aqueous dissolution of romerite produced 6 moles of acid for each mole of romerite dissolved.

$$FeSO_47H_2O(s) = Fe^{2+}(aq) + SO_4^{2-}(aq) + 7H_2O(aq)$$
 (EQ 2.5)

$$Fe^{2+}(aq) + (1/4)O_2(g) + H^+(aq) = Fe^{3+}(aq) + (1/2)H_2O(aq)$$
 (EQ 2.6)

$$Fe^{3+}(aq) + 3H_2O(aq) = Fe(OH)_3(s) + 3H^+(aq)$$
 (EQ 2.7)

$$FeSO_{4} \cdot 7H_{2}O(s) + (1/4)O_{2}(g)$$

$$= Fe(OH)_{3}(s) + SO_{4}^{2-}(aq) + (9/2)H_{2}O + 2H^{+}(aq)$$
(EQ 2.8)

The alunite-jarosite mineral group consists of sulfate minerals that are less soluble than the efflorescent sulfate salts (Table 2.1). According to Nordstrom (1982), the evaporative concentration of efflorescent iron sulfates leads to the precipitation of the more common iron minerals such as goethite and jarosite. Similar reaction of efflorescent aluminum sulfates will produce alunite. Alpers et al. (1994) reported that some jarosite is slightly soluble and can, therefore, contribute acid according to Equation 2.9. For example, preliminary leach studies on natural and synthetic jarosites conducted by the U.S. Bureau of Mines (W.W. White III, personal communication,

1994) showed a drop in pH from 6 in the deionized water leachant to 3 or 4 after contact with the jarosites. Notably, however, there is a wide variety of jarosite mineral compositions due to substitutions in their structure; therefore, their reactivity is also variable. Cation sites of the jarosite family may be occupied by Na, K, Ca, Ag, Pb, Sr, or NH $_4$ and anion sites by SO $_4$, PO $_4$, AsO $_4$, or CO $_3$. Lapakko and Berndt (2003) examined acid production by two siltite-argillite rock samples in which sulfur occurred largely as pyrite and two in which sulfur occurred largely as potassium jarosite. They reported that drainage pH values from the pyritic samples were one to two units lower and acidities were 10 to 70 times higher than associated values from the potassium jarosite samples of similar sulfur content.

$$KFe_3(SO_4)_2(OH)_6(s) = K^+ + 3FeOOH(s) + 2SO_4^{2-}(aq) + 3H^+(aq)$$
 (EQ 2.9)

C.N. Alpers (personal communication, 2000) speculated that a pure jarosite or hydronium jarosite may buffer pH in the range of 1.5 to 3. Lapakko and Berndt (2003) reported that dissolution of hydronium jarosite present in one of the siltite-argillite samples was likely responsible for pH values as low as 3.4 and sulfate release rates 5 to 10 times those attributed to subsequent dissolution of potassium jarosite. Acid generation by jarosite minerals has been reported by Desborough and Fey (1997) who studied the solubility and acid-generating capability of fine-grained natrojarosite (<1 mm) in abandoned mine wastes in Jefferson County, Montana. Other leachate chemistry studies have shown that acid-generating mine-waste samples which contained jarosite and no other potential acid-producing minerals suggested that jarosite was a factor in generating low-pH conditions (Desborough et al. 1999).

ACID NEUTRALIZATION

The balance between the rates of acid production by iron-sulfide mineral oxidation and host-rock mineral neutralization will determine the acidity of mine-waste drainage. The most effective minerals for neutralizing acid are those containing calcium carbonate and magnesium carbonate, including calcite, magnesite, dolomite, and ankerite (CaCO₃, MgCO₃, CaMg(CO₃)₂, and CaFe(CO₃)₂, respectively). The acid neutralization potential of the ankerite is reduced by the carbonate associated with iron. Equation 2.10 represents the dominant acid-neutralizing reaction of calcite (CaCO₃) above pH 6.4, whereas Equation 2.11 is the dominant reaction below pH 6.4 (Drever 1988):

$$CaCO_3(s) + H^+(aq) = HCO_3^-(aq) + Ca^{2+}(aq)$$
 (EQ 2.10)

$$CaCO_3(s) + 2H^+(aq) = H_2CO_3(aq) + Ca^{2+}(aq)$$
 (EQ 2.11)

Of the carbonate minerals, calcite (CaCO₃) dissolves most rapidly (Busenberg and Plummer 1986). Relative to calcite, the rate of dolomite [(Ca,Mg)CO₃] dissolution is about an order of magnitude slower (Busenberg and Plummer 1982) and the rate of magnesite (MgCO₃) dissolution is about 4 orders of magnitude slower (Chou et al. 1989). The rate of siderite dissolution under anoxic conditions is reported to be 3 orders of magnitude slower than that of calcite (Greenberg and Tomson 1992). However, iron and manganese carbonates do not provide net acid neutralization under oxidizing conditions, which is attributable to oxidation of the released iron or manganese, the subsequent hydrolysis and precipitation of these metals, and the consequent acid production (e.g., Equations 2.7 and 2.8). This has important implications for static test evaluation, as discussed in Chapter 4.

Dissolution of silicates such as plagioclase feldspars (e.g., anorthite in Equation 2.12; Busenberg and Clemency 1976) and olivine (e.g., forsterite in Equation 2.13; Hem 1970) can also neutralize acid. However, their rates of dissolution and consequent acid neutralization are slow relative to the carbonate minerals (Nesbitt and Jambor 1998), and they do not prevent acid formation by providing alkalinity as carbonate minerals do. For example, White et al. (1999) noted that "at near neutral pH, the dissolution rate of calcite is approximately 7 orders of magnitude faster than the dissolution of plagioclase feldspar." Nonetheless, silicate mineral dissolution can maintain neutral conditions if the rate of acid production is quite slow (Lapakko and Antonson 1994; 2002; Lapakko et al. 1997). The effectiveness of silicate minerals in neutralizing acid increases with increasing mineral surface area that, in turn, increases with increasing mineral content and decreasing grain size.

$$CaAl_2Si_2O_8(s) + 2H^+(aq) = Ca^{2+}(aq) + Al_2Si_2O_5(OH)_4(s)$$
 (EQ 2.12)

$$Mg_2SiO_4(s) + 4H^+(aq) = 2Mg^{2+}(aq) + H_4SiO_4(aq)$$
 (EQ 2.13)

TRACE METAL RELEASE BY METAL SULFIDE OXIDATION

Trace metals are metals that occur at low average concentrations in the earth's crust but can be present at elevated levels in mineralized areas. Trace metals commonly occur as or within sulfide minerals, the oxidation of which releases the trace metal from the highly insoluble sulfide phase (e.g., Reaction 2.14). Once released to solution, several types of reactions can influence the migration and fate of these metals. Smith and Huyck (1999) present a series of diagrams for the generalized relative mobility of elements under different environmental conditions for use as an initial estimate of metal behavior in surficial environments. At a regional scale, generalizations can frequently be used to estimate broad trends in metal mobility. However, as the scale becomes increasingly finer, estimating metal behavior generally becomes more difficult (Smith and Huyck 1999).

$$ZnS(s) + 2O_2(aq) = Zn^{2+}(aq) + SO_4^{2-}(aq)$$
 (EQ 2.14)

In general, metals may remain in solution or be removed in secondary phases. For removal from solution, trace metals may precipitate as oxides, hydroxides, or carbonates; be adsorbed by surfaces such as iron oxyhydroxides (Smith 1999); or co-precipitate with other solid phases such as efflorescent salts. In acidic solutions, trace metal removal is limited, and elevated trace metal concentrations are often associated with these solutions because most metals are mobile at low pH. However, circum-neutral drainages can also contain elevated concentrations of trace metals such as nickel, copper, cobalt (Lapakko 1993), zinc, manganese (Smith and Huyck 1999), molybdenum (Brown 1989), arsenic, and antimony. Concentrations of oxyanions of molybdenum, arsenic, selenium, and antimony in particular can be elevated even as pH increases above 7.

Oxidation of arsenic and antimony-bearing sulfides can produce acid, as can oxidation of the iron sulfide fraction of mixed sulfide minerals such as chalcopyrite (Plumlee 1999). Other trace metal sulfide oxidation will produce acid if and only if the metal released hydrolyzes (Reaction 2.15) or precipitates as a hydroxide, oxide, or carbonate (Reaction 2.16). For most trace metals, this will occur only at pH levels above 6, and as pH decreases below this level, the secondary phases will dissolve. Consequently, they do not generally contribute to acid production observed at lower pH levels.

$$Zn^{2+}(aq) + H_2O(aq) = Zn(OH)^+(aq) + H^+(aq)$$
 (EQ 2.15)

$$Zn^{2+}(aq) + 2H_2O(aq) = Zn(OH)_2(s) + 2H^+(aq)$$
 (EQ 2.16)

KEY SOLID-PHASE FACTORS CONTROLLING MINERAL OXIDATION AND DISSOLUTION

While the acid-producing and acid-neutralizing mineral contents, and the balance of these contents, influence mine-waste drainage quality, there are several subtle mineralogical factors that are also influential. Individual minerals may be entirely liberated from the rock matrix, occur interstitial to other minerals (partially liberated), or come about as inclusions encased within other minerals. The extent of liberation affects availability for reaction. For example, acid-producing or acid-neutralizing minerals armored within minerals such as quartz will be essentially unavailable for immediate reaction. However, gangue minerals such as quartz that are broken during blasting, crushing, milling, transport, or deposition may have increased surface area through fracturing, which would allow more rapid reaction with oxygen and fluids.

Oxidation of sulfide minerals and dissolution of carbonate minerals are surface reactions, and, therefore, the rates of these reactions are dependent on available reactive surface area. Reactivity decreases as mineral surfaces are covered with coatings, such as iron oxyhydroxides. This is important because limestone (CaCO₃), which is commonly used as a neutralizing agent such as in riprap around remediation ponds, can become surficially coated by iron oxyhydroxides and gypsum. Such coatings can, for example, form on calcite (CaCO₃) present in mine wastes and reduce the rate at which these minerals dissolve and neutralize acid (Hammarstrom et al. 2003). Mineral surface area is dependent on the extent to which the mineral is liberated from the rock matrix, mineral grain size, and the "roughness" of the mineral surface.

Any concentration of lattice defects in an evaluated mineral tends to increase reactivity. This can generally be evaluated only by more extensive detailed mineralogical work. Lattice defects may be due to structural deformation or to the presence of trace elements. Trace-element-rich minerals that undergo dissolution are commonly sources of elevated metal concentration in drainages (Diehl et al. 2003). For example, sulfide minerals such as pyrite commonly host arsenic, cobalt, or nickel; galena frequently contains silver; iron and cadmium substitute in sphalerite; and jarosite commonly contains lead, silver, copper, or zinc. Element substitution in a mineral may distort the lattice structure, which changes its physical properties, thereby increasing the susceptibility of a mineral to dissolution (Savage et al. 2000; Lehner and Savage 2004).

DATA NEEDS FOR PREDICTION

The first step in developing a program for prediction of drainage quality from mine and associated processing wastes is a detailed mine plan. This plan will identify the ore and wastes to be generated and provide a preliminary estimate of the corresponding masses, as well as an initial plan for the environmentally sound management of the wastes. Descriptions of the mining-related materials of concern and their proposed disposal facilities are required to develop a rationally based program of mine-waste characterization and drainage quality prediction. The characterization and prediction conducted might lead to changes in the mine plan, ore processing and waste management facilities, and overall water balance. Thus, the mine planning, processing options, waste facility design, water balance, and the characterization and prediction program become an iterative process. Where a positive water balance is anticipated, treatment and discharge of impacted water will be important aspects of the facilities planning.

One of the most important data needs for the prediction of ARD is an accurate representation of the rock types to be mined. This should include the overall lithology, alteration, mineralogy, major and minor trace-element chemistry, and the volume of mined material, both ore and waste. The timing of various mining and processing activities in the mine life cycle is also critical information. Conducting predictive tests on material that is not representative of material to be mined can lead to unpleasant surprises and can require dramatic changes in mine or reclamation plans and corresponding changes in financial guarantees (Kuipers et al. 2006).

Mineralogical and rock analyses are also the basis for interpreting water–rock interactions and the sources of contaminants, discussed in more detail in Chapter 3. These analyses are also critical for interpretation of static and kinetic tests. Geochemical modeling, load calculations, and water quality compliance are based on reliable water analyses. All water sampling, preservation, and analysis should follow established quality assurance and quality control protocol for sampling and analysis (ASTM D 5907). Water samples collected for the purpose of ARD characterization or prediction should be analyzed for complete major ion analysis (inductively coupled plasma emission spectrometry, flow injection, and ion chromatography), relevant trace elements (i.e., inductively coupled plasma mass spectrometry and mercury by cold vapor atomic absorption spectrometry), Fe(II/III) and other oxidation reduction species as well as alkalinity, acidity, and total dissolved solids. Any biological sampling should follow the relevant established protocols.

It is imperative that these analyses be reasonably accurate and precise. Estimates of uncertainty (accuracy, precision, errors from sampling, errors from contamination of samples, and temporal and spatial variance of the data) must be explicit. This information can subsequently be used to develop ranges of values that are often easier to defend than one specific value. See Appendix 3 of *Sampling and Monitoring for the Mine Life Cycle* (McLemore et al. 2014).

Particular care should be taken with waste rock samples because waste rock is largely composed of noneconomic overburden and waste internal to the deposit, which generally represents the bulk of the waste products produced by a mine. Inaccurate sampling and representations of waste rock can lead to large volumes of inaccurately characterized material that can be difficult and expensive to either resample and recharacterize or remediate. Tailings from metallurgical processing of ores are generally less of an issue from a sampling standpoint, as they are more homogeneous than waste rock from the mine and are stored in dedicated process waste containment facilities. However, care should be taken to ensure that tailings samples accurately represent the ore material and processes that are being used or are anticipated to be used at the processing site throughout the mine life. Spent dump and heap leach metallurgical materials provide a greater sampling challenge than mill tailings because of the larger particle size and larger quantities of waste generated by the process.

Statistical evaluations can be helpful for the sampling plan and its interpretation but do not supersede the need for process-oriented interpretations.

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CHAPTER 3

Prediction Factors

GEOLOGIC, LITHOLOGIC, AND ALTERATION CONSIDERATIONS

Accurate geologic mapping of the project area, at a regional scale and a local, more detailed scale, is a prerequisite for any detailed predictive work. As discussed in the "Geo-Environmental Models" section that follows, mineral deposits generally occur as well-defined types and classes of mineralization (Seal and Foley 2002). Regional-scale mapping can help develop information on the types of deposits that might be expected, and local mapping can detail the actual deposit. Local-scale mapping will, of course, generally be focused on the various aspects of economic mineralization, but much of the information relevant from an economic standpoint is also useful for waste characterization. Information on alteration types (see Figure 1.2), iron sulfide presence, and morphology, veining, structures, including faults, jointing and folds, host and country rock lithology, and mineralogy is all relevant from economic and environmental perspectives. To the extent possible, mapping should represent this information in three dimensions. Particular attention should be paid to features that influence water flow and availability, as these can be crucial environmental issues.

Preliminary surface mapping of an area can often detail existing evidence of natural acid rock drainage (ARD) in ferricretes and alteration scars, as shown in Figure 3.1. Figure 3.1 displays unmined, perched terraces of ferricrete, iron-oxide cemented Holocene sediments, from Limestone Hills (Figure 3.1a) and Swift Gulch in Montana (Figure 3.1b). Alteration scars in Red River, New Mexico (Figure 3.1c), are areas of unmined, highly altered rock where sulfide mineralization has been weathered out, leaving behind highly bleached and erosive material. Figure 3.1d, also from Red River, shows a road cut through ferricrete, which was deposited below the alteration scar shown in Figure 3.1c.

Geologic evidence of natural acidic drainage, such as shown in Figure 3.1, is a key piece of information that acid drainage has a strong possibility of occurring if the mineral deposit being explored is related to a historic mining disturbance or occurs in similar geologic terrane.

Where exploration activities are occurring in areas with historic mines, waste dumps and mine features may have similar indicators of acidic drainage such as lack of vegetation, bright colors, and iron staining, as shown in Figure 3.2.

GEO-ENVIRONMENTAL MODELS

Geo-environmental models (GEMs) can provide an initial assessment of potential water quality impacts based on characteristics of the mineral deposit. Initial compilations of GEMs are given in du Bray (1995) based in part on Cox and Singer's (1986) work on ore deposit models. A GEM is defined as a "compilation of geologic, geochemical, geophysical, hydrologic and engineering information pertaining to environmental behavior of geologically similar mineral deposits (1) prior to mining, and (2) resulting from mining, mineral processing, and smelting" (Plumlee and Nash 1995). Plumlee et al. (1994) also noted that "a detailed understanding of mineral deposit geology and geochemical processes, which control element dispersion into the environment, is

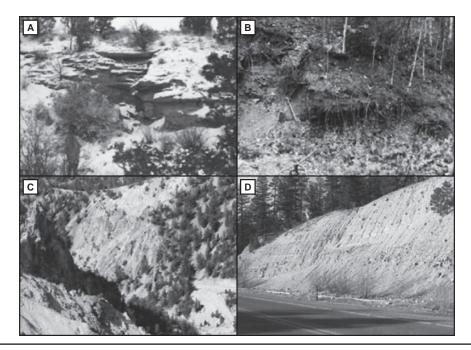


FIGURE 3.1 (a) Weathering-resistant ledges of ferricrete exposed in outcrop, east of Limestone Hills, Montana; (b) ferricrete ledge in Swift Gulch, north of Zortman–Landusky, Montana; (c) eroded and oxidized mineralized rock on a steep slope, Red River, New Mexico; and (d) a road cut through ferricrete, Red River, New Mexico

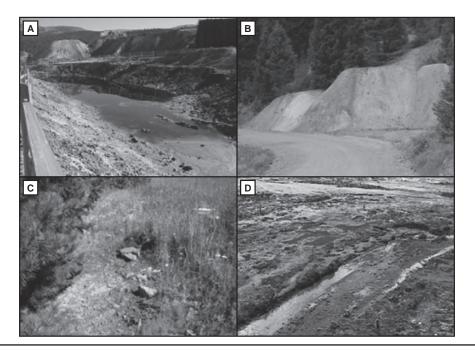


FIGURE 3.2 Mine features that demonstrate a strong likelihood of acid drainage, such as ironoxyhydroxide deposits, may be a concern: (a) acidic wastes and waters, Leadville, Colorado; (b) unvegetated acidic waste pile, High Ore, Montana; (c) acidic seep from a waste repository, Elkhorn Mountains, Montana; and (d) acidic waters and remnant tailings, High Ore, Montana

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crucial for the effective prediction, mitigation, and remediation of the environmental effects of mineral resource development." There are recent mineral discoveries and ore deposit models that do not fit into some of the earlier literature on ore deposit and GEMs and may require additional background work before use in planning an environmental program.

In developing a GEM, information from the field of economic geology is compiled and translated from the language of economic geology and mining engineering to the language of environmental science (Seal et al. 2000). Key descriptors in an environmental model are deposit type, related deposit types, deposit size, host rocks, surrounding geologic terrane, wall rock alteration, nature of ore, mining and ore processing methods, deposit trace element geochemistry, primary mineralogy and zonation, secondary mineralogy, soil and sediment signatures, topography, hydrology, drainage signatures, climatic effects, and potential environmental concerns (Ficklin et al. 1992; Plumlee 1999; Plumlee et al. 1999). Thus, using GEMs, sites can be initially assessed by determining local site characteristics and comparing them with other known deposits with similar characteristics. This can enable an assessment of the potential for adverse impacts on water quality. It can also provide an advance indication that detailed rock characterization work will be required as well as be an aid in defining the initial scope of a prediction program.

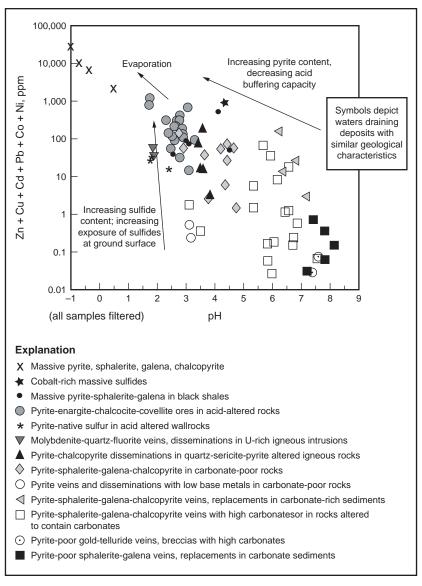
Figure 3.3 is a plot of pH verses total metal content (the sum of dissolved Zn, Cu, Cd, Co, Ni, and Pb concentrations) of waters draining from diverse mineral deposits. The purpose of this plot is to illustrate the typical metal content of waters derived from specific types of mineral deposits (Ficklin et al. 1992; Plumlee 1999; Plumlee et al. 1999).

This information can then be used to improve the development of baseline environmental programs, helping to avoid missing trace elements that might result from complex porphyry systems or other deposit types not clearly identified in earlier literature. This method can have some applicability for areas where there may be information on ore deposit types, but little supporting geochemistry or environmental work (Williams 1995). It can also serve as a representation of the range of impacts that might be considered for similar deposits by representing the differing potential metal and water quality impacts on a revised Ficklin diagram, shown in Figure 3.4.

ANALYTICAL TECHNIQUES FOR MINERALOGICAL INVESTIGATIONS

The following discussion, largely expanded from Lapakko (2002, 2003), focuses on mineralogical identification techniques most commonly applied to mine-waste samples. These include optical microscopy, whole rock analysis, X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscopy (SEM) (Table 3.1) (Lapakko 2002, 2003). XRF has been commonly used to obtain bulk chemical analyses of a sample's elemental composition, including major and some trace elements; but XRF does not provide detailed information on the mineral composition of the sample. Whole rock analysis based on inductively coupled plasma (ICP) and inductively coupled plasma mass spectrometry (ICP-MS) (along with XRF, carbon-sulfur by combustion and others) is also commonly used for the elemental composition of the samples for interpretation of XRD. The microanalytical techniques listed in Table 3.1 are used to visually identify mineral and elemental distribution and composition in mine-waste material, identify dissolution textures and other weathering features, determine the residence sites and mineral hosts of trace elements, and quantify element concentration.

Jambor and Blowes (1998) reported on the application of traditional mineralogical techniques for analysis of sulfide-bearing mine wastes. The authors noted that XRD "provides an easy and reliable method of identifying the common carbonate minerals." The integrity of mineralogical results is improved by subjecting polished, thin sections to analysis by XRD (to obtain

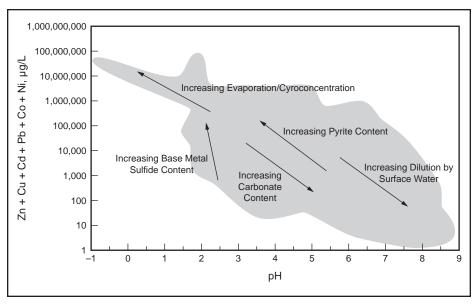


Courtesy of U.S. Geological Survey.

FIGURE 3.3 Ficklin plot

diffractograms), transmitted-light microscopy (for petrographic description), and reflected-light microscopy (to define "ore" mineralogy and mineral assemblages). Optical microscopy is recommended in addition to XRD in order to verify mineral identifications, resolve any ambiguous or overlapping XRD data, and to identify minerals unidentified by XRD because of their low content or poor crystallinity. Optical microscopy is also valuable for the identification of primary and secondary sulfide, silicate, and carbonate (among others) minerals that may have an impact on either water quality or reactivity in the material. It enables an identification of crystal habits and textural relationships that can also have an impact on reactivity. References for additional information on XRD include Klug and Alexander (1974) and Azaroff and Buerger (1958).

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Source: Adapted from Plumlee 1999 and INAP 2009.

FIGURE 3.4 Revised Ficklin diagram showing likely water quality variations resulting from the different types of deposits shown in Figure 3.3

TABLE 3.1 Microanalytical techniques

Parameter	Method	Result
Mineralogy	Petrographic microscope Transmitted and reflected Light microscopy	Identify mineral species Acid-generating minerals Non-acid-generating minerals
	Scanning electron microscope (SEM) with energy X-ray dispersion	Mineral species Mineral textures Particle size Cleavage, grain boundaries Surface weathering Deformation structures Microfaults, veins, fractures Semiquantitative data
	X-ray diffraction (XRD) X-ray fluorescence (XRF)	Bulk mineralogy, 95% of major phases
Minor and trace- element chemistry	Electron probe microanalyzer (EPMA)	Exact residence of minor and trace elements Spatial distribution of minor and trace metals Semiquantitative and quantitative data
	Laser ablation ICP-MS	Quantitative data
	Qemscan and other software	Quantitative data Spatial distribution of major to trace elements

XRD cannot identify amorphous minerals, such as oxyhydroxides of iron, aluminum and aluminosilicates, or poorly crystallized minerals or very fine-grained minerals that are commonly associated with acidic drainage. It can, however, be used to determine the amount of amorphous material present using the Rietveld method (Young 1993).

Analyses can be further augmented by SEM, which can achieve magnification roughly 100 times that of an optical microscope. Adding an X-ray spectrometer to the SEM allows chemical

analysis of small areas of interest. Two types of X-ray spectrometry are energy-dispersive (EDS or EDX) and wavelength-dispersive X-ray spectrometry (WDS), the latter of which is also referred to as electron-probe microanalysis (EPMA; Gill 1997). Each instrument has its benefits and limitations. SEM has better imaging resolution, but the detection limit for elements of concern such as As, Cd, Co, Cu, Pb, or Zn is approximately 1,000 ppm. EPMA has better X-ray spectrometry resolution with detection limits in the 100s ppm or less (60–70 ppm) for quantitative spot analysis.

SEM and X-ray spectrometry are "essential to obtain compositional data for solid-solution minerals, to obtain information on fine-scale features such as alteration rims, and to verify the identification of grains too fine-grained to be unambiguously identified by optical microscopy" (Jambor and Blowes 1998). This is of particular value in ascertaining the composition of complex carbonate minerals to determine the extent of acid-neutralizing components. A variety of approaches to determining ore and the associated environmental mineralogy are presented by Cabri and Vaughan (1998). Applications of mineralogical techniques to determine reactions within mine wastes and to assess mine-waste remediation in dry climates is presented by Esposito (1999) and Whitney et al. (1995), respectively. Newer technology (e.g., Qemscan, MLA, and others) includes testing equipment that incorporates elements of SEM and EPMA and can provide highly detailed information on individual mineral grains or finer and extend the sensitivity of mineral identification below the percentage range normally limiting XRD (Hoal et al. 2009; Figure 3.5) and below tenths of a percentage reproducibility of carbon-sulfur analysis methods for estimation of carbonate and sulfide contents (e.g., ASTM E 1915). An earlier version of this technology (e.g., QEM*SEM) was applied for mine-waste characterization, and the results compared to those from laboratory dissolution tests (Pignolet-Brandom and Lapakko 1990). Near infrared analyses also show considerable promise in highly detailed evaluation of mineral samples.

Quantitative minor-to-trace-element data may also be gathered by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS; Ridley 2000). LA-ICP-MS can determine element concentration to less than 1 ppm. Besides spot analyses, an LA-ICP-MS line scan across a composite mine-waste sample is a rapid technique to determine what metals remain in a waste pile and their mineralogic concentrations (Diehl et al. 2008). LA-ICP-MS data is recorded as counts per second in spectral peaks; these spectra can be overlaid for element correlation purposes. For example, spectral peaks of cobalt and nickel may overlap the peaks of iron, indicating that cobalt and nickel reside in pyrite or other iron-sulfide mineral.

A detailed evaluation of the minerals and their composition can be a challenging undertaking but may be required not only for the purpose of ARD potential and waste characterization but also for effective and economic recovery of the desired products. The choice of methods used for mineral and/or element analysis will depend on the questions that need to be answered; that is, is it more important to know the bulk concentration of an element of interest or the mineralogic residence site of that element?

Regardless of the technology or evaluation method selected, it is critical to remember that an understanding of the mineralogy is essential to understanding and interpreting the predictive tests, not the other way around.

FIELD MEASUREMENTS

Field measurements can be used to supplement information developed through the use of GEMs. This is often done during exploration through the evaluation of preexisting regional geologic and environmental information. Field measurements can include the identification of natural or anthropogenic ARD during exploration activities, as shown in Figures 3.1 and 3.2. The

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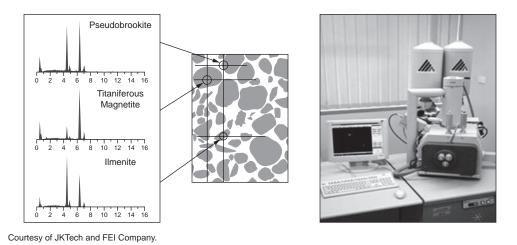


FIGURE 3.5 SEM with mineral identification software incorporated

development of a geo-environmental geographic information system (GIS) database during the exploration phase can include water quality and quantity information, and geologic and mineralogical information, which can be very helpful in establishing baseline conditions for the permitting phase of a project. Field portable pH meters and XRF units, which have become much more affordable and reliable, can be helpful in field characterization work, particularly when linked into the GIS database.

GEOPHYSICAL INVESTIGATIONS

Remote Sensing

Airborne imaging spectroscopy is a recently developed technology that has been used for screening large areas for abandoned mine wastes. This type of tool can be used to locate mine and processing wastes and assist in prioritizing sites for further characterization. Imaging spectroscopy can be used to identify minerals generated by sulfide-mineral weathering that are exposed on the earth's surface. The Airborne Visible/Infrared Imaging Spectrometer (AVIRIS), when flown at an altitude of 20 km, can cover a path 10.5 km wide with resolution to 17-m pixels (K.S. Smith et al. 2000). Other aircraft fly at 4 km for a more detailed evaluation. The data generated are digested using the U.S. Geological Survey (USGS) Tetracorder (Clark et al. 1990). Manual spectrometers allow this technology to be readily applied in the field. Swayze et al. (2000a, 2000b) used AVIRIS in conjunction with the USGS Tetracorder to map iron-bearing products (jarosite, goethite, hematite) of sulfide-bearing mine-waste weathering. Jarosite appeared to be the best indicator of acidic conditions. Dalton et al. (2000) applied the same technology to determine the presence of acid-generating and acid-neutralizing minerals in a watershed with sulfide mineralization.

Conventional Geophysical Techniques

Airborne geophysical surveying (radiometric, magnetic, and electromagnetic) can be used to augment geological mapping data for location of abandoned mine wastes and preliminary ranking of sites with regard to potential water quality impacts. These techniques can be applied to map subsurface lithology, structure, and groundwater flow. B.D. Smith et al. (2000) described application of these techniques at regional (e.g., state) and local (e.g., watershed) scales. Geoelectrical methods that have been evaluated or used for exploration and mine-waste work include direct

current resistivity (DCR), electromagnetic induction (EMI), induced polarization (IP), ground-penetrating radar (GPR), and spontaneous potential (SP). Other geophysical techniques utilized have included magnetics, exploration seismic methods, and direct push methods (Campbell and Fitterman 2000). Most of these methods rely on the electrical conductivity of sulfide minerals and associated aqueous solutions. The most successful methods, as reported by Campbell et al. (1999) for the evaluation of mine dumps, have been some of the various EMI methods. IP has proven useful in helping to distinguish mineralogy, while SP, GPR, and magnetics have had limited success in mine-waste work (Campbell et al. 1999). Recent work with resistivity (Williams et al. 2009) at Zortman–Landusky in north central Montana has helped to identify likely structurally controlled zones that are transmitting low-pH water to a sensitive drainage north of the site. This work was related to postmine closure, but the same approach could also be useful for initial characterization work, if appropriate.

HYDROGEOLOGICAL AND HYDROLOGICAL INVESTIGATIONS

As impacts to ground- and surface waters are often a critical component of ARD prediction and characterization, hydrogeological and hydrological background data and characterization by watershed can be some of the most important information to obtain. This data should be obtained as early as possible during the exploration and assessment phases of a project, because it is vital information as an environmental assessment document is prepared. Information to be collected should include groundwater flow paths, quality, quantity, elevations, and discharges. Surface water flows, quality, and storm curves should be collected. A general site water balance for the project area should be performed.

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CHAPTER 4

Overview of Prediction Methods

It is a challenge to incorporate all possible test objectives, lithologies, mineralogy, economics, mine life-cycle operating constraints, and environmental issues that might be involved at a particular mine, let alone a generic mine site, in one conceptual figure. Ultimately, the goal of Figure 4.1 is to illustrate the tools for use in an adaptive waste characterization and management program that can minimize water-quality and environmental problems in a cost-effective manner throughout the mine life. Tests methods shown in the figure are used to characterize the potential for mine waste to produce acid rock drainage (ARD) and to release other solutes, including metals, metalloids, nutrients, and process reagents. These methods can be used in varying combinations to achieve optimum waste management.

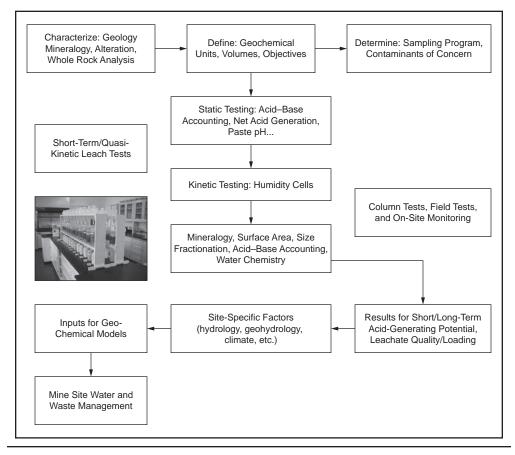


FIGURE 4.1 Generalized flowchart for acid rock drainage prediction and waste characterization

For example, in one case, a specific lithology may be a clear acid producer and can be managed using a simple static test method accordingly with monitoring throughout the mine life, mainly focused on confirming earlier predictions. In another case, a lithology may be questionable as an acid producer because of closely balanced sulfur and carbonate values; in this case, a much more detailed characterization program may be required throughout the mine life. Such a detailed program could include a range of detailed static, kinetic, and field tests to ensure adequate risk management. In some cases, simple field tests (paste pH, etc.) can be used to serve as surrogates for more detailed characterization tests. This can lead to operational and economic efficiencies in waste characterization and handling. In some situations, it may be more efficient and environmentally favorable to avoid the expense of a characterization program and handle material as if it were acid producing even though initial characterization work may be open to interpretation. Generally, the higher the potential risk posed by a particular lithology or waste facility or the greater the associated uncertainty in the available data, the more robust a proposed characterization and handling program will need to be.

Methods for the prediction of ARD are divided into two broad categories: static and kinetic. Static tests describe the geochemistry at a fixed point in time, and kinetic tests describe rate changes in response to experimental conditions over time. A third class of tests is short-term solute leach tests.

Static tests are the most commonly used tests for the estimation of ARD potential. They are relatively quick (1 day to 2 weeks) and inexpensive (\$30 to \$80) per sample. The cost per sample is highly dependent on the number of samples, tests per sample, and duration of the proposed sampling program. Static tests determine the balance between acid-producing potential and acid-neutralizing potential and are typically used to screen mine-waste samples for additional testing. The acid-producing potential is determined by the sulfur (S) content, and the neutralization potential is generally determined by a titration procedure or estimation of the calcium carbonate plus magnesium carbonate content of the sample. Numerous variations on this central theme are discussed in the following paragraphs. Static tests make no real prediction about whether ARD will actually occur; rather, they estimate the balance of acid-producing and acid-neutralizing components present in a sample. The actual ability of a sample to produce low-pH effluent will depend on the relative rates of acid production and acid neutralization.

Kinetic tests are neither quick nor inexpensive, requiring 20 weeks or more of dedicated laboratory testing, but they can provide more valuable element/solute release data and more reliable predictive leaching information than acid-base accounting methods. As with static tests, there are several variations on this central theme, which are also discussed in the following paragraphs.

Static and kinetic tests are laboratory prediction methods that typically require simplifications in the reaction environment (e.g., temperature, humidity, and gas-phase composition), physical scale, hydrologic variables, and time of reaction (Lapakko 1992). These simplifications generally preclude direct simulation of drainage quality at the field scale. For example, laboratory tests use relatively small sample masses, and for waste rock, the particle size is much finer compared to run-of-mine rock. The particle-size difference can affect the relative surface areas of sulfide and calcium/magnesium carbonate minerals and, consequently, respective rates of acid production and acid neutralization (Lapakko et al. 2006). Although laboratory predictive tests are designed to measure characteristics that affect field-drainage quality using well-defined protocols that are repeatable and reproducible to a relatively high degree, these differences affect the ability to transfer their results directly to the field environment. Their results are therefore intended to aid the geochemist in prediction of drainage quality, with an understanding of the test methods and the storage of ore and ultimate disposal conditions of the metal-mine waste and processing materials.

Influences of sample lithology, particle size, surface area, mineral composition, degree of mineral liberation, and other factors on ore and mine-waste dissolution and predictive test results have been described in numerous papers (e.g., MEND 1991, 2000; Lapakko 1993, 2003; White and Jeffers 1994; White et al. 1997, 1999; Lapakko and White 2000; White and Lapakko 2000; Bucknam 2002; Jambor 2003; Lapakko et al. 2006).

It cannot be overemphasized that an understanding of the mineralogy and petrology is critical to correctly evaluating static, kinetic, or short-term leach tests. All of these laboratory tests are predicated on either the calculation of a chemical balance or challenging the mineralogy through the promotion of an accelerated chemical reaction or the creation of a controlled dissolution setting. It is important to understand that virtually all tests make some assumptions about how the sample will respond to the testing procedures discussed in the following sections. For example, kinetic tests are designed to help predict how a sulfide-bearing sample will oxidize and dissolve in the weathering environment.

Collectively, chemical, petrologic, and mineralogic analyses in conjunction with static, short-term leach, and kinetic test results must provide a body of information consistent with scientific principles that are adequate to make mine-waste management decisions (Morin and Hutt 1999). This determination of adequacy is dependent on the levels of uncertainty associated with the data and its extrapolation to the operational scale. The mine-waste management plan, its contingencies, and associated financial assurance must be adequate to account for these predictive uncertainties. This application of data must be considered in test design and, ultimately, mine design and operation.

STATIC TEST METHODS

Static tests represent the environmental geochemistry of a mine waste at a fixed point in time. They describe the acid generation potential based on an analysis of acid-producing and acid-neutralizing minerals using any of a number of analytical methods. Since the 1990s, the original static methods have undergone numerous refinements to address known mineralogical limitations of the techniques. An additional review of static methods currently in use by the industry is provided in the International Network for Acid Prevention's (INAP's) *Global Acid Rock Drainage Guide*, or GARD Guide (INAP 2009b).

Acid-base accounting (ABA) is the generic term for static tests that estimate acid drainage potential through an independent determination of acid-generating potential (AP or AGP, generally sulfides) and acid-neutralizing potential (NP or ANP, generally carbonates).

Common laboratory methods applicable to various aspects of most ABA tests can include

- Pulverizing or grinding, commonly to a fraction ranging from -60 mesh to -400 mesh, which is typically smaller than run-of-mine rock;
- Digestion in a strong acid, generally sulfuric (H₂SO₄) or hydrochloric (HCl), or combustion at approximately 550°C;
- · Calculation of a neutralization value based on acid consumed by titration or combustion infrared spectrometry; and
- · Calculation of an acid-producing value based on either wet chemical or LECO furnace analysis for sulfur.

Acid-Base Accounting

ABA values can sometimes be correlated with other predictive tests and used as a screening tool for waste management. Static tests cannot predict the relative rates of mineral dissolution

of either sulfide or carbonate minerals, so samples that are represented as either acid or neutral based strictly on ABA testing may, in fact, turn out differently based on mineral dissolution rates. Because different minerals and mineral assemblages will respond differently to the physical aspect of these tests, and the calculations rely on assumptions about how the mineral assemblages will react, these tests provide abundant opportunities to be misinterpreted by either overestimating or underestimating neutralization or acid potential. An understanding of the detailed mineralogy of samples and the likely range of errors in the chosen ABA method are important in evaluating static tests. Ideally, a detailed understanding of a sample's mineralogy can help in the selection of an ABA test that will most accurately reflect acid potential and neutralization of the samples' particular mineral assemblage (White et al. 1999). The different types of static tests are variations on the central theme previously discussed, with various refinements attempting to correct for known inaccuracies in the test. These can include the following:

- · Sulfur in samples that do not contribute to acidity, such as gypsum (CaSO₄) and barite (BaSO₄).
- · Carbonate that does not contribute to acid neutralization (i.e., carbonate not associated with calcium or magnesium), such as siderite (FeCO₃), rhodocrocite (MnCO₃), and iron and manganese fractions of ankerite (Ca, Fe, Mg, Mn (CO₃)₂). In reality, the Fe present in the sample ultimately generates acidity and thus does not contribute to NP, so that the NP of the sample is overestimated when FeCO₃ is included (Skousen et al. 1997). Also, static tests do not accurately account for neutralization potential that can be provided by other noncarbonate minerals such as silicates. Uncertain risk of ARD identified in static tests indicates a need for further kinetic testing.

Figure 4.1 is a representation of how static test results might be displayed.

The tests described in the following subsections will be discussed in general terms, and a reference given for each actual protocol. A more detailed critique of static tests is presented by White et al. (1999). The appendices include the test protocols for many of the following tests.

Sobek method. This technique was developed by Smith and others (1974) and later modified by Sobek and others (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 that is designed to estimate the calcium carbonate and magnesium carbonate content of the sample. An acid solution comprised of one part concentrated HCl to three parts water is applied dropwise to 0.5 g of sample, and the extent of "fizzing" is observed (the fizzing is a result of the reaction of the acid and carbonate present in the sample). Based on the vigor of this reaction, the volume and concentration of HCl to be added to 2 g of minus 60-mesh mine-waste sample are determined ("no fizz," 20 mL 0.1 N; "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 Sobek method. This technique, 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, 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). 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). This will underestimate the AP if acid-producing sulfate minerals such as alunite or jarosite are present.

In place of a heated acid digestion, the modified Sobek 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 2.0 to 2.5 (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; Lawrence and Wang 1997). The final pH digestion range has been changed twice since 1990 when it was first designated as 1.0 to 1.6 by Lawrence (1990). It was subsequently modified from 1.5 to 2.0, and later as 2.0 to 2.5 (Lawrence and Wang 1996, 1997). The changes in final pH reflected an understanding that the lower pH values were resulting in dissolution of silicates that were unlikely to contribute NP maintaining a pH > 6.0. Even at a final pH value of 2.0–2.5, there is still mineral dissolution that contributes to calculated NP but will not help maintain an environmentally acceptable pH (K.A. Lapakko, personal communication, 2013).

In spite of these refinements, there are still opportunities to miss important sulfur-bearing minerals that may produce acid or minerals that can neutralize produced acid (Jennings and Dollhopf 1995). This again highlights the importance of a detailed understanding of the sample's mineralogy.

Siderite correction. This method was developed in Pennsylvania as a correction for samples that include siderite (FeCO₃), which is a common mineral in the Appalachian coal fields. Siderite is incorrectly assigned as a contributor to NP in the Sobek test. This modification involves the addition of hydrogen peroxide to the filtrate after the initial acidification. This addition oxidizes ferrous iron to ferric iron, which precipitates as Fe(OH)₃ during titration, which results in a more accurate NP value (Skousen et al. 1997). A fizzless version of this method has been standardized in ASTM E 1915-07a, using two ranges of acid additions and a pH and carbonate carbon check used to select the proper acid addition range. The ASTM version of the method recognizes that negative titration results may be due to acidic salts in the sample.

BC (British Columbia) research initial test. The AP determination in this test was originally based on total sulfur, but it now relies on sulfide sulfur. NP is determined using sulfuric acid (1.0 N) to titrate the sample to pH 3.5. It is a fairly quick and inexpensive test (Bruynesteyn and Hackl 1984).

Lapakko test. The Lapakko test (NP, pH 6.0) is the same as the BC research initial test method except for the strength of acid and the endpoint used. Specifically, a slurry comprised of 10 g solid and 100 mL deionized water is titrated with 1 N 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 1992). The method differs significantly from the previously mentioned tests in that the "digestion" occurs at pH 6 rather than in a more acidic environment.

Net carbonate value test. The net carbonate value (NCV) test was developed for use in Nevada in the Carlin Trend where a considerable amount of semiquantitative X-ray diffraction (XRD) data was available, including carbonates and sulfide minerals, which allowed for the development of a test that is expressed as % CO $_2$ as the measure of NP. This test has been standardized as ASTM E-1915-07a. The test uses combustion infrared absorption spectrometry for carbonate

(NP) and sulfide (AP) as well as a fizzless neutralization potential method to account for metal carbonate corrections.

The neutralization potential, based on carbonate carbon content, is determined by the difference between total carbon and carbon after reaction with HCl, or alternatively by the residual C content following pyrolysis at 550° C for 1 hour. Pyrolysis loss carbon should only be used to estimate ANP in the absence of significant carbonate minerals as their presence tends to bias results toward lower values. AGP is determined by the difference between total sulfur and sulfur after pyrolysis at 550° C to liberate sulfide and organic sulfur. The results of the test are expressed as NCV values as % CO₂ and range from <-5 (highly acidic) to >+5 (highly basic) with allowances for samples that are either neutral or inert (Bucknam 1997). NCV utilizes essentially the same information as the Sobek test but uses different analytical techniques. NCVs can be converted to ABA or net neutralization potential by multiplying the NCV value by 22.7.

Verification Tests

Biological acid production potential test (ASTM E 1915-07a). The biological acid production potential (BAPP) test is also known as the BC research confirmation test. This test and the Coastech test (discussed in the following subsection) are kinetic tests only in the sense that they are typically run for a longer period of time than static tests. They have generally been used as confirmatory tests for static testing, but they do not provide the level of information on kinetics that a humidity cell does.

This test is generally similar to the BC research initial test, except the sample is inoculated with bacteria (Acidithiobacillus ferrooxidans) or others). The test provides an assessment of the potential for biological oxidation or catalysis of sulfides. The test is a kinetic procedure used to determine if bacteria can catalyze reactions to maintain acidic conditions in artificially acidified slurry of pulverized rock and distilled water, following further additions of pulverized rock into the slurry (Bruynesteyn and Duncan 1979). The disadvantage of this test is the inoculation with bacteria, which may not have been well characterized or controlled, thereby introducing an added experimental variable and complexity that complicate comparisons between laboratories. Although humidity cell tests are more widely used to evaluate rates of acidification, the BAPP test, as corrected using the net acid generation (H_2O_2 [hydrogen peroxide]) test also provides useful information. This test normally runs for up to 10 weeks. Also, the test process itself, through the addition of acid to favor the bacteria, can tend to overestimate acid potential and also introduce reproducibility issues. It has been widely used in the past in Canada but recently has been used less frequently because of inconsistent results (R. Vos, personal communication, 2011).

Coastech modified biological oxidation test. This test is similar to the BC research confirmation test, but it includes an addition of NaOH at the conclusion of the test if the final test pH is below 3.5, and then an additional pH reading after 1 hour to reduce the bias toward acid classification (Lawrence and Sadeghnobari 1986).

Other Static Tests

Total inorganic carbon (TIC) test. This method relies on a calculation of total carbon minus carbon after HCl digestion to arrive at TIC (Hillebrand et al. 1953). This method suffers from the same issues as other methods that evaluate NP based on carbonates: potentially underestimating NP available from noncarbonate minerals and overestimating NP if siderite or other iron or manganese carbonates are present. It is not in common use in North America.

Chromium reducible sulfur test. This method is common in Australia (Sullivan et al. 1999). It only measures sulfide minerals and is considered effective for measuring low-level sulfur

concentrations and has been used for acid-sulfate soils. Little linkage exists between this test and the more common static tests. It is not in common use in North America.

Total actual acidity test. This method uses a titration of potassium chloride (KCl) extract to a pH of 5.5 with NaOH. It can measure actual acidity in oxidized samples and has also been used for acid-sulfate soils (Dent and Bowman 1996). It is not in common use in North America.

Acid concentration present leach test. This method is useful for verifying the amount of acidity present for estimating lime amendment when the acid neutralization potential acidity titration result is negative or the paste pH is below 7. The acid concentration present leach test is slightly more involved than a standard paste pH but supplies an estimate of acidity present rather than simply pH (Bucknam et al. 2003). A mixture of 20.0 g rock (-200 mesh) and 50 mL deionized water is agitated, the initial pH is recorded, and the mixture is titrated to pH 7 with NaOH. The volume of NaOH needed to titrate to this value is used to calculate the amount of acidity in units of % CO₂. (This can be more conveniently expressed in units of CaCO₃ to facilitate quantifying alkalinity addition required.) A negative value indicates acidity.

SHORT-TERM LEACH TESTS

This section discusses tests that add some level of solute/chemical interaction in order to evaluate more than a simple chemical balance as the ABA does to estimate ARD potential. These tests range from those that have been specifically developed and modified to be used in waste rock characterization, such as the net acid generation test, to tests like the synthetic precipitation leaching procedure that were originally developed for other "waste" characterization but have nonetheless been applied to mine-waste characterization and ARD evaluations.

Net Acid Generation Test

The net acid generation (NAG) test uses hydrogen peroxide (H_2O_2) as an oxidant to evaluate the net capacity for acid production or neutralization (Miller et al. 1997). This test is based on the principle that hydrogen peroxide accelerates the oxidation of iron sulfide minerals. The acid produced by this reaction dissolves neutralizing minerals present, and the net result of the acid production and neutralization can be measured directly. Unlike ABA or other static tests, this test does not require sulfur or carbonate determinations and can, therefore, be readily conducted in a field laboratory.

A sample of material is oxidized using H_2O_2 for a specific time period, generally until oxidation ceases. The sample is then titrated back to a specific pH, and the net acid production in kilograms per metric ton (kg/t) is calculated from the titration. Samples that report a final pH less than 4.5 are generally considered to be acid generating. Variations on the NAG test (Miller et al. 1997; AMIRA International 2002; Price 2009) include

- · Sequential NAG test for samples with high sulfide contents, in which the NAG test is repeated until pH exceeds 4.5;
- Modified NAG test, which uses extended boiling and analyzes the digestate for S, Ca, Mg, and total organic carbon; and
- · Kinetic NAG test, which monitors temperature, pH, and electrical conductivity during the test.

ASTM E 1915-09 has a version of the method in the appendix that is used to correct for false positive BAPP results. Samples that may include organic materials can complicate interpretation as H_2O_2 reacts vigorously with organics.

The NAG test is popular in Australia (Rumble and Miller 2000) and has been used for operational screening of waste material and for identification of samples that may require kinetic testing. Its popularity is due to the relative simplicity, short turnaround time, and cost (\$40–\$70). Linked with other static and kinetic tests, it can be a beneficial part of a prediction/characterization or waste management program.

Other Short-Term Leach Tests

Short-term leach tests can measure readily soluble constituents in a sample. These tests may be a regulatory requirement and are all variations on a central theme. A sample of specified mass is rinsed for a specified time, with a prescribed solution, depending on the test. The solutions produced in these tests are not the result of long-term dissolution of the samples, so these tests are not regarded as a measure of ARD potential. They can help point to potential metal/trace element release issues, particularly for metals and metalloids that may be soluble in the circum-neutral range (zinc, cadmium, selenium, and arsenic). Table 4.1 includes many of the common short-term leach tests. Chapter 5 includes a more detailed discussion of several of the tests, although these tests will not be covered in detail in this handbook because they are generally not appropriate for detailed prediction and characterization programs, even though their use may be required by regulation in some states (e.g., meteoric water mobility procedure in Nevada).

FIELD LEACH TESTS

Field tests can generally be performed at the mine site by technicians or field staff. They typically do not require sophisticated instruments or techniques and can be completed either in the field or with a quick visit to the lab. Results are generally obtained in less than one day and give an immediate report on soluble salts.

Paste pH

Paste pH is probably the most commonly used field test and is a simple test used to assess the presence of soluble acid salts on mine wastes. Most methods use a 1:1 weight ratio of distilled water to air dried solids, with measurements of the mixture made by a pH meter calibrated at pH 4 and 7. Sample mass and equilibration time of the water–solids mixture prior to pH measurement vary among methods (Hammarstrom and Smith 2000). The procedure described by Mine Environment Neutral Drainage (MEND 1990) determines the pH of a mixture of 10 g rock (–60 mesh) and at least 5 mL distilled water (water addition is adequate to saturate, but not cover, the rock). Paste pH and the other field tests discussed in the following sections test the solubility of stored sulfate or sulfo-salt minerals reaction products, which are essentially stored acidity in the sample, resulting from prior acid generation. These minerals can be missed by some of the commonly used static tests. Paste pH does not provide any information on future acid production from unweathered minerals in the sample. It indicates that this stored acidity is available and thus can be very useful as part of a detailed waste characterization program (Shaw 2005; Williams et al. 2009) and is also useful for interpreting kinetic test results. In some cases, paste pH is considered a standard part of ABA procedures (Price 2009).

USGS Field Leach Test

The U.S. Geological Survey (USGS) field leach test (FLT) was developed by USGS specifically for mine waste (USGS 2005; Hageman 2007). The FLT dictates the potential for readily soluble salts, including those which are acidic and/or contain trace metals, to be dissolved from minewaste dumps. It correlates well with standard paste pH testing but has the advantage of producing

TABLE 4.1 Common short-term leach tests

Test Method and Description	Use in Geochemical Characterization and Water Quality Prediction	Advantages	Limitations
Synthetic Precipitation Leaching Procedure (SPLP) • USEPA Method 1312 (USEPA 1994) • 20:1 Solution-to-solid ratio • Deionized water or dilute sulfuric/nitric acid to pH 4.2 or 5.0 • <9.5 mm • 18 ± 2 hours • Variant: Standard Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure (ASTM D 6234)	All Methods: Measures readily soluble constituents of mine and process wastes.	All Methods: Provides indication of short-term leaching of soluble constituents. Identifies readily dissolvable constituents.	All Methods: Provides no information on transient processes and long-term conditions. Only simulates short-term interaction. High liquid-to-solid ratio may underestimate leachability. Grain size reduction may increase reactivity.
Toxicity Characteristic Leaching Procedure (TCLP) • USEPA Method 1311 (USEPA 1992) • 20:1 Solution-to-solid ratio • Acetic acid/acetate buffer • <9.5 mm • 18 ± 2 hours	Used to determine if waste is hazardous under the Resource Conservation and Recovery Act of 1976 Intended to simulate municipal landfill containing organic wastes	Applicable standards available.	Use of acetic acid/acetate buffers not appropriate for mining applications. Short list of metals evaluated.
Meteoric Water Mobility Procedure (MWMP) • 1:1 Solution-to-solid ratio • Reagent-grade water • <2 inches • <48 hours	Same as for SPLP. Used in Nevada following implementation of their metal mine-waste characterization regulations.	Quasi-dynamic test. More realistic than SPLP due to higher solid-to-solution ratio, longer duration, and coarser material. Applicable standards available.	Weaker lixiviant than acidified SPLP.
California Waste Extraction Test (CalWET) • 10:1 Solution-to-solid ratio • Dilute sodium citrate solution • < 2 mm	Intended to simulate a municipal landfill containing organic wastes. Primarily used in California.	Lower liquid-to-solid ratio and longer test duration than SPLP and TCLP. Applicable standards available.	Use of sodium citrate not appropriate for mining applications.

Source: Adapted from INAP 2009a.

48 hours

a volume of leachate, which can be used for additional analyses. The test also correlates well with the U.S. Environmental Protection Agency's (USEPA's) Method 1312 synthetic precipitation leaching procedure test, which uses a different extraction method (USEPA 1994). The FLT can be used for screening by qualitatively characterizing waste-rock dump material. The Colorado Division of Minerals and Geology has a similar test that uses a more concentrated mix and produces less leachate for testing (Herron et al. 2001).

Wall Washing

Wall washing involves washing and collecting water from a small, isolated portion of a pit wall. There is no standardized protocol, but the goal is to normalize the surface area rinsed to a known volume of rinsate; for example, 1 m² of rinsed surface (1.5 m³ of surface area on joints, fractures, and outcrops) rinsed with 1 L collected from an area isolated with tape or plastic sheeting (INAP 2009a). One problem with this approach is that the rock surface area is highly variable due to fracture and joint density changes, which can create significant differences in rock—water interactions. Some advances have been made in correcting for this uncertainty through application of photogrammetric methods.

KINETIC TEST METHODS

Kinetic tests evaluate the rate of chemical change in weathering parameters (such as sulfide oxidation and metal release) in response to controlled oxygen exposure and leaching over time. Bucknam et al. (2009) indicate that kinetic tests are dissolution tests conducted either in the laboratory or field for relatively long periods (months to years), typically under oxidizing conditions. Although there are several types of kinetic tests, ASTM D 5744-96 and subsequent revisions (D 5744-07e1, D 5744-13) provide guidance that is applicable to these tests in general. These methods are reviewed in greater detail in the following paragraphs.

The following considerations are key for any kinetic test: clearly defined test objectives; appropriate methods of testing; preliminary and post-testing analysis of solids, including static and mineralogical analyses; appropriate test duration; and a well-understood goal for application of results.

Kinetic tests are more expensive than virtually any of the static tests and require dedicated laboratory or field space, and skilled laboratory technicians and evaluators. Kinetic tests subject mine-waste samples to dissolution, typically under oxidizing conditions, in either the laboratory or field. The ore and waste material used in kinetic tests should be characterized in detail before and after testing. Kinetic tests can provide information on acid generation and solute leaching from the material being tested.

Kinetic tests can be used to verify static test predictions of AGP and have been used to estimate long-term water quality for ore and waste materials. The leachate from the kinetic test cycle is collected and analyzed, most typically for ${\rm SO_4}$ (sulfate) as an indicator of sulfide dissolution; pH for related acid generation/neutralization; and acidity, alkalinity, calcium, magnesium, and associated metals as an indicator of potential drainage quality. These tests can be designed to address several objectives regarding mine-waste and processing-waste dissolution: to predict drainage pH, to determine rates of chemical release, and to assess the relative potentials of mine wastes to influence water quality under controlled conditions. Additional descriptions and assessments of kinetic test methods are provided by Lawrence (1990), MEND (1991, 2000), Morin and Hutt (1997), and Lapakko (1993, 2003).

Humidity Cell Tests

Humidity cell (HC) tests were standardized as ASTM D 5744-96 and revised as ASTM D 5744-07e1 (Bucknam et al. 2009). Repeatability and reproducibility assessments of D 5744-96 (Lapakko and White 2000; White and Lapakko 2000) have also been summarized in an expanded "Precision and Bias" section published in the method's most recent revision as ASTM D 5744-13. The standard test method "...calls for the weekly leaching of a well-characterized solid material sample (weighing at least 1000-g), with water of specified purity, and the collection and chemical characterization of the resulting leachate" (ASTM D 5744-13).

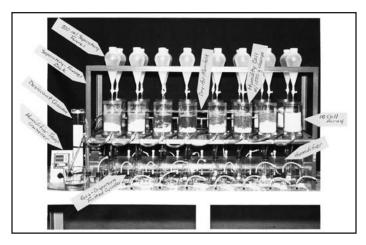
The sample mineralogy and petrology inform interpretation of HC test results. Consequently, samples tested should be thoroughly characterized before and after the test (ASTM D 5744-07e1, D 5744-13; Diehl et al. 2003). This is particularly important for samples with more complex mineralogy, as it may be difficult to accurately interpret the HC test results in the absence of detailed characterization. ASTM D 5744-13 addresses characterization of particle size distribution, chemistry, mineralogy, and petrology of the bulk samples to be tested. Determination of sulfide mineral and carbonate mineral contents, and degrees of liberation as a function of particle size is also recommended (ASTM D 5744-13). The geologic and structural characteristics of mine area will be essential to application of the HC data to the operational setting. This applies to other types of kinetic tests as well as HC tests.

The revised method contains two protocol choices, Options A and B. Under Option A, the cycle is three days of dry air (<10% humidity) followed by three days of humid air (>95% humidity) pumped through the sample (Figure 4.2). Under Option B, each cell is stored for six days under conditions of controlled and relatively constant temperature and humidity, and oxygen is passively supplied to the sample by diffusion (and possibly advection) of ambient air rather than by pumping (Figure 4.3). For both options, the cells are weighed during the weekly cycle to determine water retention. This is particularly important to ensure that cells are not drying completely during the dry air cycle of Option A. The flood leach is the preferred method of water addition for both options (Figure 4.3).

The method recommends that leachate be evaluated on a scheduled basis for diagnostic ARD parameters typically including pH, Eh (reduction potential), specific conductivity (SC), Fe, SO₄, alkalinity, and acidity (ASTM D 5744-13). Determination of Ca, Mg, Na, and K can be valuable in assessing acid depletion of neutralization, providing insight into the mineral phases and providing neutralization and their respective dissolution. Selected additional metals can also be included, depending on the specific requirements or test objectives. There may be other requirements for evaluating the leachate based on the site-specific/deposit mineralogy or metals of concern. The method recognizes that if changes in leachate chemistry are slow, it may not be necessary to analyze metals every cycle. "Test duration is determined by the user's objectives of the test" (ASTM D 5744-13). Additional discussion of test duration is presented in Chapter 5.

ASTM D 5744-07e1 and D 5744-13 eliminated the bacterial inoculation of samples based on unpublished U.S. Bureau of Mines reports (R.R. Corwin, personal communication, 1995) and practical experience from practitioners of kinetic testing (Morin and Hutt 1997, 2004). Recent work suggests that the elimination of bacterial inoculation should be an area of continued study and may need to be reconsidered (L.B. Kirk, personal communication, 2011).

It is important to understand that HC tests should *not* be considered a "one stop" test for all solute release and kinetic reaction questions. The HC tests were designed as assessments of the intrinsic sulfide oxidation rate, although they are broadly applied for many other purposes. These tests are simply another tool in the toolbox, and the results of any HC tests must be considered



Source: Reprinted with permission from ASTM D 5744-13, Standard Test Method for Laboratory Weathering of Solid Materials Using a Humidity Cell, © ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. A copy of the complete standard may be obtained from ASTM International, www.astm.org.

FIGURE 4.2 ASTM D 5744-13 Option A humidity cell array



Courtesy of the Minnesota Department of Natural Resources.

FIGURE 4.3 ASTM D 5744-13 Option B flood leach phase

in conjunction with all the other static, short-term leach tests, as well as kinetic and mineralogical work that must be a part of comprehensive rock characterization.

Pre- and post-testing characterization. In most cases, kinetic testing is initiated following completion of initial mineralogic, whole rock, and static test work, which indicates the need for further kinetic analysis of ARD and solute release potential. Solids analysis guidance provided in ASTM D 5744-13 is also relevant to other kinetic tests. The drainage quality in these tests is dependent on solid-phase composition, and diligent characterization is required to interpret drainage chemistry and extrapolate results to the field. Given the cost of conducting the dissolution tests, it makes economic sense to have a detailed description of the solid that is generating

the observed water quality. Furthermore, it is critical that the relationship between solid-phase and drainage quality in the laboratory tests be well understood given (1) the small number and mass of samples subjected to testing relative to the enormous masses and variability in the field and (2) the short duration of laboratory tests relative to the long period of concern in the field. This understanding requires detailed insight on the solid-phase characteristics that control the drainage quality composition.

Timeline. Compared to virtually any of the "static" tests, kinetic tests are run for longer time frames. The duration and termination of kinetic tests has historically been contentious and remains a focus of ongoing discussion. There is no commonly agreed-upon test duration for kinetic tests, and ASTM D 5744-13 indicates the duration is dependent on the test objectives. An appropriate test duration will depend on a wide variety of factors, which are discussed at length in Chapter 5 and Appendices K and L of this handbook. A longer test duration can increase the level of certainty with respect to the various parameters evaluated, which may be an important consideration for questionable assumptions about material's long-term behaviors under site-specific conditions. Criteria for terminating tests should be considered prior to test initiation, as discussed further in Chapter 5. The timeline and intended application for risk management are inherently linked. If the resulting data indicate that a material should be managed as potentially acid generating, relative to the management objectives, regardless of whether the geochemistry is fully resolved from an analytical standpoint, it may be appropriate to terminate a test on a shorter timeline than may be otherwise deemed appropriate, as discussed in the framework provided in Appendix L.

Application. Kinetic tests provide empirical solute release rates under conditions unique to an individual rock (multiple mineral, variably textured, etc.) and testing protocol. Although the drainage quality in kinetic tests does not typically represent that expected under field conditions (ASTM D 5744-13), interpretation of results can be applied to prediction of water quality impacts. The results of kinetic testing can be applied to identify mine waste that requires selective handling during operation. Mine-waste management approaches, such as dump design or cover construction strategies, can then be tailored to the predicted solute release associated with a specific mine waste. Long-term predictions of water quality should consider sample mineralogy, static test results, short-term leach data, and kinetic test results in the context of their mutual method limitations.

Application of laboratory HC results might require scaling to estimate rates of solute release in the field for purposes of assessing potential impacts or sizing treatment facilities. Scaling of laboratory results for application to field sites is not a trivial exercise, and there is no widely accepted method for this extrapolation. Among other requirements, it should have a sound conceptual basis, respect scientific principles and empirical data, and yield results expressed as a probability distribution as opposed to singular values (Lapakko 2010). Extrapolation of laboratory results to field conditions must consider the differences between materials and reaction conditions in the two settings. For example, particle size, reaction temperature, water added to rock ratio, timing of solute release intervals, and extent of rock contacted should be among the variables considered.

Geochemical equilibria (solubility) must also be assessed, for example, based on geochemical modeling and, where possible, using empirical examples at multiple scales. Empirical comparative analysis of laboratory and field kinetic test results (e.g., HC and field test-pile weathering tests of the same lithology) will help to quantify a relationship between laboratory and field release rates (Lapakko and Antonson 1994; White et al. 2002). This can be viewed as a modeling exercise, and guidance on such efforts is provided by the National Research Council (2007). Other

applications of results are feasible, and these functions must be considered and agreed upon among stakeholders during design of the kinetic test program.

Column Tests

Like HC tests, column tests are also leach tests, but they are not standardized (INAP 2009a) and can have variable dimensions, leaching/drying cycles, and solution-to-solid ratio. Therefore, they are more flexible in terms of modifying test procedures to more closely resemble site conditions. Column tests have been used in attempts to simulate a wide variety of geologic situations and to investigate numerous conditions and phenomena, including those more commonly considered in batch tests. Column tests of various sizes have been used to evaluate the dissolution of mine wastes. Ordinary batch tests typically address only static physicochemical processes, (e.g., sorption, desorption, water/solid/gas equilibrium), although they can be operated in a timedependent mode to study chemical kinetics. Column tests, on the other hand, are inherently dynamic and add the complexities of liquid and gaseous fluid flow in porous media and may include biological agents found in soil or waste profiles. Columns can provide dynamic data in the form of time- and/or volume-dependent chemical characteristics of column effluent or variations in chemical conditions along the flow path of the column. Geologic material in a column may be packed disaggregated soil or rock (most common) or it can be soils, tails, or waste rock collected in situ. The subject is nicely summarized by Zachara and Streile (1991). Several alternatives have been evaluated by Hood and Oertel (1984), Bruynesteyn and Hackl (1984), and Bradham and Caruccio (1990).

In general, columns may be operated in one of two modes: saturated or unsaturated. Saturated columns are best operated as an upflow apparatus in which all pore space is occupied by the experimental fluid (typically, clean or amended water) and all air is expelled. This situation simulates groundwater flow, allows the most controlled and best understood fluid dynamics, and can foster anoxic conditions. Theories for interpreting saturated columns are well established and widely available. Another commonly attempted column simulation is that of rainfall infiltrating the vadose (unsaturated) soil profile. This seemingly simple experiment is accomplished in a downflow apparatus with finite fluid application rates but is much more difficult to design, operate, and understand. Before contemplating such experiments, one should consult van Genuchten and Wierenga (1986).

The intended use for data from column experiments must be carefully considered before the decision is made to use columns in the first place, and prior to selection of the appropriate type of column design and mode of operation. Unfortunately, there are no formal standards for column design, largely because of the inherent complexities associated with materials and objectives. There are loosely defined guidance rules for dimensions based on grain size and diameter/length ratios, but the variability in the literature is evidence of uncertainty about the influence of these design parameters. Although well-constrained column tests are costly (in terms of time, materials, required operating skills, and analytical requirements) and complicated in design, they may also be as simple as illustrated by Figure 4.4. Of course, the quality and usefulness of the data varies considerably.

Disadvantages cited by Bradham and Caruccio (1990) for the technique are the long time required, relatively high cost, the large sample volume required, and the potential for channelization in the columns. J.J. Renton (personal communication, 1991) further cited problems with reproducibility of column test results. The relative lack of recent literature citations suggests that inherent reproducibility issues have limited the recent use of this experimental approach.



Courtesy of Inter-Mountain Labs Inc.

FIGURE 4.4 Option B humidity cell test for flotation tailings

The wide variety of designs, poor documentation of important operating parameters, and common failure to acknowledge variables in the literature is evidence of the risks associated with conducting and interpreting column experiments, not to mention relating the results to field conditions. This does not mean they have no purpose or potential value, but they are very complex and should not be undertaken lightly.

Field Test Methods

Field tests can range from simple wall washing (INAP 2009a) to tubs, barrels, and test piles containing representative mine waste or mine-waste piles designed and constructed to collect all drainage (Figure 4.5), to sophisticated monitoring systems on operational scale disposal facilities. Such systems can include remote weather data collection, lysimeters for collecting leachate samples, and temperature and gas composition probes on constructed waste rock piles (Figure 4.6).

Tubs. Caruccio and Geidel (1986) used 75-L plastic tubs containing each of three size fractions, ranging from 2.5 to 10 cm, to examine the dissolution of pyrite-bearing rock. The tubs were placed 1 m above the ground surface in an open field. For two years, drainage from the tubs was quantified and analyzed for specific conductance, pH, acidity, and sulfate. The drainage volume and chemical concentrations were used to determine rates of mass release per unit mass waste. The results from the tubs were used to represent actual field weathering, to which results from laboratory test results were compared.

Barrels. Renton et al. (1988) used 35-gal (132-L) plastic barrels and a 50-ft² (4.6 m²) plot 4 ft (1.2 m) deep to examine the weathering of coal cleaning plant waste. A top size of 1.5 in. (3.8 cm) was used in the barrels and a corresponding value of 3 in. (7.6 cm) was used for particles in the plot test. The barrel test results were used to extrapolate bench-scale elevated temperature test results to field scale, using a first-order reaction mathematical model. The barrel tests provided an accurate intermediate between the bench tests and the large-scale field plot.

TestpPiles. Eger and Lapakko (1985) examined the drainage quantity and quality from six test stockpiles containing 820 to 1,300 metric tons of low-grade copper-nickel material from the Duluth Complex in northeastern Minnesota. During their construction in 1977, each pile was



Courtesy of the Minnesota Department of Natural Resources.

FIGURE 4.5 Constructed field tests of Archean greenstone waste rock



Source: McLemore et al. 2011.

FIGURE 4.6 Monitoring site on a constructed waste rock dump in Questa, New Mexico

underlain by an impervious membrane to facilitate drainage collection. The approximate major mineral content of the rock was 60% plagioclase, 15% pyroxenes, and 10% olivine. The sulfur content of the piles varied from 0.6% to 1.4%, reflecting the variation in pyrrhotite content in the piles. Flow was quantified and water quality analyzed at the site. By conducting small-scale field tests on well-characterized samples representative of operational scale wastes, laboratory experiments can be extrapolated to operational scale (Lapakko and Antonson 1994). The laboratory data was scaled to field conditions based on the particle size distribution, chemical composition, and water quality data from the test piles in the field.



Source: McLemore et al. 2011

FIGURE 4.7 Field evaluation of a deconstructed waste rock dump in Questa, New Mexico

As with the simulated environment experiment, the cost, sample mass required, and duration of the experiment are problems in examining the behavior of a wide variety of wastes during the period between mine proposal and permitting. The initial construction cost is high, but subsequent sampling and maintenance costs would be expected to be less than those of the simulated environment test since environmental control is not required. These tests can provide information on the quantity of drainage generated and the effect of waste composition on water quality, and they allow examination of the effectiveness of mitigation techniques (Murray and Okuhara 1980; Eger et al. 1980). Since this information is generated under conditions that most closely approximate those of the actual mine-waste disposal, this type of test can be of great benefit during mine development. By starting such tests as soon as an adequate waste mass is available, results derived can aid drainage quality projection and mine-waste reclamation design.

Additional information at an operational scale can be obtained by visual examination and sample collection and analysis during deconstruction (Figure 4.7, McLemore et al. 2011).

It can be extremely useful to have ongoing field-scale tests that can be linked with static and kinetic tests. These tests can be relatively inexpensive to set up but do require a commitment of resources and staff to conduct the necessary sampling and detailed analysis that can make them a useful reference point for site-specific static and kinetic testing. Field-scale tests can be particularly useful in correlating kinetic tests to actual site-specific reaction and metal release rates, as discussed later in Chapter 5.

DATA MANAGEMENT

Mining projects from exploration through mine development create vast amounts of data covering the entire range of information to support the investment, design, development, operations, and closure of a mine. This data should be collected using accepted standards, then maintained and updated in a secure electronic format. Increasingly, databases are constructed using a geographic information system format, which ties data to a specific three-dimensional (3-D) point in space. This 3-D format may be specific to the mine or it may use other common formats: UTM

(Universal Transverse Mercator), latitude/longitude, and so on. Data developed during waste characterization, as well as other data developed in support of exploration, development, operations, and closure, should conform to accepted information technology standards for data input, storage, maintenance, and backup.

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CHAPTER 5

Evaluation

The results of geochemical testing should be interpreted in context of site-specific conditions and risk management goals for each individual project. These goals will depend on the site conceptual model, material characteristics (geology, mineralogy, alteration, and volumes), and results of geochemical testing, as discussed in Figures 1.4, 1.5 (Chapter 1), and 4.7 (Chapter 4).

STATIC TESTS

Sobek and Modified Sobek Static Tests

The most commonly used static tests are the Sobek and modified Sobek acid–base accounting (ABA) tests. These tests balance the acid potential (AP), sometimes referred to as acid generation potential or AGP, with the neutralization potential (NP) to produce a net neutralization potential (NNP). This number is sometimes simply represented as "ABA." These values are calculated in a stoichiometric convention so they can be balanced against each other and a value assigned to the sample. In North America, this convention is generally expressed in tons or metric tons (i.e., $CaCO_3/kt$ [kiloton]) and in Australia, metric tons of H_2SO_4/kt of mine waste. North American convention uses negative ABA numbers, sometimes expressed as either net acid production potential (NAPP) or NNP, to represent samples with greater acid generation than neutralization potential. Australian convention, because it is represented in metric tons H_2SO_4/kt , uses positive numbers to represent samples with higher AGP.

Stoichiometrically, the standard convention is to multiply the weight percentage of contained sulfur by 31.25 (based on the assumption that all available sulfide is, in fact, pyrite) to obtain the AP in units of kg CaCO₃ equivalent per metric ton (or t/kt). This number is then subtracted from the corresponding value assigned to the NP. Negative numbers mean that the sample has more capacity to produce acid than to neutralize acid, and positive numbers mean the opposite. Generally, numbers below –20 NNP are considered to indicate a sample is strongly acidic and very likely to generate acid. Numbers greater than +20 NNP are considered basic and unlikely to generate acid. Samples between –20 and +20 NNP are considered to represent a zone of uncertainty requiring additional detailed evaluation in order to make a determination about the likelihood of acid production.

Another means of evaluating the likelihood of acid generation is to use the ratio of NP to AP (NP/AP) to represent the likelihood of acid production. This value is sometimes referred to as the neutralization potential ratio or NPR; this is also sometimes referred to as the net potential ratio. NPR values varying from 1.2:1 to as high as 4:1—meaning the sample has 1.2 times or 4 times as much NP as AP—have been used as threshold values indicative of material that is unlikely to produce acid. The range of values is provided because this approach is an oversimplification that overlooks the fundamental importance of an understanding of the geology and mineralogy of a sample.

Use of NPR for samples with low NP and AP should be avoided, as the low values can cause the NPR to vary nonsensically depending on minor changes in either value. A sample that has no

sulfur and no carbonate—a barren quartzite, for example—would have an NNP of 0 and an NPR of 0 (temporarily allowing for division by 0), and thus would fail the criteria discussed previously, when in fact the sample has no possibility of producing acid. This highlights the importance of the mineralogy being a key to understanding static test results; if one does not understand the mineralogy, he or she probably does not understand static test results.

The Sobek and modified Sobek tests may not accurately represent either the AP or NP in the samples analyzed because of mineralogical complexities that the testing protocol cannot accurately capture. Anecdotal evidence suggests that adaptations to the modified Sobek test which attempt to address some of these inaccuracies through changes in the digestion sequence, temperature, or type of leach can lead to additional reproducibility issues between labs (Schafer 2001; Fisher and Brown 1994). This only highlights what has been repeatedly stressed: a detailed understanding of the mineralogy is absolutely essential to evaluating static tests and their interpretation.

White and others (1997, 1999) concluded that static tests conducted on a variety of metal-mine-waste samples showed that NP variability for a given sample was (1) most strongly influenced by differences in sample particle size, amount of acid addition, and back-titration endpoint; (2) influenced in one test by digestion duration; and (3) virtually unaffected by acid type and temperature of digestion. It was also shown that extent of influence on NP variability is a function of sample mineralogy.

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). This method will underestimate the AP if acid-producing sulfate minerals such as alunite $\mathrm{KAl_3(SO_4)_2(OH)_6}$, jarosite $\mathrm{KFe_3(SO_4)_2(OH)_6}$, copiapite (Fe, Mg)Fe₄(SO₄)₆(OH)₂, among others are present.

Siderite Correction

As discussed in Chapter 4, siderite is less common as an accessory mineral in metallic mineral deposits. If detailed mineralogy indicates siderite may be present, this test is one method of correcting the NP values when siderite is present.

BC (British Columbia) Research Initial Test, Lapakko Test

If the acid consumption value (in kilograms of acid per metric ton of sample) exceeds the acid-producing potential (kg/t), the sample is not predicted to be a source of acid mine drainage and no additional work is necessary. If the acid consumption is less than the acid production potential calculated from the sulfur content or the difference is marginal, the possibility of acid mine water production exists and the BC research confirmation test is conducted. A pH value of 3.5 is chosen for titration. The Lapakko test uses a pH value of 6.0, as this is a common water quality criterion. The objective of the test is to determine how much acid the sample can neutralize and maintain a pH of 3.5 or 6.0 (Lapakko test).

Net Carbonate Value Test

The net carbonate value (NCV) test was developed by Newmont Metallurgical Services (NMS) and has been used extensively in Nevada's Carlin Trend. It has been standardized as ASTM E 1915. NCV has some of the same issues as other static tests, and these are recognized in Newmont's Standard ARD Waste Rock Evaluation Methods guide (NMS 2003): "There is no one method that will properly estimate all sulfide minerals, so it is necessary that NCV analyses be backed up by mineralogy and other confirmation testing in order to select the best method for characterization of each deposit during waste management operations."

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TABLE 5.1 Standard NCV val	TARI I	- 5 1	Standard	NCV	values
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Classification	Criteria for Classification
Highly acidic (HA)	NCV ≤ -5
Acidic (A)	$-5\% < NCV \le -1\% CO_2$
Slightly acidic (SA)	$-1 < NCV \le -0.1\% CO_2$
Neutral (N)	$-0.1 < NCV < 0.1$ and $(ANP \ge 0.1 \text{ or } AGP \le -0.1)$
Inert (I)	-0.1 < NCV < 0.1 and (ANP < 0.1 and AGP > -0.1)
Slightly basic (SB)	0.1 ≤ NCV < 1
Basic (B)	1 ≤ NCV < 5
Highly basic (HB)	5 ≤ NCV

Source: Table first published in *World Gold 2011* by the Canadian Institute of Mining, Metallurgy and Petroleum (CIM), available at www.cim.org/en/Publications-and-Technical-Resources/Publications/Books/2011/10/WorldGold2011hardcoverbookandCD.

As discussed in Chapter 4, NCV values are represented as % CO₂ in the convention for internal assessments by Newmont shown in Table 5.1.

Ore or waste rock from a particular lithology or geological classification will likely overlap several of these NCV classifications. These classifications are used by NMS and others as a guide for selection of material to perform more in-depth testing that covers the range of the acid–base characteristics, such as kinetic tests and short-term leaching tests for each of the major geological NCV classifications. Based on those results, acid-neutralizing potential (ANP), AGP, or NCV cut-off criteria can be selected on a site-specific basis for properly routing the material during active mining. The eight NCV classes based on the % CO₂ as noted in Table 5.1 are not designed to be an independent criterion for waste classification.

Other considerations for the evaluation of static tests include use of a spreadsheet or table that includes a variety of the tests performed at a site, including kinetic and short-term leach tests, so that a comparison can be made showing which mix of tests is the most effective for characterizing waste as shown in Table 5.2.

SHORT-TERM LEACH TESTS

Net Acid Generation Test

The standard (single addition) net acid generation (NAG) test evaluation relies simply on the final pH after reaction with hydrogen peroxide (H_2O_2) for a set period of time (AMIRA International 2002). A pH value of less than 4.5 is considered to be acid producing (see Table 5.2). Following this determination, the sample is titrated back to a pH of 7.0, and the volume of acid used for this titration can be represented in kilograms per metric ton (metric tons per kiloton) $CaCO_3$.

Because samples with sulfide content greater than 1% may not react completely using the standard NAG test protocol, it may be necessary to use sequential NAG testing procedures to fully evaluate high-sulfide samples. Essentially, the sequential NAG test involves simply repeating the single addition NAG test process on the same sample. At the end of each stage, the sample is filtered and the solution is used for measurement of NAG pH and NAG capacity. The NAG test is then repeated on the solid residue. The cycle is repeated until such time that there is no further catalytic decomposition of the peroxide or when the NAG pH is greater than 4.5. The overall NAG capacity of the sample is then determined by summing the individual acid capacities from

TABLE 5.2 Comparison of static, kinetic, and quasi-kinetic (meteoric water mobility procedure, acid concentration present leach) test results

Sample Information	formation			Static Test Results	t Results			e Information Static Test Results Kinetic Test Results	t Results
		NCV Class	MWMP pH, s.u.	Modified ABA Ratio Class	Modified ABA NNP Class	Peroxide Acid Generation Class	ACPL Titration	Biological Acid Produc- tion Potential Class	Humidity Cell Class
Composite Number/Type	Material Type	NCV Value, % CO ₂	Δ pH, s.u. extract–initial	Modified ABA Ratio	Modified ABA NNP, TCaCO ₃ /kT	Final pH, s.u.	Acidity, % CO ₂	Final pH, s.u.	Final pH, s.u.
1 Pulp	Siltstone 1	Slightly acidic -0.54	8.16	Potentially acid generating 0.40:1	Potentially acid generating -22.2	Acid producing 2.86	0.00	Acid producing 3.18	Not acid producing 7.25
3 Pulp	Limestone	Basic 3.52	Not tested	Not potentially acid generating 3.55:1	Not potentially acid generating 48.0	Not acid producing 10.41	0.00	Not acid producing 7.36	Not tested
4 Pulp	Limestone	Basic 2.49	Not tested	Not potentially acid generating 41.46:1	Not potentially acid generating 52.7	Not acid producing 10.16	0.00	Not acid producing 7.35	Not tested
5 Pulp	Oxidized siltstone	Basic 2.81	Not tested	Not potentially acid generating 4.54:1	Uncertain 17.7	Not acid producing 6.7	Not tested	Not acid producing 4.08	Not tested
6 Pulp	Oxidized siltstone	Slightly basic 0.77	Not tested	Potentially acid generating 0.95:1	Uncertain -0.3	Not acid producing 8.28	0.00	Not acid producing 3.71	Not tested
16 Pulp	Oxidized siltstone	Slightly basic 0.8	Not tested	Not potentially acid generating 29.83:1	Uncertain 17.3	Not acid producing 10.8	0.00	Not acid producing 5.71	Not tested
34 Reject	Oxidized siltstone	Slightly basic 0.31	7.27 +1.4	Potentially acid generating 0.80:1	Uncertain -1.1	Not acid producing 7.8	0.00	Not acid producing 3.7	Not acid producing 6.45

Source: Tetra Tech 2007. ACPL = Annual Certified Parameter List; MWMP = meteoric water mobility procedure; s.u. = standard units.

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each stage. The number of stages required before a sample turns acidic provides a guide to the length of geochemical lag time (AMIRA International 2002).

The kinetic NAG test is the same as the single addition NAG test except that the temperature, pH, and sometimes electrical conductivity of the liquor are recorded. These recorded values may help to understand the behavior of the tested material in the field and in humidity cells (AMIRA International 2002).

The advantages of the NAG test include its simplicity, low cost, and quick results, which enable its operational use. The limitations of the test include the breakdown of hydrogen peroxide through various factors that can lead to inaccurate characterization of material; high sensitivity to organic material, which can distort the results; and the rapid oxidation caused by hydrogen peroxide, which can create unrealistic geochemistry, again leading to inaccurate characterization. These advantages and limitations are well documented by Shaw (2005), O'Shay et al. (1990), Day et al. (2003), and Morin and Hutt (1999) and in general highlight the importance of linking the NAG tests results to ABA values and detailed mineralogy, regardless of the NAG test protocol selected.

Other Short-Term Leach Tests

A wide variety of short-term leach tests commonly used in the United States are available to measure readily soluble constituents in a sample, such as the synthetic precipitation leaching procedure (SPLP, USEPA Method 1312 [USEPA 1994]), toxicity characteristic leaching procedure (TCLP, USEPA Method 1311 [USEPA 1992]), meteoric water mobility procedure (MWMP), and California waste extraction test (WET or CalWET) and are summarized in Table 4.1. Similar tests used historically, less frequently, or elsewhere in the world include the multiple extraction procedure (MEP), extraction procedure toxicity (EPTox), special waste extraction procedure (SWEP), ASTM D 3987-85, AS 4439-1997 (as cited in Ilyushechkin et al. 2012), physiologically based extraction procedure (PBEP), EN series (Europe), and others.

CalWET and TCLP (and similar European/Brazilian tests) are specifically designed for the evaluation of municipal wastes and are not well suited for characterizing mine wastes. SPLP and MWMP can provide some indication of readily soluble constituents and can thus highlight potential metal/trace metal release issues, particularly for metals that may be soluble in the circum-neutral range (zinc, cadmium, selenium, and arsenic), but the relatively high solution-to-solid ratios and use of variably sized materials must be considered in evaluation of results. The MWMP is used routinely in Nevada following implementation of their metal mine-waste characterization regulations.

These tests may be a regulatory requirement for evaluation of potential impacts to water but are not are highly regarded as a measure of acid rock drainage (ARD) potential because oxidation is not induced by the test itself. They may not be commonly included in detailed waste characterization work for that reason (see Table 5.2). Nonetheless, there may be cases where one of these short-term leach tests can be linked with more traditional ARD tests as part of a prediction or characterization program, particularly if metal/trace element release is a potential issue. It may be possible to use the metal release information from the MWMP or SPLP as a key indicator for potential environmental risk, particularly if oxidized and weathered materials are available. This information might also be useful in identifying specific metals and release rates to focus on in interpreting humidity cell (HC) tests and developing projections for contaminant release and water quality. There are no established standards for compliance for this type of leach test, and these results are most often compared with surface and groundwater quality standards.

FIELD LEACH TESTS

Field leach tests are generally conducted on weathered material that is easily available on site. These tests are generally not considered to be representative when conducted on unweathered material.

Paste pH

Paste pH is probably the simplest of ARD tests. A simple measurement of the pH of a mixed solid and water mix gives a pH reading that provides evidence of immediately available acidity stored in soluble sulfate or sulfo-salt minerals from prior acid production. The paste pH test is applicable for samples in the weathered/oxidized portion of a deposit, as it will not provide information on long-term AGP from unweathered sulfide minerals. Since the pH of deionized water used for the test is approximately 5.8, a reduction in pH of the water below that value generally indicates the presence of acidic salts that identify the material as potentially acid generating. Paste pH will *not* provide information on the long-term ability of a sample to produce acid and so must be linked with other testing information as part of a comprehensive characterization program. In conjunction with other static, short-term, and kinetic tests, paste pH can aid in identifying immediately soluble constituents that can impact water quality.

Acid Concentration Present Leach

The acid concentration present leach test is slightly more involved than a standard paste pH but supplies an estimate of acidity present rather than simply pH (C. Bucknam, personal communication, 2000). After recording the initial pH, the mixture is titrated to pH 7 with sodium hydroxide (NaOH). The volume of NaOH needed to titrate to this value is used to calculate the amount of acidity in units of % $\rm CO_2$. A negative value indicates acidity. This test is subject to the same limitations for predicting the long-term ability of a sample to produce acid as paste pH. Its application should follow the same general guidance, being an integral part of a waste characterization program.

USGS Field Leach Test

The U.S. Geological Survey (USGS) field leach test (FLT) (Hageman 2007) is similar to the paste pH test but includes electrical conductivity measurements and trace element signatures (inductively coupled plasma mass spectrometry, or ICP-MS), if necessary. Similar to the SPLP, the results of the FLT are based on comparison of analytical results with water quality standards.

Wall Washing

This test is not standardized and is essentially an instantaneous evaluation of reaction products on the pit walls. It is thought to present a useful first-order approximation of contribution to ARD from exposed pit walls (INAP 2009). In some cases, the procedure can be repeated weekly (Price 1997). The collected leachate is analyzed and the results are expressed in milligrams per square meter per week of anion or cation.

KINETIC TESTS

Humidity Cells

Objectives of HC laboratory-weathering tests include the following tasks: (1) determine the variation of drainage quality as a function of mine-rock compositional variations (e.g., iron sulfide and calcium + magnesium carbonate contents) within individual mine-rock lithologies, (2) measure the amount of NP accessible in a mine-rock sample to neutralize acid and maintain drainage

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pH \geq 6.0 under the conditions of the test, (3) estimate mine-rock weathering rates to aid in predicting the environmental behavior of mine rock, and (4) estimate mine-rock weathering rates to aid in experimental design of site-specific kinetic tests (ASTM D 5744-13).

These objectives are implemented through collection of diagnostic data during the test. The generated data can be used to

- · Determine the likelihood that a solid material will produce acidic, alkaline, or neutral effluent (pH, acidity, and alkalinity);
- · Identify solutes in the effluent that represent dissolved weathering products formed during a specified period of time (e.g., Fe and SO₄ from iron-sulfide oxidation; Ca and Mg from dissolution of Ca+Mg carbonates);
- · Quantify the mass of solute release (mg/kg); and
- · Calculate the rate at which solutes are released (from the solids into the effluent) under the closely controlled conditions of the test (mg/kg/week).

Examples of data analyses. A common representation of data developed through various static and kinetic tests is to develop a table format that can be used to quickly cross-reference and summarize the results.

Table 5.2 is a representation of a typical range of waste characterization results developed to support proposed mine operations. The table shows the analysis of seven samples and the results of six static test methods (NCV, MWMP, Modified ABA Ratio Class, Modified ABA NNP Class, Peroxide Acid Generation Class, and ACPL [Annual Certified Parameter List] Titration) and two kinetic tests (Biological Acid Production Potential Class and Humidity Cell Class). Not all tests were performed on all samples, and this is noted as "not tested" in the table. The purpose of presenting this table is to demonstrate the range of tests typically performed and the comparative outcome of the tests. The multiple results are taken in aggregate to support risk assessment and decision making.

Another common way of representing HC test data is to plot weekly solute concentrations and cumulative solute release, as shown in Figures 5.1–5.11. The HC results displayed in Figures 5.1–5.5 have been selected to show a range of HC results that occurred as the % S changes for samples with relatively simple mineralogy. The samples are all siltstones containing mostly quartz and minor clay minerals. The specific mineralogy is noted for each of the samples. Figures 5.1 and 5.2 are the 20-week runs (based on historic but not current ASTM recommendations), followed by a longer run (Figures 5.3–5.5) for a sample with similar lithology and sulfide sulfur content. Figures 5.6–5.8 show a sample with similar lithology run as a longer test. Figure 5.9 is a sample with lithology similar to Figures 5.1–5.8 but with much higher sulfide sulfur. Figures 5.10 and 5.11 show three samples with more complex mineralogy.

The key considerations for any kinetic test as discussed in Chapter 4 are (1) clearly defined test objectives; (2) appropriate methods of testing; (3) preliminary and post-testing analysis of solids, including static and mineralogical analyses; (4) appropriate test duration; and (5) a well-understood goal for application of results. These considerations are evaluated in the following list as an example for Figure 5.1:

- Clearly defined test objectives. The objective for the sample was to determine whether or not it would produce acid. The static test results put the sample in the uncertain range. The pH from weeks 12 to 20 was relatively constant between 6.0 and 6.2.
- · Appropriate methods of testing. The HC test was a necessary part of the sample evaluation. The comprehensive mineralogical, static test work, and interpretation of static test results was inconclusive on whether the sample may be acid producing. Thus the use of the

HC test is appropriate to provide additional information on the acid-producing potential of the sample.

- · Preliminary and post-testing analysis of solids, including static and mineralogical analyses. The HC test for the sample included the recommended preliminary and post-testing analysis as well as the recommended static and mineralogical analyses.
- · **Appropriate test duration.** The 20-week test (historic) duration was sufficient to meet the defined test objective, which was to evaluate whether or not the sample was likely to produce acid. The kinetic test objectives were met, thus the test duration was appropriate. If the test objectives had not been met, then the test duration would have been revised.
- A well-understood goal for application of results. The goal for the application of the HC results was to provide information for the development of a waste characterization and management plan. The results of the HC test shown in Figure 5.1 did meet the stated test objective of providing evidence on whether the material is unlikely to produce acid. The results do not provide information on the long-term rate of acid production. However, the HC data, in conjunction with mineralogy and static tests results, were taken in aggregate in the development of a waste characterization and management plan. The actual waste material has not produced acid in a field setting over several years.

This sample in Figure 5.1 is a siltstone with the following approximate mineralogical composition: 82% quartz (SiO₂), 8% sericite/illite ((K,H₃O)(Al,Mg,Fe)₂(Si,Al)₄O₁₀[(OH)₂,(H₂O)]), 4% alunite (KAl₃(SO₄)₂(OH)₆), 2% barite (BaSO₄), and 4% iron oxides (Fe₂O₃, Fe₃O₄). Note the low alkalinity and acidity values and the relative stability over the last 9 weeks of the test.

The mineralogical composition of the siltstone in Figure 5.2 is similar to Figure 5.1. The pH did not stabilize in the 20-week duration of the test. The pH was always above 5.5 between weeks 12 to 20 and varied between 5.6 and 6.7. The slopes of the cumulative alkalinity, acidity, and sulfate curves are similar and were still increasing when the test was terminated. The production rate of acidity and sulfate is relatively low compared to the acid-producing material shown in Figure 5.9. The HC results are inconclusive on the likely production of acid. However, the mineralogical, static, and HC results in aggregate were used to make the decision to manage this material as non-acid-producing.

Figures 5.3–5.5 show a long run humidity cell for a sample containing 0.02% sulfide sulfur with an NNP of 6.62 that was classified as either uncertain or non–potentially acid generating based on mineralogy and static tests.

The HC results presented in Figures 5.3–5.5 are similar to Figure 5.2 in the variation of the pH (between 5 and 7) over the last 10 weeks. The pH in conjunction with the acidity/alkalinity data suggests the sample has low acidity and alkalinity production. Again, the rate and accumulation of aluminum and iron production are relatively low compared to a known acid-producing material shown in Figure 5.9. The aggregate mineralogy, static test, and HC data used in the decision to manage this was as non-acid-producing.

Figures 5.6–5.8 show a long run test for a similar lithology with a sulfide-sulfur content of 0.12%, NNP of 6.82, and an NP/AP ratio of 2.82. The relative stability of the values between weeks 18 and 30 illustrates the inherent risk in relying on a transient period of "stability" in an HC test.

In Figures 5.6 and 5.8, effluent from the HC sulfate is below 30 mg/L, and iron and aluminum are below 0.1 mg/L between weeks 36 and 48. The pH ranges between 4.7 and 5.3 between weeks 36 and 48. During this period, effluent values are varying by as much as 20%, thus the effluent values have not stabilized. The continued sulfate release, as well as high conductivity numbers

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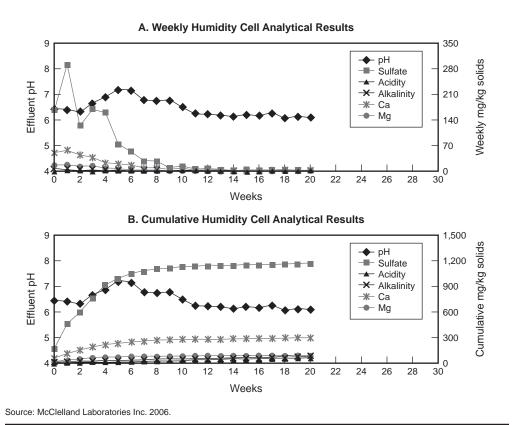


FIGURE 5.1 (a) Weekly and (b) cumulative humidity cell results for a sample with 0.06% sulfide sulfur (AP = 1.9, NP = 0.5, NNP = -1.4, NPR = 0.26)

compared to other samples of similar mineralogy (IML 2012) suggest that the test should be maintained until a more determinative endpoint is reached, or the sample should be identified as an acid producer and managed accordingly. The latter management decision was made for this sample.

For comparative purposes, Figure 5.9 presents a sample of a siltstone with 0.44% sulfide sulfur, showing it is clearly acid generating. In comparison to Figures 5.1 and 5.2, the rate rapidly drops to less than 3 within 20-weeks and the cumulative release of sulfate is much higher and continuing to increase. The aggregate information on the mineralogy, static tests, and humidity cell was used to make the decision for this material to be managed as acid producing.

Weekly HC results for samples with more complex mineralogy are displayed in Figures 5.10 and 5.11. These figures provide examples of the long period of time that might be required for drainage to acidify in kinetic tests. HC test results from three quartz-carbonate samples T6, T9, and T10 are presented in Figure 5.10. The results illustrate the time required for leachate to reflect exhaustion of the neutralizing ability of the material. These figures present data generated from the continuation of the HC tests of samples up to 550 weeks (results from the first 151 weeks are described in Lapakko and Wessels 1995).

Figure 5.11 presents more detailed data from sample T9 over the first 151 weeks. The effluent pH was relatively steady at pH 8 for 100 weeks and then began to decline significantly at 120 weeks. The effluent pH reached \approx 3.5 at 151 weeks. The projected time to elimination of calcite

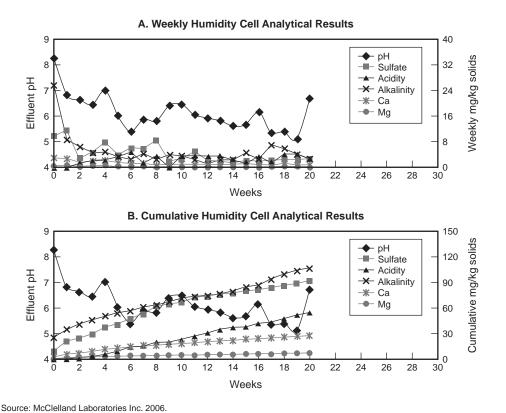


FIGURE 5.2 (a) Weekly and (b) cumulative humidity cell results for a sample with <0.01% sulfide sulfur (AP = <0.3, NP = 0.5, NNP = 0.2, NPR = >1.7)

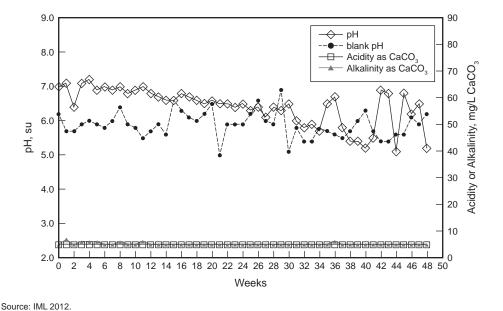
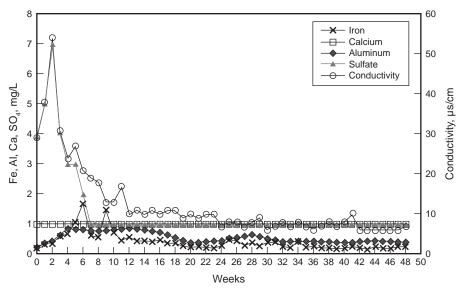
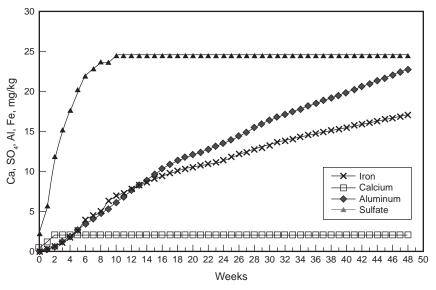


FIGURE 5.3 Weekly long run humidity cell pH and acidity/alkalinity concentrations for a silt/ chertstone unit. The blank is a washed sand.



Source: IML 2012.

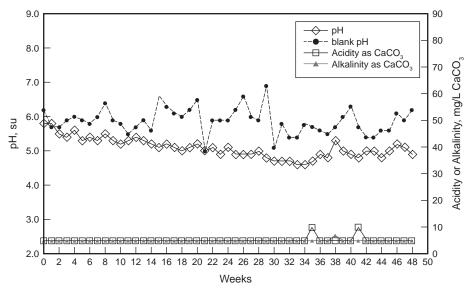
FIGURE 5.4 Weekly concentrations for Figure 5.3



Source: IML 2012.

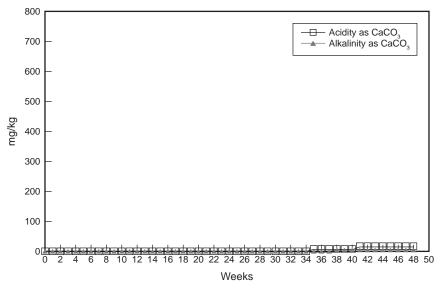
FIGURE 5.5 Cumulative concentrations for Figure 5.3

(based on 30 weeks) was close to the time when the acid-neutralizing capacity of the sample was consumed. The projection of calcite elimination and thus loss of neutralizing capacity is an example of "time" scaling of HC results. Additional discussion of time scaling HC results can also be found later in this chapter in the "Humidity Cell and Column Test Scaling" section and in Appendixes K and L.



Source: IML 2012.

FIGURE 5.6 pH, acidity, and alkalinity. The blank is a washed sand.

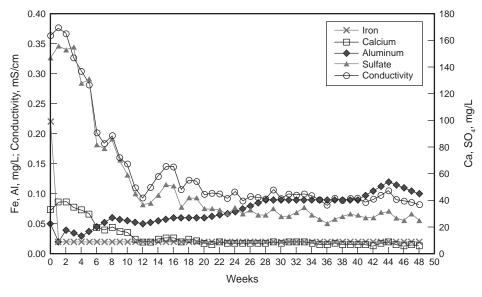


Source: IML 2012.

FIGURE 5.7 Cumulative acidity and alkalinity

Evaluating kinetic tests. Collectively, Figures 5.1–5.11 illustrate some of the complexities involved in evaluating kinetic tests, discussed in the following paragraphs.

One major difficulty is in establishing exactly when to terminate a kinetic test. If no objectives for application of the test results to waste characterization and risk management have been established, then criteria to establish the appropriate duration cannot be objectively set. As a



Source: IML 2012.

FIGURE 5.8 Conductivity and various other parameters on a weekly basis

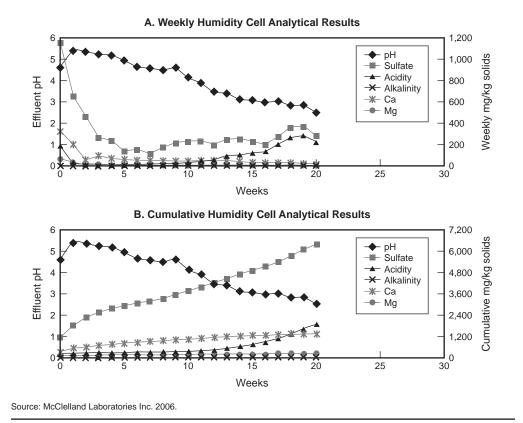
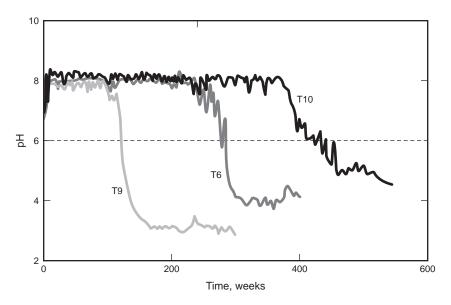


FIGURE 5.9 Sample with 0.44% sulfide sulfur (AP = 13.8, NP = <0.3, NNP = -13.8, NPR = <0.02) showing a clear trend toward ARD, illustrated by the final pH of 2.51



Courtesy of the Minnesota Department of Natural Resources

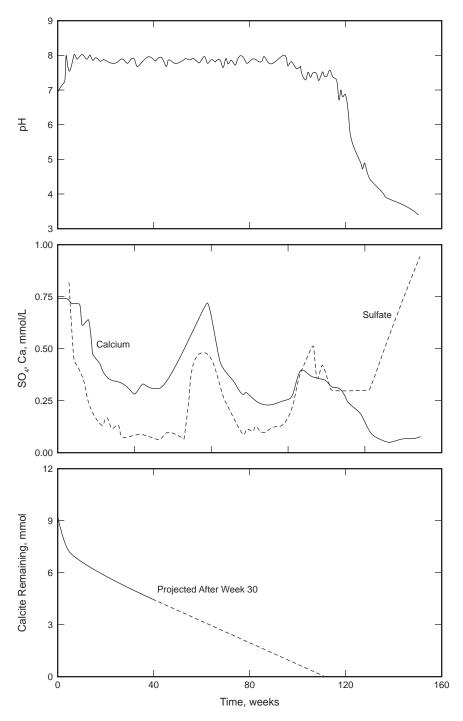
FIGURE 5.10 pH versus time from HC tests of three quartz-carbonate samples: T6, T9, and T10. Nominal characteristics of the samples: T9 AP = 157, NP = 14, NNP = -143; T6 AP = 66, NP = 45, NNP = -21; T10 AP = 197, NP = 200, NNP = +3.

result, decisions about the management of waste materials are sometimes made based on incomplete information.

The HC test for the sample shown in Figure 5.1 was terminated at 20 weeks because of historical test methodology. The management decision for the low-sulfide material represented in Figure 5.1 was that this sample was not likely to produce acid based on the detailed mineralogy and static test results, supplemented by mass balance calculations. The placed waste rock has not produced acid after more than 7 years (T. Schmidt, personal communication, 2013).

The sample in Figure 5.2 with <0.01% S and classified as net neutralizing based on static tests was ultimately managed as unlikely to produce acid based on a recommendation of McClelland Laboratories Inc. (2006). The kinetic test on this sample did not produce stable values by 20 weeks; however, the test was concluded at 20 weeks. The decision to manage the material as non-acid-producing was based on the mineralogy, a static test, and low relative amounts of releases from the sample (McClelland Laboratories Inc. 2006). Price (2009) recommends that kinetic tests should be operated until the rate of sulfate generation and metal leaching have stabilized at relatively constant rates, defined as the average of the last five weeks of testing. The GARD Guide notes that a common termination criteria is when leachate parameters are relatively constant with time (INAP 2009). The effluent from the HC test presented in Figures 5.6–5.8 illustrates the difficulty in reaching stable pH values and interpreting the rate and behavior of mineral leaching. In the 36–48-week time period, the effluent pH varied from 4.7 to 5.3, and aluminum and sulfate varied by 20%. Thus, even for a sample with relatively simple mineralogy, establishing the duration of the kinetic test is challenging, particularly since there is no consensus regarding what constitutes "stability" alluded to in some of the guidance.

Samples with complex mineralogy (Figures 5.9–5.11) may need to be evaluated through more detailed mass balance calculations. Mineral dissolution rates could then be estimated but



Courtesy of the Minnesota Department of Natural Resources.

FIGURE 5.11 HC test results from quartz-carbonate sample T9 showing relationship between leachate pH, and corresponding calcium and sulfate mass release over a 151-week period of record and a projection of calcite consumption based on 30 weeks of data

not conclusively established. The more complex the mineralogy, the higher the uncertainty with regard to when is an appropriate time to terminate an HC test. The uncertainty in the estimated weathering rates only highlights the importance of (1) obtaining detailed knowledge of the lithology and mineralogy of the sampled material; (2) conducting selected tests (field or laboratory) for extended durations to provide empirical data on long-term dissolution behavior that can be used to support other characterization work; (3) noting the language in the ASTM D 5744-13 standard method that "...criteria for ending the testing will depend on the objective of the test, which may be site specific"; and (4) understanding the extent of uncertainty that is acceptable in the specific mine's plan of operations and ensuring that financial assurance instruments are adequate to address those uncertainties. An adaptive management plan (Appendix J) is one possible mechanism for addressing uncertainties.

Understanding the actual test procedures is important. Deionized water is typically used for kinetic tests. It has essentially no buffering capacity and so will equilibrate with the atmosphere at a pH of approximately 5.6 (depending on ambient temperature, pressure, and atmospheric CO₂ concentration). Thus, an effluent pH of 5.6 could suggest that the sample is not reactive anymore, or it could be an artifact of balanced acidity and alkalinity, or it could be transitional on the way to acidic pH. Solution conductivity is an indirect indicator of the amount of total dissolved solids in the solution. Higher conductivity suggests that higher concentrations of ions have leached from the test material into the test solution. The blank sample in Figure 5.3 and the sample pH after week 34 both reflect the difficulty in measuring the pH in low-ionic-strength solutions.

As a practical matter, it is often less expensive and time consuming to simply run humidity cells for a longer time period than it is to run them for a set time period, and then argue the technical merits of the selected time period for the samples.

Other considerations should be addressed as objectives and may have considerable bearing on the test duration. These could include site-specific high environmental risk, problematic mineralogy, erratic HC results, or complex waste management issues. In some cases, it may be useful to establish criteria for a decision based on HC results but still maintain the HC test. In other cases, it might be useful to have duplicate humidity cells and use one as a basis for a decision point and subsequent destructive evaluation. This could support a decision concerning mine-waste management and planning, including an adaptive management plan. The duplicate cell would be maintained to confirm the basis for the decision and be used to update the adaptive management plan and financial guarantee if necessary. This is discussed in Appendixes K and L.

An example of a more robust evaluation of HC tests is to evaluate the percentage of acid-producing and acid-neutralizing material dissolution on a weekly and cumulative basis to develop comparative rates for NP and AGP. This can be presented as shown in Tables 5.3–5.5 and Figures 5.12 and 5.13. This example illustrates a method for analyzing the HC data to estimate comparative dissolution and oxidation of acid-consuming and acid-producing minerals.

The samples represented in Tables 5.3–5.5 are siltite-argillite lithologies. Samples 1-C and 1-D contain 3% and 24% pyrite, 45%–50% quartz, and 5%–10% potassic and sodic feldspars, respectively. Sample 1-D also contains about 3% carbonate minerals (ankerite, siderite, ferroan dolomite/magnesite). The Duluth Complex sample is composed of a mix of plagioclase feldspars (Ca₂Al₂SiO₈ to NaAlSi₃O₈), olivines ((Mg, Fe)₂SiO₄), pyroxenes ((Ca, Na, Fe⁺²)(Al, Fe⁺³, Mg) (Si,Al)₂O₆), magnetite (Fe₃O₄), and minor sulfides. Hydrothermal quartz-carbonate samples T6, T9, and T10 contain roughly 5%–7% sulfide minerals, 1%–53% carbonates (calcite, ankerite, siderite), and a total of 37%–74% quartz, feldspar, mica, and chlorite.

Tables 5.3 and 5.4 show that siltite-argillite samples 1-C and 1-D were projected to deplete their respective acid-producing minerals over a period of 7 and 39 years, respectively. Only sample

TABLE 5.3 Siltite-argillite samples 1-C and 1-D (SO₄)

		SO ₄ Release Rate, mg/kg/wk		Time for S ^{Tot} Oxidation, years	
Sample	S ^{Tot} as SO ₄ , mg/kg	Weeks 5-21	Weeks 5-51	Rate from Weeks 5–21	Rate from Weeks 5-51
1-C	390,000	418	1,048	18	7
1-D	45,900	27	23	33	39

Source: White and Jeffers 1994.

Note: Average weekly sulfate (SO₄) release rates after 21 and 51 weeks of laboratory weathering (humidity cells) and time in years to total sulfide (S^{Tot}) oxidation for listed mine-waste samples.

TABLE 5.4 Siltite-argillite samples 1-C and 1-D (Ca + Mg)

	CaCO,	Ca + Mg Release Rate, mmoles/week		Time for NP Depletion, years	
Sample	Equivalent, mmoles	Weeks 5-21	Weeks 5-51	Rate from Weeks 5–21	Rate from Weeks 5–51
1-C	0	0	0	0	0
1-D	33	0.3	0.25	2.0	2.5

Source: White and Jeffers 1994.

Note: Average weekly calcium + magnesium (Ca + Mg) release rates after 21 and 51 weeks of laboratory weathering (humidity cells) and time in years to total neutralization-potential (NP) depletion for listed minewaste samples.

TABLE 5.5 Mixture of Duluth Complex rock with rotary-kiln fines

Sample	S ^{Tot} as SO ₄ , mmoles	SO₄ Release Rate, mmoles/week	Time for S ^{Tot} Oxidation, weeks
Duluth + rotary-kiln fines	44	0.047	950
Sample	CaCO ₃ Equivalent, mmoles	Ca + Mg Release Rate, mmoles/week	Time for NP Depletion, weeks
Dulth , retem kiln fines	3.7	0.093	40
Dulth + rotary-kiln fines	3.7	0.073*	51

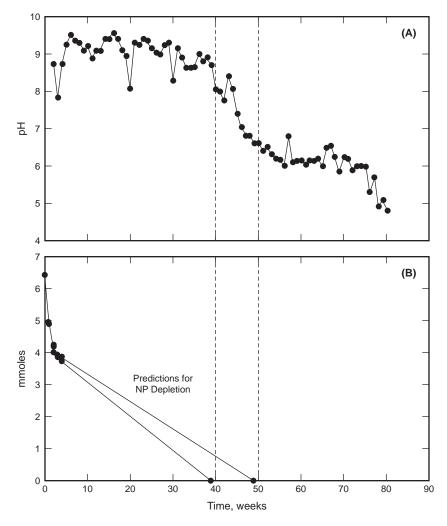
Source: Lapakko 1990.

*Release rate of Ca without Mg, in mmoles/week.

Note: Average weekly sulfate (SO_4) and calcium + magnesium (Ca + Mg) release rates after 20 weeks of laboratory weathering and time in weeks to total sulfide (S^{Tot}) oxidation and NP depletion for listed mine-waste samples. See also Figures 5.3–5.5.

1-D contained any neutralizing minerals. Using the rate of calcium plus magnesium release, these neutralizing minerals were projected to be depleted after 2 years. Based on these projections, sample 1-D drainage quality was predicted to become acidic after 2 to nearly 3 years of HC testing. In contrast, sample 1-C contained 9 times the amount of sulfide minerals (pyrite), was balanced by zero NP, and produced acid from the start of testing.

Table 5.5 summarizes the calculated time to depletion of acid-producing and acid-neutralizing minerals for a mixture of Duluth Complex and rotary-kiln fines. Depletion of acid-producing minerals was projected to take 950 weeks (18+ years), whereas acid-neutralizing mineral depletion was projected to require 40 to 51 weeks (<1 year). This is graphically illustrated in Figures 5.10 and 5.11, which shows a correlative drop in pH within the same 40-to-51-week period. Based on these data, one would anticipate that these samples would generate acidic drainage quality once the neutralizing minerals were depleted.



Source: Lapakko 1990.

FIGURE 5.12 Predicted depletion of solid phase neutralization potential (mmoles Ca) and observed pH versus time for a mixture of Duluth Complex rock with rotary-kiln fines (alkaline solid to sulfur ratio of 0.5:1.0; mixture AP = 65, NP = 8.4)

The same method was used to calculate the dissolution required to deplete acid-producing and acid-neutralizing minerals from hydrothermal quartz-carbonate tailings samples T6, T9, and T10. Predicted depletion of acid-producing minerals ranged from 22 to 27 years, whereas projected depletion of acid-neutralizing minerals ranged from 2 to 10 years. Consequently, in this example, one would anticipate that these samples would generate acidic drainage once the neutralizing minerals were depleted. Actual pH trends for the three samples show significant pH reductions, which approximate the projected time to acid-neutralizing mineral depletion (Figure 5.12a). Figure 5.12b shows projected calcite depletion versus actual calcium depletion for sample T9.

Figure 5.13 is a similar plot showing mass balance calculations. Plots comparing the relative mass of pyrite and carbonate consumed weekly are useful in understanding the relative mass transfer during the weathering test and enable the interpretation of the potential for acid production.

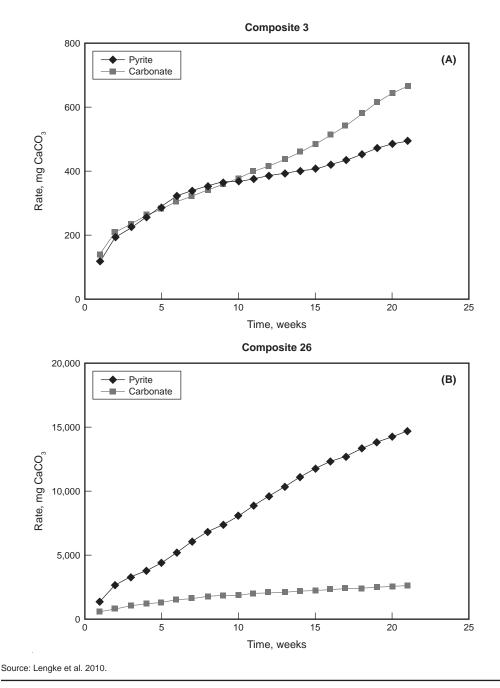


FIGURE 5.13 Mass balance calculations based on HC results showing carbonate dissolution and pyrite oxidation in two composite samples: (a) composite 3 is a non-acid-generating sample based on several criteria; (b) composite 26 is an acid-generating sample

In the case of the sample shown in Figure 5.13a, the relative sulfide consumed each week is equal to the carbonate, and no acidity would be expected. In the latter sample, the pyrite consumed well exceeds the available carbonate, and acidity would be anticipated.

Lapakko (1990), Lapakko and Wessels (1995), USEPA (2009), Lengke et al. (2010), and Price (2009) have suggestions on mass balance calculations that can be quite useful. This information can be a critical supplement to the graphical representation present in the weekly HC results.

The discussion of HC duration, stakeholder objectives, and criteria in the following section and in Appendixes K and L is meant to provide a *framework* for stakeholders for understanding HC test duration issues. This discussion can also be used to aid in developing appropriate objectives and criteria for identifying a duration or decision point and to avoid extending testing beyond what is scientifically necessary, defensible, or cost-effective.

Humidity-Cell Test Duration

HC tests are often required to support decisions regarding how mine and processing waste will be managed. It is important for stakeholders to understand that HC tests lead to decisions about managing waste that can be included in a proposed mine plan. Because of long permitting lead times, humidity cells are often started early in the mine and process development cycle, before the final mine plan is selected or proposed for permitting. Decisions on waste management may require the geochemical data generated by the HC test in order to support a waste management plan.

There is no simple formula for determining test duration. Ultimately, test duration must be a function of reducing outcome uncertainty and level of risk through the development of detailed objectives. It is these detailed objectives that should determine the duration, not a predetermined amount of time.

Because the results that will be generated are not known in advance, it is not possible to establish a specific test duration prior to test initiation. The decision to terminate testing must be based on an assessment of whether the detailed objectives that were developed for the project have been met in light of information gathered during testing. One process that fits this description is to assess the extent to which objectives have been met based on interpretation of the solid-phase characterization and HC data generated at given time intervals. For example, after a set period of testing, data would be evaluated to determine whether objectives had been addressed. This interpretation would include consideration of uncertainty associated with applying the data to mine-waste management decisions and the risk (environmental and financial) to be managed. If objectives have been addressed, testing would be terminated. If not, testing would be continued for an additional period during which time attainment of objectives would be assessed based on the interpretation of additional data generated. The State of Nevada uses a similar incremental method of HC-test data analyses (Karl McCrea, personal communication, 2011). Additional guidance criteria are discussed in Appendices K and L.

If uncertainty regarding the environmental behavior of a given rock type has not been adequately reduced, managing the environmental risk in place of additional testing could be considered. A comprehensive strategy could include options for extensive monitoring, effluent collection, and treatment developed through an adaptive management plan (Appendix J). Regardless of how the duration and termination decision is reached, ultimate responsibility for the permitting decision lies with the permitting agency(s), and the ultimate environmental liability lies with the mining company.

Existing test duration guidance. ASTM D 5744-96 prescribed a minimum test duration of 20 weeks, but the 20-week minimum was eliminated from subsequent revisions ASTM D 5744-07e1 and D 5744-13. However, the 20-week minimum test duration is still used by some investigators for reported HC test duration (Bouzahzah et al. 2012; McClelland Laboratories Inc. 2006) in spite of lacking specific technical or scientific support. The original ASTM

statement was replaced with the following specific data-supported guidance derived from a 5-year intralaboratory study and other published research (Lapakko 1990; Lapakko and Wessels 1995; Lapakko and White 2000; White and Lapakko 2000; ASTM D 5477-13): "The criteria for ending the testing will depend on the objective of the test, which may be site specific." For example, "...if the objective is to determine the relative reactivity (for example, drainage pH, rates of sulfate release) as a function of compositional variations (for example, iron sulfide and calcium + magnesium carbonate contents) within individual mine-rock lithologies for the purpose of segregating mine wastes, then a specific test duration is not prescribed." Consequently, the duration might be quite long if tests are intended to empirically determine if drainage will acidify or to quantify the availability of neutralization or acid generation potentials (ASTM D 5744-13).

The more recently released GARD Guide (INAP 2009) recognized the lack of sound foundation for limiting kinetic tests to a 20-week duration, stating: "The required duration of kinetic testing is an area of controversy. The duration of the test depends on the characteristics of the sample and test objective. Although a minimum length of 20 weeks is sometimes referenced, there is little technical basis for the 20-week recommendation." The GARD Guide notes: "Another common endpoint is when leachate parameters are relatively constant with time." Price (2009) does not reference ASTM D 5744-96, instead suggesting the test continue until the rate of sulfate generation and metal leaching have stabilized at relatively constant rates, defined as the average of the last five weeks of testing. In the event that uncertainties remain in spite of the five weeks of constant rates, the test must continue until those uncertainties can be resolved (W. Price, personal communication, 2011). However, the rationale for this suggestion is unclear given that rates can stabilize for years, and drainage acidification can follow this period of long-term stability (e.g., Figure 5.12a). As noted previously, there is no consensus on exactly what "relatively constant" or "stability" means in the context of a humidity cell.

The ASTM recommended that post-test solid material analyses of HC residues (ASTM D 5744-13) should not be initiated until the test objectives have been met. If the HC test is prematurely terminated, it may be necessary to re-start humidity cells under some circumstances.

Collectively, the lack of well-defined criteria for HC duration highlight the importance of a detailed understanding of the mineralogy, static test results, and sound, site-specific objectives.

HC OBJECTIVE AND CRITERIA FRAMEWORK

This discussion is designed to provide an introduction to a framework for stakeholders to use in developing objectives and criteria relevant to determining an endpoint or a decision point to HC tests. As a result of the historic ASTM D 5744-96 method, test durations as short as 20 weeks have been used for HC testing (see Figures 5.1–5.5). The current ASTM method revision deleted reference to a 20-week testing period and recognizes that the duration of testing should depend on the objectives and outcomes of the testing (ASTM D 5744-07e1 and D 5744-13).

Possible criteria include concentration versus pH, load release, and trace metal release rate trends. This information can be correlated with mass balance graphs showing consumption and release of sulfides or carbonates, as shown in Figure 5.13.

It may be appropriate to establish specific criteria for each of the different lithologies/rock types or alteration patterns at a deposit, as each of these units should have its own characteristic governing mineralogy. More complex mineralogy may require longer kinetic test run times to come to a conclusive result. It is generally accepted practice to fully characterize the material subjected to HC testing before and after the test procedure (ASTM D 5744-13; USEPA 2009). For samples with complex mineralogy, this can be critical. This information is useful in evaluating the

mass removed during the test. This information can be particularly important if one is attempting to link HC results to proposed or existing modeling work or existing field-scale tests.

Additional discussions regarding possible frameworks for use with HC tests are included as Appendix L.

Additional HC Test Data-Management Needs

Because the HC test protocol has been designated an ASTM standard, potential exists for creation of a database that demonstrates how various mine-waste lithologies respond to the standardized test under intra- and interlaboratory conditions. To provide a more extensive empirical foundation for assessing kinetic-test results from the wide variety of mine-waste lithologies of interest, the following are needed:

- · A published, comprehensive catalog of dissolution data from long-term laboratory or field kinetic tests performed on multiple mine-waste lithologies
- · A detailed solid-phase characterization and pertinent reaction variables associated with tested lithologies
- · A database that documents problematic lithologies (including mineral alteration suites) and subsequent field performance monitoring of managed mineral-processing waste and waste rock

Unfortunately, none of these critical databases currently exist, though Morin and Hutt (2010) have an online database that may be helpful.

COLUMN TESTS

Column test results would generally be similar to HC test results in terms of the monitored components and leachate. Column tests can be structured to include such variables as particle size, subaerial or subaqueous disposal, aerobic or anoxic conditions, reduced temperatures, reduced infiltration, and other complex operational or environmental factors. Column tests are typically monitored in much the same fashion as HC tests and thus have many of the same operational and interpretation issues. Column tests are custom protocols designed to represent unique environmental conditions at individual sites, and as such, results may not be inherently comparable between mine sites. Material studied in column tests should be well characterized using established methods of mine-waste characterization prior to initiation of column work. Column test results are generally represented using similar plots and figures of solute chemistry.

HUMIDITY CELL AND COLUMN TEST SCALING

Geochemical weathering reactions are scale dependent, which means that the results obtained in the laboratory will be different from those observed at full-scale mine facilities. Controlled laboratory HC tests and column tests can be used to gain insight into more complex field monitoring data, and vice versa. Access to weathering test data from laboratory *and* in situ conditions offers the benefits of both settings and will greatly strengthen the understanding of environmental geochemistry. By comparing the results of both, it may also be possible to infer the relative duration and magnitude of solute loading over time.

Laboratory-scale HC and column tests evaluate the behavior of specific mineral assemblages under imposed test conditions, and it is important to consider these limitations when interpreting and projecting HC results to field-scale predictions. The rate of flushing in HC tests is intentionally accelerated in the laboratory to prevent the accumulation of secondary minerals and thus optimize the intrinsic rate of oxidation on primary mineral surfaces. As a result, rates of secondary

mineral formation and dissolution are different in the laboratory tests than would be expected in the field. The relative rock/water ratio, flushing rates, water residence times, availability of oxygen, and other test conditions present in the laboratory may represent very different, often more aggressive weathering conditions when interpreted and scaled to the full-scale field condition. Knowledge of flow rates and reactive surface area, as well as rock/water ratios in field-scale analogs, will allow laboratory data to be more meaningfully interpreted and applied to field-scale predictions. It may be appropriate to reduce the solute release in proportion to the relative surface area and adjust predicted concentrations to reflect the change in rock/water ratio prior to using the data in mass load interpretations; for example, in predictions of mass load modeling from the pit wall in pit lake modeling. Changes in HC solute release over time can then be used to infer potential changes in long-term solute loading with greater confidence, based on the mass of rock that will be exposed to water under field conditions.

Column tests can be adapted to incorporate other site-specific variables as noted previously and so may be more flexible than the ASTM HC test. Likewise, this can mean results may not be representative or transferable to conditions other than the exact conditions of the column.

The extent of weathering that takes place during a short-term laboratory test may thus correspond to real-time weathering over a longer timeline (e.g., solute release over a set time period in a humidity cell could correspond to 200 years, depending on the relative weathering conditions). If sufficient data are available characterizing the weathering variables listed previously for material studied in a humidity cell with a field-scale analog, where comparable mineralization has weathered over a prolonged period of time under monitored conditions, HC test data can be scaled for more accurate application to full-scale facilities.

Field-Scale Tests

Detailed evaluation of field-scale tests can typically include monitoring installations that track moisture infiltration/content, evaporation, temperature, radiation, oxygen/CO₂, particle size, and water quality. This information can be used to give a detailed portrait of geochemical reactions and related unsaturated water flow in a waste dump. Figure 5.14 represents a compendium of several years of detailed dump monitoring at a site in Montana. This information can be critical in developing detailed mitigation measures and improving reclamation performance.

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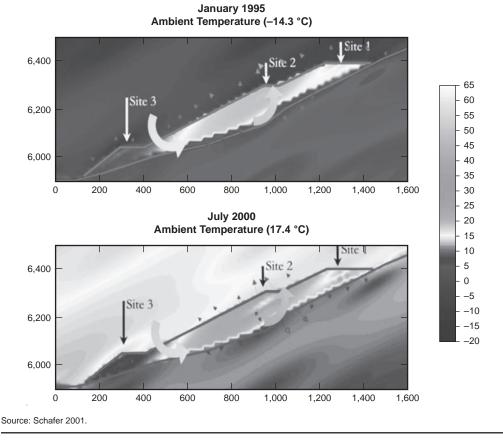


FIGURE 5.14 Detailed interpretation of heat/geochemical reactions in a waste dump based on extensive monitoring of boreholes in the waste dump. Large arrows indicate zones of air movement, and the smaller arrows represent heat flux.

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CHAPTER 6

Application and Conclusion

The characterization and classification of material at a mine site is an iterative process that should begin early in the exploration process and continue throughout the mine life, as discussed in Chapter 1 and graphically illustrated in Figures 1.4, 1.5, and 4.7. This iterative process seeks to answer a series of questions:

- · Will the material to be mined and processed produce acid?
- · Will the properties of the material change as mining proceeds?
- · Are there units to be mined that can be used to neutralize or isolate acidic units?
- · Will any acid produced impact ground- or surface water(s)?
- · What series of test protocol accurately characterizes the material?
- · Of these tests, which can be used to operationally segregate material that might need special handling or storage?
- · Can any of the tests be interpreted to supplement geochemical modeling?
- · Can any of the tests be used to develop field-scale tests that may help to confirm the laboratory tests?
- · Can field-scale tests be interpreted to confirm geochemical predictions and modeling?

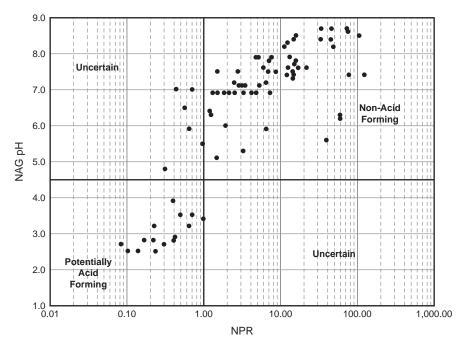
Generally, rock characterization schemes rely on the use of static testing to identify benign and problematic materials that are then subjected to kinetic testing to confirm how the materials are most likely to behave in the weathering environment. This information is generally represented graphically to display the combinations of tests that most effectively characterize the material. For operational management of mine wastes, it is useful to have tests that are reliable, timely, and can be performed at the mine site. Generally, mines rely on a combination of total sulfur, sulfide sulfur, carbonate carbon, neutralization potential (NP), acid–base accounting (ABA) or net carbonate value (NCV), net acid generation (NAG) or paste pH to characterize waste operationally. Sometimes predictive results can be correlated with metal values from routine blasthole assays, which can simplify operational waste management. Table 6.1 and Figures 6.1–6.3 show possible ways to represent data. This data can be interpreted to select possible characterization and waste management alternatives. Ideally, this process identifies a series of static predictive tests that may be capable of accurately representing acid generation potential (AGP). The static tests that do this most accurately are confirmed through application and interpretation of kinetic testing results and converted into an ongoing operational testing program.

How this might work at an actual mine site is shown in the samples discussed in this chapter. Table 6.1 shows test values and rock classification using several static and kinetic tests. This can be a useful way of looking for correlations in the various testing methods used. Figure 6.1 shows a correlation between neutralization potential ratio (NPR) and NAG that might prove useful in an operational waste characterization scheme. Figure 6.2 shows a correlation in an alternative using NCV values and paste pH. The correlation in Figure 6.2 is supported by other tests as well, which

TABLE 6.1 Summary table showing range of static and kinetic test results

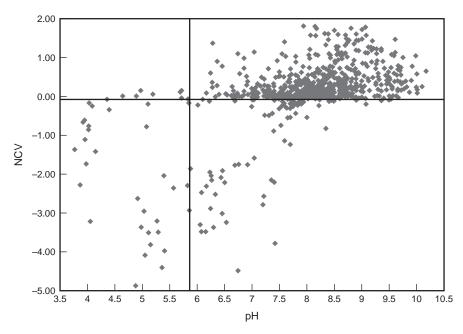
Sample Information		Static Tes	Static Test Results		Kinetic Test Results	st Results
Lithologic Unit	NCV Value (NCV Class)	NPR (NPR Class)	ABA (ABA Class)	NAG (NAG Class)	Biological Acid Production Potential Final pH (HC Class)	Humidity Cell Final pH (HC Class)
Unoxidized siltstone	-0.54	0.40:1	–22.2	2.86	3.18	7.25
	(Slightly acidic)	(PAG)	(PAG)	(PAG)	(PAG)	(Not acid producing)
Oxidized siltstone	0.31 (Slightly basic)	0.80:1 (PAG)	-1.1 (Uncertain)	7.8 (Not acid producing)		6.45 (Not acid producing)
Oxidized siltstone	0.15	0.13:1	-6.5	6.38	3.47	6.27
	(Slightly basic)	(PAG)	(Uncertain)	(Not acid producing)	(PAG)	(Not acid producing)
Oxidized siltstone	0.11	<0.04:1	-7.2	4.37	3.18	4.98
	(Slightly basic)	(PAG)	(Uncertain)	(PAG)	(PAG)	(PAG)
Oxidized siltstone	0.1	2.32:1	-5.3	6.3	3.35	5.37
	(Slightly basic)	(Uncertain)	(Uncertain)	(Not acid producing)	(PAG)	(Not acid producing)
Unoxidized siltstone	-0.377	<0.02:1	-1.9	2.88	2.41	2.51
	(Slightly acidic)	(PAG)	(Uncertain)	(NAG)	(NAG)	(PAG)
Limestone	31.83 (Highly basic)	>2400:1 (Not acid producing)	724.0 (Not acid producing)	Not tested	Not tested	Not tested
Unoxidized siltstone	-0.22	<0.01	–35.8	3.09	2.15	2.91
	(Slightly acidic)	(PAG)	(PAG)	(PAG)	(PAG)	(PAG)
Oxidized siltstone	0	>1.67:1	0.5	5.47	3.32	6.73
	(Inert)	(Uncertain)	(Uncertain)	(Not acid producing)	(PAG)	(Not acid producing)

Source: Adapted from Tetra Tech 2007. PAG = potentially acid generating.



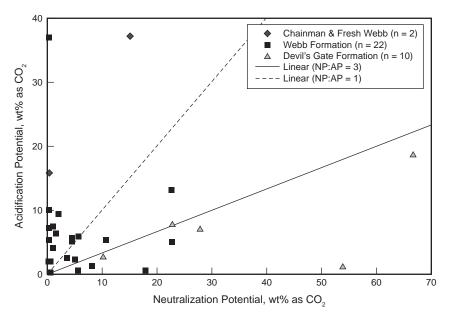
Source: INAP 2009.

FIGURE 6.1 Proposed rock classification based on a combination of NPR and NAG values



Courtesy of Charles Bucknam.

FIGURE 6.2 Rock classification table based on NCV and paste pH showing correlation between NCV and paste pH



Source: Tetra Tech 2007.

FIGURE 6.3 Neutralization potential plotted against acid potential showing NPR values

may make this an effective alternative waste characterization scheme. Figure 6.3 shows there is a clear correlation for some lithologies, and virtually none for others. Any characterization scheme is likely to be valid only for specific lithologies or alteration zones and should not be expected to be effective without documentation for other zones or lithologies.

NPR has been used in the past as part of waste characterization in several countries with values ranging from an NP/AGP from 2:1 to 4:1 being suggested as viable criteria for differentiating waste as non-acid-generating (Price 2009). As noted by Lapakko (2003), there is no general agreement on what would constitute a "safe NPR," and other researchers have come to the same conclusion (Morin and Hutt 1997). NPR is particularly unreliable for samples with low S and NP values, as small changes in values can distort a classification scheme. NPR should generally be used with discretion for carefully differentiated lithologies, and its use should be backed up and confirmed with other predictive tests.

REPORTING PREDICTIVE RESULTS

Reporting predictive results can be another challenging aspect of acid drainage prediction work because it needs to satisfy several objectives. Reports will need to be understood by mine managers, technical and operational staff, regulatory staff, and citizens or nongovernmental organizations. Because it may form the basis for operational sampling and characterization of mine material, it should be something that all levels of mine and regulatory staff can easily understand and that technical and operational staff can actually implement. The best waste characterization work will be of no value if it cannot be effectively administered in an operational mine setting.

Because reviewers may not be familiar with various testing protocols or even the fundamental mineralogy and geochemistry, the report should include a summary section that clearly explains what was done to characterize material, why it was done, and what the results were. This should be followed with a detailed discussion of the sampling protocol: what tests were performed, what

the results were for each of the testing protocols, and an interpretation of what the results mean. Tests that were inconclusive should be explained as well. If kinetic tests were performed, which samples were selected for kinetic tests and how were they selected? The results of the kinetic tests should be explained in detail, then interpreted and linked to the operational waste characterization scheme actually selected. If appropriate, mass balance calculations and interpretations of kinetic tests should be displayed and explained. All work performed should be referenced to the various protocol, including statistical calculations, modeling calculations, interpretations, and code relied on. A detailed discussion of quality assurance and quality control procedures followed should also be included.

In many cases, it may be useful to propose ongoing field verification tests that will help to confirm interpretation of predictive tests and modeling and, if appropriate, proposed mitigating measures.

The report should propose technologies or methods to address and resolve any identified uncertainties. If uncertainties involve environmental values at risk, these must be resolved to avoid lengthy permitting delays and disputes.

CONCLUSION

Prediction of the acid-generating material at mine sites is now recognized as a crucial element of mine feasibility, permitting, and environmental review. Predictive technology has advanced considerably in recent years but still requires a detailed understanding of the entire range of complexities in the various units at a mine site needing evaluation. This volume has attempted to present a thorough discussion of the variety of predictive tests that might be considered for evaluating material for prediction of acid-generating potential.

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APPENDIX A

Sobek Method

PRINCIPLES

The amount of neutralizing bases, including carbonates, present in overburden materials is found by treating a sample with a known excess of standardized hydrochloric acid. The sample and acid are heated to ensure that the reaction between the acid and the neutralizers goes to completion.

The calcium carbonate equivalent of the sample is obtained by determining the amount of unconsumed acid by titration with standardized sodium hydroxide.

COMMENTS

A fizz rating of the neutralization potential is made for each sample to ensure the addition of sufficient acid to react all the calcium carbonate present. This can introduce an element of subjectivity to the test.

During digestion, do not boil the samples because this can cause losses of hydrogen chloride. If boiling occurs, discard the sample and rerun the test. Before titrating to standardize base with acid, fill the burette with acid and drain it completely. Before titrating sample solution and standard acid with base, fill the burette with base and drain it completely to ensure that free titrant is being added to the sample.

CHEMICALS

- Carbon dioxide-free water: Heat distilled water to just boiling in a beaker. Allow to cool slightly and pour into a container equipped with an Ascarite tube. Cool to room temperature before using.
- 2. Hydrochloric acid (HCl) solution, 0.1 N, certified grade (Fisher So-A-54 or equivalent).
- 3. Sodium hydroxide (NaOH), approximately 0.5 N: Dissolve 20.0 g of NaOH pellets in carbon dioxide–free water and dilute to 1 L. Protect from CO₂ in the air with the Ascarite tube. Standardize the solution by placing 50 mL of certified 0.1 N HCl in a beaker and titrating with the prepared 0.5 N NaOH until a pH of 7.00 is obtained. Calculate the normality of the NaOH using the following equation:

$$N_2 = (N_1 V_1) / V_2$$

where

 N_2 = calculated normality of NaOH

 $N_1 = normality of HCl used$

 V_1 = volume of HCl used

 V_2 = volume of NaOH used

- 4. Sodium hydroxide (NaOH), approximately 0.1 N: Dilute 200 mL of 0.5 N NaOH with carbon dioxide–free water to a volume of 1 L. Protect from $\mathrm{CO_2}$ in the air with an Ascarite tube. Standardize the solution by placing 20 mL of certified 0.1 N HCl in a beaker and titrating with the prepared 0.1 N NaOH until a pH of 7.00 is obtained. Calculate the normality of the NaOH.
- 5. Hydrochloric acid (HCl), approximately 0.5 N: Dilute 42 mL of concentrated HCl to a volume of 1 L with distilled water. Standardize the solution by placing 20 mL of the known normality NaOH prepared in a beaker and titrating with prepared HCl until a pH of 7.00 is obtained. Calculate the normality of the HCl using the following equation:

$$N_1 = (N_2 V_2) / V_1$$

where

 N_1 = calculated normality of HCl

N₂ = normality of NaOH used

 V_2 = volume of NaOH used

 V_1 = volume of HCl used

- Hydrochloric acid (HCl), approximately 0.1 N: Dilute 200 mL of 0.5 N HCl to a volume of 1 L with distilled water. Standardize the solution as before, but use 20 mL of the known normality NaOH.
- 7. Hydrochloric acid (HCl), 1 part acid to 3 parts water: Dilute 250 mL of concentrated HCl with 750 mL of distilled water.

MATERIALS

- · Flasks, Erlenmeyer, 250 mL
- · Burst, 100 mL (one required for each acid and one for each base)
- Hot plate, steam bath can be substituted
- · pH meter (Corning Model 12 or equivalent) equipped with combination electrode
- · Balance, can be read to 0.01 g

PROCEDURE

- 1. Place approximately 0.5 g of sample (less than 60 mesh) on a piece of aluminum foil.
- Add one or two drops of 1:3 HCl to the sample. The presence of CaCO₃ is indicated by a bubbling or audible "fizz."
- 3. Rate the bubbling or fizz in step 2 as indicated in Table A.1.
- 4. Weigh 2.00 g of sample (less than 60 mesh) into a 250-mL Erlenmeyer flask.
- 5. Carefully add HCl indicated by Table A.1 into the flask containing sample.
- 6. Heat to nearly boiling, swirling flask every 5 minutes, until reaction is complete. NOTE: The reaction is complete when no gas evolution is visible and particles settle evenly over the bottom of the flask.
- 7. Add distilled water to make a total volume of 125 mL.

APPENDIX A 97

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Fizz Rating	HCI, mL	HCI, normality	•	
None	20	0.1		
Slight	40	0.1		
Moderate	40	0.5		
Strong	80	0.5		

TABLE A.1 Volume and normality of hydrochloric acid used for each fizz rating

Source: Sobek et al. 1978.

- 8. Boil contents of flask for 1 minute and cool to slightly above room temperature. Cover tightly and cool to room temperature. CAUTION: Do not place rubber stopper in hot flask as it may implode upon cooling.
- 9. Titrate using 0.1 N NaOH or 0.5 N NaOH (concentration exactly known) to pH 7.00 using an electrometric pH meter and burette. The concentration of NaOH used in the titration should correspond to the concentration of the HCl used in step 5. Note: Titrate with NaOH until a constant reading of pH 7.0 remains for at least 30 seconds.
- 10. If less than 3 mL of the NaOH is required to obtain a pH of 7.0, it is likely that the HCl added was not sufficient to neutralize all of the base present in the 2.00 g of sample. A duplicate sample should be run using the next higher volume or concentration of acid as indicated in Table A.1.
- 11. Run a blank for each volume or normality using steps 5, 7, 8, and 9.

CALCULATIONS

- 1. Constant (C) = (mL acid in blank) / (mL base in blank)
- 2. mL acid consumed = (mL acid added) (mL base added \times C)
- 3. Metric tons $CaCO_3$ equivalent/thousand metric tons of material = (mL of acid consumed) × (25.0) × (N of acid)
- 4. The acid-generating potential of the sample should be calculated on the basis of the sulfur, or sulfide content (AP = $S^{=} \times 31.25$), which can be determined either through LECO sulfur analysis or wet chemical means.
- 5. The acid-base accounting value or net neutralization potential value is determined by subtracting the acid-generating potential from the neutralization potential.

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APPENDIX B

Modified Sobek Method

PROCEDURE

- Add a few drops of 25% HCl (hydrochloric acid) to 1 to 2 g of pulverized sample on a
 watch glass or piece of aluminum foil. Observe the degree of reaction and assign one of
 the following fizz ratings: none, slight, moderate, or strong fizz.
- 2. Weigh approximately 2.00 g of pulverized sample into a 250-mL conical flask and add approximately 90 mL of distilled water.
- 3. At the beginning of the test (time = 0), add a volume of certified or standardized 1.0 N HCl according to the fizz rating shown in Table B.1.
- 4. Place the flask on a shaking apparatus such a reciprocating shaker, maintained at room temperature. After approximately 2 hours, add the second acid quantity as indicated in Table B.1.
- 5. After approximately 22 hours, check the pH of the pulp. If it is greater than 2.5, add a measured volume of 1.0 N HCl to bring the pH into the range 2.0 to 2.5. If the pH is less than 2.0, too much acid was added in steps 2 and 3. In this case, repeat the test adding a reduced volume of HCl.
- 6. After 24 hours, terminate the test and add distilled water to the flask to bring the volume to approximately 125 mL. Measure and record the pH, making sure it is in the required range of 2.0 to 2.5.
- 7. Titrate the contents of the flask to a pH of 8.3 using certified or standardized 0.5 N or 0.1 N NaOH (sodium hydroxide).
- 8. Calculate the neutralization potential (NP) of the sample as follows:

$$\begin{split} & \text{Modified NP} \big(\text{kg CaCO}_3/t \big) \\ &= \big[\big(N \times \text{vol (mL) of HCl} \big) - \big(N \times \text{vol (mL) NaOH} \big) \times 50 \big] / \big[\text{weight of sample (g)} \big] \end{split}$$

TABLE B.1 Volume of HCl added for various fizz ratings, the modified acid-base accounting procedure for neutralization potential

	Volume of 1.0 N HCl, mL	
Fizz Rating	At Time = 0 hour	At Time = 2 hour
None	1.0	1.0
Slight	2.0	1.0
Moderate	2.0	2.0
Strong	3.0	2.0

Source: Lawrence and Wang 1997.

9. The acid-base accounting value or net neutralization potential value is determined by subtracting the acid-generating potential from the NP.

The acid-generating potential (AGP) of the sample should be calculated on the basis of the sulfide-sulfur content (AGP = $S^= \times 31.25$). Sulfide-sulfur is typically determined as the difference between total sulfur and sulfate-sulfur, although analysis of other sulfur species such as elemental sulfur and barite-sulfur is sometimes justified. Caution should be exercised for certain samples in interpreting sulfate-sulfur analyses, as this form can be either inert (e.g., gypsum) or essentially stored products of acid drainage that could become mobilized if conditions within a waste change. Reference to the MEND Report 1.16.1b is suggested (Coastech Research 1991) and ASTM E 1915-13 for a discussion of sulfur species.

REFERENCES

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- Coastech Research. 1991. *Acid Rock Drainage Prediction Manual*, MEND Project Report 1.16.1b. Ottawa, ON:MEND.
- Lawrence, R.W., and Wang, Y. 1997. Determination of neutralization potential in the prediction of acid rock drainage. In *Proceedings of the 4th International Conference on Acid Rock Drainage*, Vancouver, BC, pp. 449–464.

APPENDIX C

BC (British Columbia) Research Initial Test

SAMPLE

The sample must be taken in such a manner that it is representative of the type of mineralization being examined. A composite consisting of split drill core or randomly selected grab samples should be satisfactory. The number of samples to be examined will depend on the variability of the mineralization and must be left to the discretion of the geologist.

The bulk sample is cone crushed to minus 10 mesh. A representative 250-g portion is split out, dried, and pulverized to around 60% minus 400 mesh for assay, the titration test, and, if necessary, the confirmations test.

ASSAY

The pulverized sample is assayed in duplicate for total* sulfur in a LECO furnace or by wet chemical methods. The acid production potential of the sample, expressed as kilograms of sulfuric acid† per metric ton of sample, is calculated on the basis of the total* sulfur assay.

TITRATION TEST

Duplicate 10-g portions of the pulverized sample are suspended in 100 mL of distilled water and stirred for approximately 15 minutes. The natural pH of the sample should be recorded. The sample is then titrated to pH 3.5 with 1.0 N sulfuric acid using an automatic titrator. The test is continued until less than 0.1 mL of acid is added over a 4-hour period. The total volume of acid added should be recorded and converted to kilograms per metric ton of sample.

For a 10-g sample, the acid consumption is given by

$$mL 1.0 N H_2 SO_4 \times 4.9 kg/t$$

INTERPRETATION

If the acid consumption value (in kilograms of acid[†] per metric ton of sample) exceeds the acid-producing potential (kg/t), the sample will not be a source of acid mine drainage and no additional work is necessary. If the acid consumption is less than the acid production potential or the difference is marginal, the possibility of acid mine water production exists and the confirmation test should be conducted. A pH of 3.5 is chosen for titration, as above this value, the acid-generating bacterium *Thiobacillus ferrooxidans* is not active.

^{*} It is current practice to use sulfide sulfur, not total sulfur.

[†] It is current practice to express acid and neutralizing potential in kilograms per metric ton of CaCO₃, not H₂SO₄.

ORIGINAL REFERENCE

Duncan, D.W., and Bruynesteyne, A. 1979. Determination of acid production potential of waste materials. *Met. Soc. AIME*, paper A-79-29.

APPENDIX D

Lapakko Method

This method is a modification of the BC (British Columbia) research initial test (Lapakko 1994). The major difference between the two methods is the use of a pH 6.0 endpoint instead of pH 3.5.

SAMPLE PREPARATION

Grind or pulverize the sample to 70% passing 325 mesh (i.e., 70% of particles should have a diameter of less than $44 \mu m$).

TITRATION TEST

Prepare a stirred mixture of a 10-g sample in 100 mL of distilled water.

Using an automatic titrator (Type 45 AR Chemtrix pH controllers with Cole Parmer electrodes were used for the original work), and titrate with 1.0 N sulfuric acid until a pH of 6.0 is reached. The test is complete when less than 0.1 mL acid is added over a 4-hour period.

NOTES FROM KIM LAPAKKO

The proposed alternative method of neutralization potential (NP) determination is the same as the BC research initial test, except a titration endpoint of 6.0 was used in the present study rather than 3.5. pH 6 was selected since it is a commonly applied water quality standard. Therefore, the NP available above this pH represents the amount of acid that a mine waste could neutralize while maintaining drainage pH in a range that meets water quality standards.

This method is intended to determine the NP present rather than the NP available for reaction in the field. Consequently, the value determined for waste rock will exceed that which is practically available in the field. This is the natural consequence of not running the test on field-size particles, which would consider the mode of occurrence of neutralizing minerals. It is just a bit difficult to squeeze some of that field-sized material into these beakers. Nonetheless, for future testing I would consider using a larger particle size for waste rock. In our laboratory, we have made the additional modification of using 2 g rather than 10 g in the test.

For some solids, the procedure can be quite slow (e.g., Lawrence and Wang 1996). This is particularly the case for elevated carbonate contents and cases where the carbonate occurs with magnesium or magnesium and iron mixtures.

REFERENCES

Lapakko, K.A. 1994. Evaluation of neutralization potential determinations for metal mine waste and a proposed alternative. In *Proceedings of the International Land Reclamation and Mine Drainage Conference and Third International Conference on the Abatement of Acidic Drainage*, Pittsburgh, PA, April 24–29. Vol. 1, pp. 129–137.

Lawrence, R.W., and Wang, Y. 1996. *Determination of Neutralization Potential for Acid Rock Drainage Prediction*. MEND Project 1.16.3. Ottawa, ON: Mine Environment Neutral Drainage.



APPENDIX E

Siderite Correction

Siderite (FeCO₃) is a common accessory mineral in Appalachian coal field overburden (Cecil et al. 1985; Morrison et al. 1990). Meek (1981) calculated that the neutralizing potential (NP) of these overburden units would be overestimated when siderite is present.

Skousen et al. (1997) describe in detail the sequence of reactions that result in overestimation of NP when siderite is present. Because the standard NP procedure as outlined by Sobek et al. (1978) does not account for these complex reactions, erroneously high NP values can be generated with samples containing high amounts of siderite. Such an analytical oversight can lead to incorrect postmining water quality predictions; result in premature mine closure; and produce costly, long-term reclamation liabilities for mining companies (Wiram 1992).

Meek (1981) was the first to suggest adding a small quantity of 30% hydrogen peroxide (H_2O_2) to the filtrate of an HCl-digested siderite overburden sample to oxidize ferrous iron to ferric iron before back-titration. Because the resulting ferric iron is precipitated as $Fe(OH)_3$ upon titration, the solution yields a more accurate NP value. The addition of H_2O_2 after the initial titration results in the formation of additional $Fe(OH)_3$ due to enhanced oxidation at higher pH values.

This laboratory method was formalized by a Pennsylvania–West Virginia Overburden Task Force (Leavitt et al. 1995) and is detailed in Skousen et al.'s (1997) paper as well as at http://technology.infomine.com/enviromine/ard/acid-base%20accounting/acidbase.htm#Siderite.

Siderite is not a common accessory mineral in metallic ore deposits, but if its presence is determined through the detailed characterization and mineralogy recommended in this volume, then the siderite correction can be used to determine accurate NP values for use in waste characterization and management.

REFERENCES

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- Sobek, A.A., Schuller, W.A., Freeman, J.R., and Smith, R.M. 1978. Field and Laboratory Methods Applicable to Overburden and Minesoils. EPA 600/2-78-054. Cincinnati, OH: U.S. Environmental Protection Agency.
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APPENDIX F

Net Carbonate Value

PROTOCOL FOR DETERMINATION OF NCV

Each ore or waste sample or composite is assayed for total carbon and sulfur, residual carbon, and sulfur after pyrolysis at 550°C for 1 hour, and residual carbon after acid digestion with hydrochloric acid (HCl). The acid neutralization potential (ANP) is calculated based on the estimated carbonate carbon content of each sample, if any. The NCV method uses neutralization potential units of percent carbon dioxide (% $\rm CO_2$), due to the availability of certified standard reference materials with known carbon dioxide content.

ANP for the sample is estimated from the carbon assays required for determination of NCV. Carbonate carbon is calculated from the difference between the total carbon in the sample and the residual carbon after reaction with hydrochloric acid, which causes loss of the carbonate minerals present as gaseous carbon dioxide. ANP is calculated as % CO₂, as follows:

$$ANP = 3.67(CTOT - CAI)$$

where

ANP = acid neutralization potential, % CO₂

 $3.67 = factor for C to CO_{2}$

CTOT = total carbon content, % C

CAI = residual carbon acid insoluble, % C

In the absence of significant carbonate or organic carbon, which reports as residual carbon acid insoluble in the sample, the residual acid insoluble carbon method may be omitted and the ANP estimated from the residual carbon after pyrolysis, as follows:

$$ANP = 3.67 CAP$$

where

ANP = acid neutralization potential, % CO,

 $3.67 = factor for C to CO_{3}$

CAP = residual after pyrolysis, % C

ANP estimation by the pyrolysis loss method may result in lower ANP, due to losses of some carbon dioxide during pyrolysis, but it is more reliable than estimation of ANP using the total carbon determination, due to oil contamination in many samples. Metal carbonates, such as siderite, do not provide net neutralization potential and are included in ANP estimations, so it is necessary to use the acid neutralization potential acidity (ANPA) titration methods where significant metal carbonates occur (see ASTM E 1915-13). Estimation of ANP by pyrolysis loss may be more appropriate when metal carbonates and organic carbon minerals are absent, since the ANP

estimates are typically lower than those obtained using hydrochloric acid loss on samples containing carbonates.

The acid generation potential (AGP) of each sample is estimated by the sulfur loss on pyrolysis (J.M. Rendu, personal communication, 1990). Sulfides due to copper sulfide minerals, which may be present in the samples, are expected to be underestimated by this technique (McGuire and Thomas 1993). It may be necessary to estimate the sulfide content of the samples based on alternate sulfur speciation techniques (i.e., sodium carbonate residual sulfur), but the pyrolysis loss is most useful since it includes the primary acid-generating sulfides: pyrite, marcasite, and pyrrhotite.

AGP for samples is estimated by the difference between total sulfur and sulfur after pyrolysis of the sample. Pyrite, marcasite, arsenopyrite, and pyrrhotite are readily volatilized under the pyrolysis conditions used in the test, but copper sulfide minerals are only partially lost (i.e., bornite 27%, chalcopyrite 65%). AGP is estimated from the sulfur-phase analysis results as follows:

$$AGP = -1.37(STOT - SAP)$$

where

AGP = acid generation potential, % CO,

-1.37 = factor for sulfide to form H₂SO₄ and dissolve carbonate to form H₂CO₃

STOT = total sulfur, % S

SAP = residual sulfur after pyrolysis, % S

There is no one method that will properly estimate all sulfide minerals, so it is necessary that NCV analyses be backed up by mineralogy and other confirmation testing in order to select the best method to be used for each deposit during ore and waste management operations.

Negative ANP and positive AGP estimates must be corrected before calculation of NCV by setting them to zero in order to prevent inaccuracies in the estimation methods for NCV. The net carbonate value of each sample is calculated by balancing the ANP with the AGP, after correction as follows:

$$NCV = ANP(or ANPA) + AGP$$

where

ANP = acid neutralization potential, % CO₂

 $AGP = acid generation potential, % CO_{3}$

Many jurisdictions require that acid-base accounting results be reported in kilograms of calcium carbonate per metric ton of waste. ANP, AGP, and NCV results may be converted to those units of measurement by multiplication of the percent carbon dioxide by 22.7 kg $CO_3/t/\%$ CO_3 for reporting purposes.

PROTOCOL FOR CLASSIFICATION OF WASTE TYPES BY NCV

The analytical results for each ore and waste type are statistically analyzed, and appropriate classifications of ore and waste material are defined based on the NCV and waste type. The standard Newmont classifications based on the ANP, AGP, and NCV results are listed in Table F.1.

Newmont Mining Corporation ore, waste rock, and processing products acid rock drainage (ARD) evaluation procedures require sampling and NCV analyses of each waste type with enough samples or composites to cover the full range of NCV classifications that are applicable to

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TARI F	F F 1	Standard	NCV values

Classification	Criteria for Classification
Highly acidic (HA)	NCV ≤ -5
Acidic (A)	$-5\% < NCV \le -1\% CO_2$
Slightly acidic (SA)	$-1 < NCV \le -0.1\% CO_2$
Neutral (N)	$-0.1 < NCV < 0.1$ and (ANP ≥ 0.1 or AGP ≤ -0.1)
Inert (I)	-0.1 < NCV < 0.1 and (ANP < 0.1 and AGP > -0.1)
Slightly basic (SB)	0.1 ≤ NCV < 1
Basic (B)	1 ≤ NCV < 5
Highly basic (HB)	5 ≤ NCV

Source: Table first published in *World Gold 2011* by the Canadian Institute of Mining, Metallurgy and Petroleum (CIM), available at www.cim.org/en/Publications-and-Technical-Resources/Publications/Books/2011/10/World Gold2011hardcoverbookandCD.

that ore or waste type. It is recommended to test both ore and waste intervals during the studies so that meaningful relationships can be established in the cross sections through the deposit using modeling techniques. The NCV database generated in this manner will continue to be useful, even if the cut-off grades change during the development and mining of ore deposits.

PROTOCOL FOR NCV CONFIRMATION STUDIES

The NCV confirmation studies test ore and waste composites prepared by blending waste samples of similar NCV values within each waste type in ore and waste zones to fulfill and exceed the requirements of the original Nevada regulations using

- · Alternate acid-base classification techniques and X-ray diffraction-inductively coupled plasma (XRD/ICP) or X-ray fluorescence (XRF) on assay pulps;
- · Meteoric water leach testing (synthetic precipitation leaching procedure [SPLP] or meteoric water mobility procedure [MWMP]) on drill cuttings or field samples; and
- · Kinetic testing on pulps, drill cuttings, and/or field samples.

The NCV confirmation techniques may include the following methods:

- · ANPA (acid neutralization potential acidity) titration procedures with peroxide corrections for iron hydrolysis
- · ACPL (acid concentration present low [Sobek range]) slurry titrations to determine acidity present in samples
- · Acetic acid soluble calcium assays for calcite estimation
- · Sodium carbonate residual sulfur for sulfide estimation
- · Hydrochloric and nitric acid residual sulfur for pyritic sulfur estimation
- · Carbon disulfide sulfur loss to estimate elemental sulfur
- · Elemental characterization to the best detection limits for toxic release inventory
- · Biological acid production potential to confirm NCV static classifications and metals
- · Peroxide acid generation to confirm NCV static classifications and metals
- · XRD/XRF and MLA software analysis to identify and quantify the carbon, sulfur, and metal-bearing minerals
- · Microscopy to identify minerals and their relationships and morphology

- · MWMP or SPLP to determine soluble metals and anions
- Humidity cell testing to determine accelerated acid, metals, and alkalinity generation rates and
- · Site waste column testing to determine realistic acid, metals, and alkalinity generation rates in the field at the site during the life of the mine for water treatment closure and postclosure design

Results from the NCV confirmation studies are used to determine which ore and waste rock materials from the ore deposit have the potential to generate ARD; to determine suitable methods and costs necessary to manage ARD, if any, during production; and to properly plan and estimate costs for closure and postclosure.

REFERENCES

ASTM E 1915-13. 2013. Standard Test Methods for Analysis of Metal Bearing Ores and Related Materials for Carbon, Sulfur, and Acid Base Characteristics. West Conshohocken, PA:ASTM International.

McGuire, M.A., and Thomas, L.A. 1993. Differentiation of Carbon and Sulfur Phases in Ore Deposits, Progress Report III. Denver, CO: Newmont Metallurgical Services.

APPENDIX G

Net Acid Generation Tests

STATIC NAG TEST

Sample Preparation

Drill-core and bulk rock samples should be crushed to nominal 4 mm and a subsample pulverized to approximately 200 mesh ($<75 \mu m$). Tailing and process residue samples can be tested "as received."

Reagents

- · Reagent 1: H₂O₂—BDH "Analar" analytical reagent 30% w/v (100 V), or equivalent, diluted 1:1 with deionized H₂O to 15% (refer to note 1 later in this appendix).
- · Reagent 2: NaOH—0.50 M standardized solution.
- · Reagent 3: NaOH—0.10 M standardized solution.

Method

- 1. Add 250 mL of Reagent 1 (15% $\rm{H_2O_2}$) to 2.5 g of pulverized sample in a 500-mL wide-mouth conical flask, or equivalent. Cover with a watch glass, and place in a fume hood or well-ventilated area (refer to note 2 later in this appendix). The $\rm{H_2O_2}$ should be at room temperature before commencing the test.
- 2. Allow the sample to react until "boiling" or effervescing ceases. Heat the sample on a hot plate and gently boil until effervescence stops or for a minimum of 2 hours. Do not allow sample to boil dry—add deionized water if necessary.
- 3. Allow solution to cool to room temperature, then record final pH (NAG pH).
- 4. Rinse the sample that has adhered to the sides of the flask down into the solution with deionized water. Add deionized water to give a final volume of 250 mL.
- 5. Titrate solution to pH 4.5 while stirring with the appropriate NaOH concentration based on final NAG solution pH as shown in Table G.1.

TABLE G.1 NaOH concentration required for titration, static NAG procedure

NAG Solution pH	Reagent	NaOH Concentration
>2	3	0.10 M
<2	2	0.50 M

Source: Miller et al. 1997.

Calculation for Net Acid Generation

 $NAG = 49 \times V \times M/W$

where

NAG = net acid generation, kg H₂SO₄/t

V = volume of base NaOH titrated, mL

M = molarity of base NaOH, moles/L

W = weight of sample reacted, g

Note: If NAG value exceeds 25 kg $\rm H_2SO_4$ per metric ton, repeat using 1.00-g samples (sequential NAG).

Notes and Precautions

- 1. The pH of the H₂O₂ used in the NAG test should be checked to ensure that it is between pH 4 and 7. If the pH is less than 4, add dilute NaOH (use a solution made up by adding 1 g NaOH to 100 mL deionized H₂O) until the pH is greater than 4 (aim for a pH between 4 and 6). The pH is adjusted to greater than pH 4 to ensure that the phosphoric acid, used to stabilize H₂O₂ in some brands, is neutralized. The pH of the 15% H₂O₂ should always be checked to ensure that any stabilizing acid is neutralized; otherwise, false positive results may be obtained.
- The NAG reaction can be vigorous and sample solutions can "boil" at temperatures up to 120°C. Great care must be taken to place samples in a well-ventilated area or fume cupboard.
- 3. Caution should be taken in the interpretation of NAG test results for coal reject samples and other materials that may contain a high content of organic material (such as potential acid sulfate soils, dredge sediments, and other lake or marine sediments). All organic material must be completely oxidized; otherwise, acid NAG results can occur that are unrelated to sulfides. Several aliquots of H₂O₂ reagent may be added to the sample to break down any organic acidity.
- 4. Samples with positive net acid production potential value, high sulfur content, and high acid-neutralizing capacity must be carefully evaluated.

SEQUENTIAL NAG TEST

Sequential NAG tests involve simply repeating the single addition NAG test process on the same sample. At the end of each stage, the sample is filtered and the solution is used for measurement of NAG pH and NAG capacity. The NAG test is then repeated on the solid residue. The cycle is repeated until such time that there is no further catalytic decomposition of the peroxide or when the NAG pH is greater than 4.5. The overall NAG capacity of the sample is then determined by summing the individual acid capacities from each stage. The number of stages required before a sample turns acidic provides a guide to the length of geochemical lag (AMIRA International 2002).

KINETIC NAG TEST

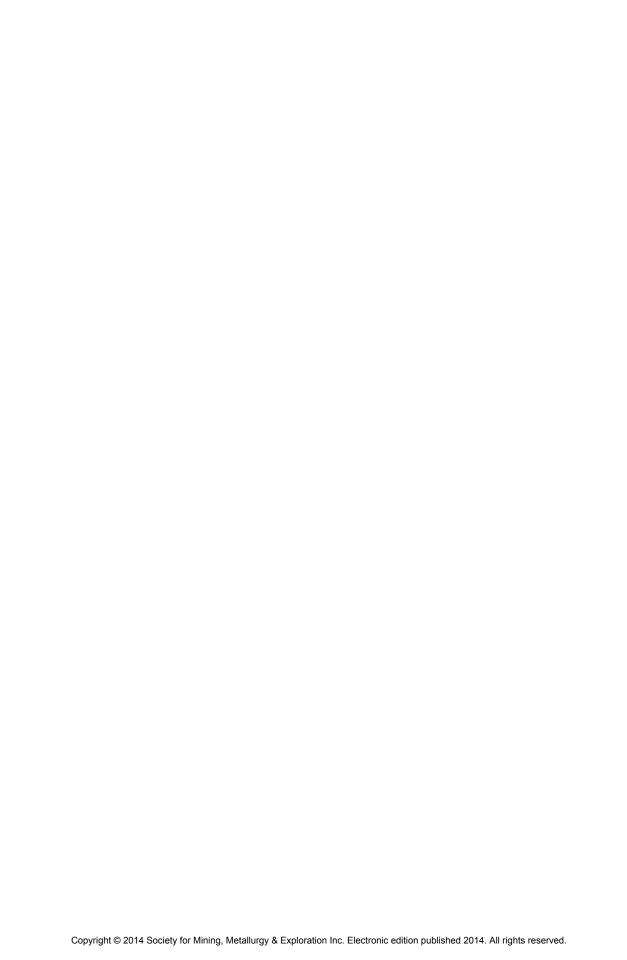
The kinetic NAG test is the same as the single addition NAG test except that the temperature, pH, and sometimes electrical conductivity of the liquor are recorded. These recorded values may

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help to understand the behavior of the tested material in the field and in humidity cells (AMIRA International 2002).

REFERENCES

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APPENDIX H

BC Research Confirmation Test

OBJECTIVES

The objectives of the BC (British Columbia) research confirmation test are to

- · Confirm the results of static prediction testing, and
- Determine if sulfide-oxidizing bacteria can generate more acid from a sample than can be consumed.

PRINCIPLES OF TEST

The acid potential derived by assuming total oxidation of sulfur (sulfides) in static prediction testing may not, under field conditions, be realized. To determine the degree to which the sulfur content of a sample might be oxidized and to assess whether this amount of acid is sufficient to overcome the neutralizing capacity of the sample, a biological oxidation test can be carried out. The test is usually performed only if the sample is shown to be potentially acid producing in static testing.

A preacidified pulp containing the finely ground test sample is inoculated with an active culture of sulfide-oxidizing bacteria such as *Thiobacillus ferrooxidans* and agitated under ideal conditions for bacterial oxidation. The pulp pH is monitored until it becomes stable, indicating the end of oxidation. An equivalent weight to the original sample is then added in two increments after 24 and 48 hours, and the pH is measured 24 hours after each addition. If the pH is above 3.5 at these times, the sample is classified as a non-acid producer, since the pH is out of the range considered essential for the growth and oxidative activity of the bacteria. If the pH remains below 3.5, a potential for acid generation is indicated.

MATERIALS

- · 250-mL Erlenmeyer flasks, preferably with a baffled base to facilitate oxygen mass transfer during agitation
- Temperature-controlled gyratory or reciprocating shaker/incubator equipped with clamps for Erlenmeyer flasks (provision for CO, enrichment of the air is desirable)
- · pH meter equipped with a combination pH electrode
- · Pipette, 5 mL

REAGENTS

- · Sulfuric acid, 6 N or 12 N
- · Distilled or deionized water

- · Reagent-grade nutrient salts, typically $(NH_4)_2SO_4$, KCl, K_2HPO_4 , $MgSO_4\cdot 7H_2O$, and $Ca(NO_3)_2$
- · Bacterial culture containing *T. ferrooxidans* (Cultures should be selected based on their known ability to be able to oxidize ores, waste rock, or tailings of similar mineralogy to the test sample.)

PROCEDURE

- 1. Crush and pulverize the sample to pass a 400 mesh (Tyler) screen.
- 2. Prepare bacterial cultures for use as inocula using standard laboratory techniques. The cultures should have been previously grown on and adapted to ore or waste rock containing pyrite with a sulfur content at least as high as the test sample. Whole pulp inocula are preferred to cells recovered from solution only. If solids-free inocula containing very low soluble metal concentrations are required, these should be prepared by differential centrifugation techniques.
- 3. In duplicate, weigh out 15–30 g (low weight for high sulfur contents) of sample into an Erlenmeyer flask with 70 mL of a nutrient medium containing (typically) 3 g/L (NH₄)₂SO₄, 0.1 g/L KCl, 0.5 g/L K₂HPO₄, 0.5 g/L MgSO₄·7H₂O, and 0.01 g/L Ca(NO₃)₂.
- 4. Place the flask on a shaker and periodically add sulfuric acid (6 N or 12 N) as required to bring pH to a stable value between pH 2.5 and 2.8. Do not proceed until pH is stable.
- 5. Inoculate flasks with 5 mL of an active *T. ferrooxidans* culture. Record weight of flask, cap flask with a cotton or foam plug, and place on shaker at 35°C.
- 6. Monitor the flask regularly for pH. Before each measurement, add distilled water to bring the flask to original weight to allow for evaporation. In some cases, the sampling of flasks for a soluble species (e.g., Fe, Cu, Zn) might assist in determining progress of the oxidation process.
- 7. When oxidative activity has ceased, as evidenced by a stable pH (or metal concentration), add half the weight of sample originally used and continue shaking for 24 hours.
- 8. Record the pH and if above 3.5, terminate the test. If not, again add half the weight of sample and agitate for up to an additional 72 hours and record the final pH.

Typically, about 3 to 4 weeks following inoculation is required to complete this test.

INTERPRETATION OF RESULTS

A decreasing pH following inoculation of the test pulp with bacteria indicates the biochemical oxidation of sulfides contained in the sample.

Once a stable pH or soluble metal concentration has been achieved, it is assumed that all sulfide available for oxidation has reacted and the maximum of acidity has been generated. By adding more sample equal to the original weight after reaction and observing the pH change, the ability of the sample to generate acid in excess of its neutralizing capacity can be assessed. Specifically, the sample is confirmed to be an acid producer if the pH remains below 3.5. Above this pH, biochemical oxidation is considered to be unlikely, and the sample is classified as a non-acid producer.

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REPORTING OF RESULTS

The results of the test should be tabulated to provide the following information:

- Sample description
- · Test duration (days after inoculation)
- · Initial pH
- · pH after biological oxidation
- · pH after first increment of sample addition
- · pH after second increment of sample addition (final pH)
- · Confirmation of acid production potential (yes/no)

ADVANTAGES OF TEST

- · Relatively low cost and rapid kinetic test.
- · Has been widely used in Canada.
- · Provides an assessment for the potential of biochemical oxidation.

DISADVANTAGES OF TEST

- · Although the procedure is apparently simple, tests involving the use of bacteria require experience and can have the problems of (1) nonoptimum use of cultures, (2) use of non-adapted cultures, and (3) difficulties of comparing results between laboratories.
- The acidification procedure creates an unrealistic condition in that the inhibitory effects of the alkaline components of the waste on oxidation reactions are not evaluated, and the method does not allow evaluation or modeling of the initial acid rock drainage production stages in the upper pH ranges.
- The amount of acid generated in the test is not corrected for the initial acid added to bring pH into the range suitable for inoculation. This can bias the result toward an acid classification.
- · For samples with high sulfur contents, the amount of sample required by the test procedure might be too large, leading to incomplete oxidation of the available sulfur due to inhibition of reaction by reaction products and low pH.

ORIGINAL REFERENCE

Bruynesteyn, A., and Hackl, R.P. 1984. Evaluation of acid production potential of mining waste materials. Miner. Environ. 4(1):5–8.



APPENDIX I

Humidity Cell Procedures

Humidity cell procedures are available as ASTM D 5744. The general procedures and the most recent modifications are summarized in this appendix (Bucknam et al. 2009).

HISTORICAL METHOD

In the ASTM D 5744-96 method, a 1-kg charge is placed into a small, cylindrical column (or cell), subjected to three days of dry air, three days of wet air, and rinsed on the seventh day. The effluent, or drainage, is weighed and analyzed to determine the solute release from the solids. The method suggests cell dimensions for coarse particles (d < 6.3 mm) and fine particles (d < 150 mm), provides the procedure for loading cells, and prescribes air-flow rates and humidities for the wet-air and dry-air cycles. Solids to be tested, as well as the leached solids after test termination, are characterized with respect to particle size distribution, chemistry, and mineralogy. Little guidance on mineralogical analyses is provided. The method provides guidance for the quality of water added for rinsing reaction products and presents alternatives for both the volume of water used (500 or 1,000 mL) and the method of its application (drip or flood-leach). It also outlines determinations of water mass retained in the cell after the weekly leach and the dry-air and wet-air portions of the weekly cycles. A minimum test duration of 20 weeks is recommended (although section 4.1 of the standard recommends a test duration of 20 weeks, section 11.6 and note 12 clarify that this is a minimum duration). Additional detail on the method is presented in White and Lapakko (2000).

The revision of ASTM D 5744-96, published in 2007 as D 5744-07e1, considered data from an interlaboratory study, comments from the ASTM International peer review process, and other reviewer input.

One major outcome of this review was improved replication of drainage pH and rates of solute release within and among laboratories. Second, the protocol from the Minnesota Department of Natural Resources was incorporated as Option B in the ASTM D 5744-07 method. This protocol had features that provided for consistent reaction conditions (temperature, humidity, pore water retention) and was less costly than the original ASTM D 5744-96. Third, the flood leach was designated as the preferred method of water application because data indicated little difference between the drip and flood leaches, and the flood leach had practical and possibly technical advantages.

Additional concerns raised during the peer review process, by solicited comments (Morin and Hutt 2004), and in a related publication (Lapakko 2003) were addressed and amplified in the following sections of ASTM D 5744-07:

- 1. Expanding characterization of preleach (notes 12 and 13, section 9.6) and postleach solids (paragraph 11.6.5.1, note 23);
- 2. Allowing for increased water volume for the initial rinse (note 16);
- 3. Recommending determination of pore water retained (note 24);

- 4. Suggesting minimum drainage analysis frequency (paragraph 11.5.2);
- 5. Providing guidance for estimation of missing values (note 22) and values reported as less than detection (section 12.5.2); and
- 6. Recommending charge balance determination to improve quality assurance/quality control (note 21).

Furthermore, the number of explanatory notes was increased from 15 to 27 to provide insight and guidance to maximize information derived from testing, and references cited were updated to further address the aforementioned objective (ASTM D 5744-07e1).

Two aspects were also eliminated in ASTM D 5744-07e1. Testing of tailings was eliminated because no intralaboratory or interlaboratory testing was conducted on them. Bacterial inoculation was also eliminated from the appendix based on unpublished U.S. Bureau of Mines data (R.R. Corwin, personal communication, 1995) and practical experience from practitioners of kinetic testing (Morin and Hutt 1997, 2004).

REFERENCES

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- ASTM D 5744-13. 2013. Standard Test Method for Laboratory Weathering of Solid Materials Using a Humidity Cell. West Conshohocken, PA: ASTM International.
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APPENDIX J

Proposed Adaptive Management Plan Outline

1.0 INTRODUCTION AND BACKGROUND

2.0 OBJECTIVES

3.0 STAKEHOLDERS

- 3.1 Newmont Mining Corporation
- 3.2 Nevada Division of Environmental Protection
- 3.3 Bureau of Land Management-Elko District Office

4.0 WASTE ROCK MANAGEMENT PLAN

- 4.1 Mine-Waste Rock Management Plan
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5.0 ADAPTIVE MANAGEMENT PLAN

- 5.1 Supplemental Waste Rock Characterization Studies
 - 5.1.2 Kinetic Test Decision Criteria
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- 5.2 Acid-Generating Waste Rock Management
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 - 5.2.2 Acid-Generating Management Actions Based on Soil Water Retention Curve Results
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6.0 WASTE ROCK MANAGEMENT MONITORING PROGRAM REVIEW

7.0 PERFORMANCE REVIEW

8.0 REFERENCES



APPENDIX K

Humidity Cell Test Duration and Testing Background*

Humidity cell (HC) tests are one source of information to help guide environmentally sound mine-waste management decisions. As opposed to solid-phase analyses, HC tests provide an empirically determined relationship between solid-phase composition and drainage quality under controlled laboratory conditions. Because drainage quality is a function of the solid-phase composition, HC testing is typically conducted on specific mineral assemblages (or rock types) that will be among the mine wastes generated. By testing individual mineral assemblages, many of the mineralogical and petrologic variables are normalized (mineral content, mineral chemistry, grain size, etc.), and testing can focus on the effects of solid-phase variables such as sulfide-sulfur content. The resultant data can be interpreted to determine critical sulfide-sulfur cutoffs for acidic drainage production and as a basis for modeling solute release rates from mine-waste facilities. These results can be used to inform mine-waste management decisions.

The extent of published data describing the relationship between well-characterized samples from individual rock types and their drainage quality in HC tests is limited. Even more limited are examples of long-term field drainage quality associated with mine wastes of known composition. Consequently, developing waste characterization programs in general—and, more specifically, HC testing programs—is a unique exercise for each operation, and possibly for minewaste disposal facilities within each operation. Details of the characterization program design are dependent on variables that are unique to individual mine settings, both physical and political.

Because each proposed and existing mining operation has substantial variation in host-rock and mine-waste mineralogy, endpoint designation of pertinent HC tests must logically be based on the site-specific data and their subsequent geochemical analyses, rather than some artificially prescribed endpoint. Attempting to designate a "one size fits all" test duration and endpoint based on a proposed "safe level" of sulfide-sulfur content, or minimal rate of sulfate mass release, and then suggest that having achieved either of these thresholds constitutes a safe criterion for terminating a test is risky at best. This is especially critical if the samples being tested represent large tonnages of waste rock. Therefore, many details of waste characterization and HC testing programs are most appropriately determined on a site-specific basis.

HUMIDITY CELL TEST DURATION—ANALYSIS AND RECOMMENDATIONS

Laboratory data generated from HC tests can be interpreted to help make informed decisions regarding environmentally sound waste-rock management. Ultimately, the testing, interpretation, and the consequent decision-making processes are complex and dependent on site-specific technical, regulatory, and political factors. Consequently, selection of test design components, including the duration, are best addressed on a site-specific basis, where appropriate consideration

^{*} This appendix was prepared by Kim Lapakko and Bill White, with minor revisions by Charles Bucknam.

can be given to the multitude of technical and nontechnical factors that are unique to the locale and affect these decisions.

HC test design and application of results is technically complicated. The test data are interpreted in order to contribute to a logical foundation for (1) segregating waste rock for disposal in engineered storage facilities, if required, based on the relationship between rock lithology and resulting drainage quality; and (2) predicting solute release rates in the field. To construct this foundation, HC test data are commonly interpreted and used as input to models designed to extrapolate laboratory data to field conditions. In this process, the HC data must be interpreted with respect to the technical and scientific aspects of the testing (e.g., test conditions, solid-phase characteristics, drainage quality data), the mine plan (mine wastes generated; mine-waste characteristics, mass, disposal options), the conditions at the site (e.g., climate, geology, hydrology, hydrogeology, topography), and scientific interpretation of the aforementioned information (e.g., consideration of geochemistry, hydrology, gas transport). The scientific and technical information relevant to HC test design, interpretation, and application is largely site specific. Consequently, test design, including duration, interpretation, and application, are most appropriately addressed on a site-specific basis by personnel adept in the scientific and technical aspects of environmentally sound mine-waste management.

Equally important to HC test design and interpretation are site-specific corporate, regulatory, and risk factors. The constituents of concern for potential environmental impacts and mode of drainage discharge (surface water, groundwater, point source, non-point source) vary among operations. The water quality standards applied are dependent on the operating company and regulatory agency, the constituents of concern, the receiving water, the mode of discharge, and other factors. These factors must be considered in the design of HC testing programs and will vary from site to site. Due consideration of these factors, and the HC program dependent on them, is best done by stakeholders familiar with the operating and regulatory environment at a specific site.

Ultimately, risk must be considered by the mining and processing operators' regulatory agency and community (stakeholders), and assessment of this risk depends to some degree on the reliability of the data on which modeling is based, which includes the HC data. The level of acceptable risk will vary among mine site locations, type of mining and ore processing, regulatory agencies, and other variables. Design of HC test programs and test variables such as duration is best left to those who accept the risk. They are the only ones who can establish the level of risk that is acceptable.

In conclusion, the determination of HC test duration is best left jointly to the stakeholders for a specific operation. A generic recommendation would not adequately assess the technical, regulatory, and risk factors that are unique to each site. Considering all these variables, and the fact that environmental-review time frames may vary between corporate, state, and federal jurisdictions, prescription of a set HC test endpoint is unrealistic. This is consistent with the judgment of both the GARD Guide (INAP 2009) and Price (2009), neither of which recommended a test duration. However, nonprescriptive guidance for test duration based on identified test objectives and long-term HC test data is presented in the ASTM HC test standard method (D 5744-13).

EXISTING GUIDANCE REGARDING TEST DURATION

There is a persistent but erroneous perception that the current ASTM HC test standard method (ASTM D 5744-13) prescribes a minimum 20-week test duration. *It does not.* Although minimum test duration of 20 weeks was contained in the original D 5744-96 (paragraphs 1.3 and 4.2), *this language was eliminated in D 5744-07* (the 2007 revision of the ASTM method). Although the subject of test duration is discussed in the D5744-07 "Scope," "Summary of Test

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Method," and "Test Duration" sections (paragraphs 1.3, 4.2, and 11.4), no specific test-duration time period is prescribed. The only mention of the term "20 weeks" is in paragraph 4.2, which states, "Although a test duration as short as 20 weeks may be suitable for some samples, more recent research indicates that a test duration well beyond 20 weeks may be required depending upon the objectives of the test (emphasis added)."

Specifically, the method dictates

- 1. Test duration is to be "...determined by the user's objectives of the test..." (paragraph 1.3);
- 2. "...Criteria for ending the testing will depend on the objective of the test, *which may be site specific.*.." (paragraph 11.4.1); and
- 3. If the objective is to determine the relative reactivity as a function of compositional variations within individual mine-rock lithologies for the purpose of segregating mine wastes, "...then a specific test duration is not prescribed..." (paragraph 11.4.2).

Additionally, paragraphs 11.4.2.1 and 11.4.3 provide specific data-supported examples of how low concentrations of magnesium and calcium carbonate in sulfide-bearing samples can influence interpretation of time to acid breakthrough. Consequently, this results in test durations of as much as 320 and 204 weeks, respectively, if the objective of the testing is to run the HC test until acid breakthrough occurs.

ADDITIONAL RECOMMENDATIONS

- 1. Mine-waste management decisions must consider not only HC test data but also other geochemical data collected in advance of the proposed operation. These data must be collected with diligent consideration of geology, lithology, mineralogy, geochemistry, mining engineering, hydrology, climate, and other technical aspects affecting mine-waste management. These data and proposed mine-waste management plans must be considered in design of HC testing programs (if available at the time that the tests are initiated), subsequent evaluation of HC test result interpretations, and application of interpretations on an operational scale.
- 2. The degree of uncertainty in HC test result interpretations often decreases with the test duration. It is therefore reasonable to conduct tests for a sufficient period of time to reduce uncertainty in interpretation, and the associated environmental risk, to an acceptable level. To this end, it is beneficial to begin testing far enough in advance of the operation so that sufficient data can be collected and confidently interpreted, and then make an informed decision that can be included in a detailed proposal.
- 3. Because each proposed and existing mining operation has substantial variation in host-rock and mine-waste mineralogy, endpoint designation of pertinent HC tests must be logically based on interpretation of the site-specific data and their subsequent geochemical analyses, rather than some artificially prescribed endpoint. Attempting to designate a "one size fits all" test duration, an endpoint based on a proposed "safe level" of sulfide-sulfur content, or minimal rate of sulfate mass release, may not meet the objectives of the testing. The testing should be continued until initial stakeholder, clearly defined test objectives are met. There are no rigorous, systematic studies on a wide range of rock types that provide a technically defensible foundation for any of these criteria. This is especially critical if the samples being tested represent large tonnages of waste rock.

For example, a sulfur threshold for acid production of 0.2% might be proposed with the inference that a sample of lower sulfur content will not produce acid. Acid rock

drainage is known to be produced by some materials and sites within or even below this sulfide-sulfur range, although many forms of sulfur are non-acid-forming. Such assertions must be supported with data. Counter examples exist in the experience of operating companies, the Minnesota Department of Natural Resources, and others. Greenstone rock with 0.2% sulfur produced a minimum pH of 3.9, and low-carbonate Virginia Formation rock with sulfur contents of 0.16% and 0.17% produced minimum pH values of 4.9 and 4.6, respectively. Establishing test criteria based on variables such as sulfur content must be supported with extensive empirical data, and it is likely such criteria will be site specific. At a minimum, criteria should be based on sulfide sulfur, unless the sulfur is known to be all sulfidic. Additional effort is needed to generate, compile, and catalog data to develop such criteria.

4. Industry, government, and academia can advance the present state of knowledge regarding humidity cells and their role in prediction. Efforts should be made to publish information from laboratory testing, field testing, and operational scale monitoring, thereby making results available to the community involved in the environmentally sound management of mine wastes. Our understanding of predictive testing and its application to the field will grow with the diligent generation and compilation of relevant laboratory and field data. More specifically, our understanding is dependent on data from long-term HC tests on well-characterized samples from specific lithologies and associated drainage quality and quantity data in the field. Systematic generation, compilation, and analysis of such data are needed to advance our understanding of predictive testing and its application to the field. This will reduce predictive uncertainty and the associated risk in the future.

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APPENDIX L

Possible Duration Framework for Use with Humidity Cells

This appendix is intended *only* to present a series of examples on how a framework for determining reasonable objectives for humidity cells might be structured and some possible criteria. This framework was developed following a presentation at the 2012 SME Annual Conference (Williams and Diehl 2012), the ADTI-MMS Steering Committee's desire for a format for addressing humidity cell (HC) duration issues, and discussions with reviewers familiar with HC review and evaluation (W.S. Schafer and L.B. Kirk, personal communications, 2011, 2012). They have found that establishing a framework which includes agreed-upon objectives and criteria to be a useful format in developing a detailed waste characterization and management program.

It is also possible to use the criteria/framework to establish a decision point rather than an endpoint to the HC test. Continuation of the HC test beyond this decision point may or may not provide important information regarding the acceleration or deceleration of oxidation and metal leaching. More detailed leachate information from a longer HC test may be critical information for designing waste management or water treatment facilities as accounted for in an adaptive management plan (see Appendix J), but an agreed-upon endpoint of test objectives would allow for a decision that advances mine planning and permitting.

This framework is based on the existence of detailed mineralogical work and static tests that provide a basis for anticipating and interpreting HC results. Detailed mineralogical work might lead a reviewer to suspect either neutralization potential (NP) or acid potential (AP) minerals have questionable availability, which would be a significant factor in evaluating HC results and potential test duration.

In some cases, it may be useful to establish criteria for a decision based on HC results but still maintain the HC test. In other cases, it might be useful to have duplicate humidity cells and use one as a basis for a decision point and subsequent destructive evaluation. This could support a decision concerning mine-waste management and planning, including an adaptive management plan. The duplicate cell would also be maintained to confirm the basis for the decision and be used to update the adaptive management plan and financial guarantee if necessary.

This framework does not necessarily resolve the need for accurate prediction of long-term metal leaching and drainage quality but is recommended as a tool for making decisions about how to conduct testing with the objective of determining how ore and waste will be handled and monitored, and the potential level of risk involved in related decisions for specific sites and materials.

Continuing an HC test for an extended period of time may also provide a higher level of certainty, as discussed in Chapter 5 and Appendix K. Depending on the site-specific resources at risk and behavior of waste materials, an extended HC test duration may be an important consideration for stakeholder groups to use in evaluating HC tests.

Given that a mine typically involves using very large quantities of waste, which will be leached by at least some amount of incident precipitation for extended times, ongoing monitoring of

dump performance, including any produced effluent or leachate, is almost always required as a condition of permit approval. Performance monitoring of permitted facilities can be a critical element in the development of an HC performance database, as well as support for the evolving HC test termination criteria/framework proposed here.

A model framework with possible objectives and criteria has been developed through discussions with technical specialists familiar with attempting to interpret HC tests is presented as an example in the following section. Variations in specific framework requirements and criteria (% S, sulfide, carbonate, pH, sulfate release, etc.) will depend on the site-specific objectives, deposit mineralogy and characterization, including various static test results and management plans agreed upon by stakeholders. Regardless of the site-specific stakeholder objectives, instability in metal release rates should strongly suggest continuation of testing.

Regardless of the decision process followed, the ultimate responsibility for the permitting decision lies with the permitting agency(s), and the ultimate environmental liability lies with the mining company.

STAKEHOLDER OBJECTIVES

1. Confirmation of Static Testing Results

Strongly acidic (ABA values of $\leq -20 \text{ kg CaCO}_3/t$) or basic lithologies (ABA values \geq +20 kg CaCO₃/t). Confirmation of static test results using humidity cells should be initiated early in the mine and process-development cycle to identify whether certain waste materials will require further study when the plan of operations for the facility is better defined. If the objective of the HC test is to *confirm* static test results for samples that show **high potential for acid gener**ation (slightly acidic to highly acidic) or high net potential available for neutralization of acid (basic to highly basic), the historic (ASTM D 5744-96, but no longer suggested) 20-week short duration test period (or even shorter durations, particularly in the case of highly reactive samples with significant acid generation potential) should be adequate. The tests should be supplemented by the recommended ASTM weathered solid material analyses (ASTM D 5744-13, paragraph 11.6 and note 23, which detail the mineral and/or chemical analyses of weathered material) or mass balance calculations as appropriate. Notably, even for highly acidic (high potential for acid generation) or highly basic (high net neutralizing potential) samples, a longer test duration may provide important information regarding the acceleration or deceleration of oxidation and metal release behavior that may be critical information for designing waste management or water treatment facilities. If this information is critical, it is probably appropriate to identify a different objective and testing protocol, as discussed in the following section.

Neutral/inert or slightly basic lithologies (acid-base accounting [ABA] ± 2.5 kg CaCO₃/t, ± 23 kg CaCO₃/t, AP and NP $\approx \pm 0.1$ kg CaCO₃/t). If the overall objective is to evaluate a material that is classified as having an unlikely or lower potential (neutral/inert to slightly basic) to produce acid leachate by static testing, then the criteria developed will be more site specific (mineralogy, lithology, environmental risk) and more complex.

If the objective is to identify material that *may* produce acid leachate and *will be managed as acid-producing material*, then the historic (ASTM D 5744-96, but no longer suggested) 20-week short duration test period should be adequate and should be supplemented by the recommended ASTM weathered solid material analyses (ASTM D 5744-13) or mass balance calculations as noted previously.

All cells with static test classification of material ranging from neutral/inert to slightly basic that have become moderately to strongly acidic, owing to sulfide oxidation (e.g., stable average pH < 4.0 during the last 10 weeks of testing), will be determined to be potentially acid generating,

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and testing can be terminated after the end of the 10 weeks at pH < 4.0. In this case, it is possible that the historic (ASTM D 5744-96, but no longer suggested) 20-week short duration test period will be sufficient.

All cells with static test classification of material ranging from neutral to slightly basic that have maintained neutral to alkaline conditions (e.g., a stable average cell effluent of pH \geq 6.5 for at least 10 weeks) and that have calcium + magnesium to sulfate ratios that are consistent with carbonate neutralization (e.g., molar ratio > 1:1) and that have an acid-neutralizing potential/acid-generating potential (ANP/AGP) ratio greater than 2, or no significant sulfide detected, can be terminated after the end of 10 weeks averaging a stable pH \geq 6.5.

Cells with static test classification of material as inert or nonreactive lithologies (samples with <0.2% sulfide sulfur *and* positive ABA and static test results) with net carbonate carbon in the range of $\pm 0.2\%$ CaCO₃ and NP $\geq 0.2\%$ CaCO₃ and AP $\leq 0.2\%$ CaCO₃, and that have average sulfate release of less than 3 mg/kg/week for each of the last 10 weeks, can be terminated after 10 weeks of sulfate release <3 mg/kg/week. These criteria must be developed on a site/project-specific basis based on the site-specific lithology, mineralogy, trace-metal characteristics, and potential environmental risks.

Samples with very low sulfide sulfur can exhibit anomalous pH readings based on a sample having little to no buffering capacity. While interesting in a geochemical sense, it should *not* necessarily be interpreted as representing potential acidity. Low pH leachate results generated by these test materials can be verified by comparison with the pH of the laboratory water used to flush the cells.

The values suggested for % sulfide, CaCO₃, sulfate release, and weeks of stability were suggested by researchers familiar with HC evaluations and are presented *only as reference values*. These values may need to be adjusted depending on the deposit-specific lithology, mineralogy, and resources at risk as noted herein.

2. Evaluation of Reactivity and Leachate Quality for Segregating Mine Waste

If the objective is to evaluate the relative reactivity and leachate quality for the site-specific purpose of segregating and managing mine waste in engineered storage facilities, the following evaluation criteria may be appropriate:

- · All cells with static test classification of material ranging from neutral/inert to slightly basic that have become moderately to strongly acidic, owing to sulfide oxidation (e.g., stable average pH < 4.0 during the last 10 weeks of testing), will be determined to be acid generating, and testing can be terminated after the end of the 10 weeks at pH < 4.0. In this case, it is possible that the historic (ASTM D 5744-96, but no longer suggested) 20-week short duration test period will be sufficient. In some cases, it may be necessary to continue testing until the specific parameters of interest for the project have stabilized. Based on either conflicting static test results or complex mineralogy, uncertainties may result in the continuation of the testing as noted next.
- · All cells with static test classification of material ranging from neutral to slightly basic and with calcium + magnesium to sulfate ratios that are consistent with carbonate neutralization (e.g., molar ratio > 1:1) and an NP/AP ratio greater than 2, or no significant sulfide detected, can be terminated under the following results. The sample has maintained neutral to alkaline conditions (e.g., a stable average cell effluent of pH \geq 6.5 for at least 10 weeks) after the end of the 10-week period averaging pH \geq 6.5. Based on either conflicting static test results or complex mineralogy, which could include deposit-specific

questionable availability of either AP or NP, uncertainties may result in the continuation of the testing as noted next.

· Cells with static test classification of material as inert or nonreactive lithologies (samples with <0.2% sulfide sulfur *and* positive ABA and static test results) with net carbonate carbon in the range of $\pm 0.2\%$ CaCO₃ and NP $\leq 0.2\%$ CaCO₃ and AGP $\geq -0.2\%$ CaCO₃, and that have average sulfate release of less than 3 mg/kg/week for each of the last 10 weeks, can be terminated after 10 weeks of sulfate release < 3 mg/kg/week. Releases of metals should also demonstrate minimal solubility (less than maximum contaminant levels) or declining releases to support test termination. Any uncertainties that concern ore or gangue mineralogy (e.g., the presence of sulfate minerals gypsum CaSO₄·2H₂O or alunite (KAl₃(SO₄)₂(OH)₆)) could result in anomalously high sulfate release values that would not necessarily indicate acidic drainage.

As noted previously, the values suggested for % sulfide, CaCO₃, sulfate release, and weeks of stability were suggested by researchers familiar with HC evaluations and are presented *only as reference values*. These values may need to be adjusted depending on the deposit-specific lithology, mineralogy, anomalous test results, and resources at risk.

3. Evaluation of Quantity of Neutralization Potential Available to React with Produced Acid

In the event that detailed information on the actual availability and timing of neutralization is critical to developing a comprehensive waste management plan, the following factors must be considered:

- If the objective is to interpret the amount of NP chemically and physically available to neutralize produced acid, the testing must continue until acidic effluent is produced or interpretation of leachate analysis indicates that all the carbonate mineralization has been depleted *and* the pH has maintained a circum-neutral value (≥6.5 or laboratory leach water pH) for at least 10 weeks after the depletion of carbonates. This interpretation can be for the purpose of either detailed waste management planning or detailed geochemical work, which can support modeling efforts for contaminant release. The time period for determining that an HC test for this objective can be terminated is indeterminate but has been as long as 320 weeks. In some cases, it will likely be more cost-effective to assume an amendment rate and incorporate that into a plan of operations than to try and predict a depletion rate.
- The final residues from the extended testing should be supplemented by the recommended ASTM weathered solid material analyses (ASTM D 5744-13) as noted previously.
- · Additional detailed discussion on test duration and frequency of analyses can be found in ASTM D 5744-13 (paragraphs 11.4 and 11.5.2, respectively) and is also addressed in Appendix K.
- Given the potential duration of HC tests, the importance of early test initiation cannot be overstated. Other considerations include (1) ongoing field pilot study tests, which can be used to supplement HC results and support detailed geochemical modeling; (2) geoenvironmental comparisons with closely related deposits; and (3) the possible need for duplicate cells for lithologies with complex mineralogy or conflicted static test results as an alternative to restarting humidity cells with disputed termination decisions. In this case, should a cell be terminated, the ASTM-recommended post-termination weathered solid material analyses (ASTM D 5744-13) should be completed and the results interpreted. If

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this analysis indicates continued uncertainties, including the unexpected release of metals at circum-neutral pH values, the duplicate cell can be continued in order to resolve identified uncertainties, which may include detailed metal leaching information.

 Deviations from the ASTM D 5744 protocol are discouraged but should be detailed and agreed upon by the stakeholders prior to test initiation. ASTM D 5744 will continue to be reviewed and updated at a minimum frequency of every 5 years (ASTM D 5744-13).

Monitoring the field performance of waste units will be critical to developing a detailed HC performance database as well as the continued use of this proposed framework (note item 4 in Appendix K). Monitoring the field performance of waste units, of course, is also critical to ensuring environmental protection, which is why it is commonly included as a condition of permit approval. This information may be useful in the development of a field-scaled timeline for specific mineral assemblages or deposits as discussed in Chapter 5 in order to assign a timeline for kinetic tests that has some basis in reality.

It is important to understand that HC tests should not be considered a "one stop" test for all solute release and kinetic reaction questions. Concerns about the lack of control of microbially mediated processes within HC tests are an ongoing research topic. HC tests are simply another tool in the toolbox, and the results of any HC tests must be interpreted in conjunction with all the other static, short-term leach tests, and kinetic and mineralogical work that must be a part of detailed rock characterization.

Note: This framework is suggested as a model to be used by the involved stakeholders for their determination of when it is appropriate to schedule and terminate a humidity cell. The specific parameters (% sulfide sulfur, % CaCO₃, SO₄ release rates, metal release rates, etc.) involved will likely vary depending on site-specific factors, which could include the lithology, petrology and mineralogy, climate, regulatory framework, environmental risk for the units, and ore-deposit type being evaluated. The criteria ultimately selected for waste characterization should rely on a combination of parameters because any criteria based on a single parameter value like % sulfide sulfur will not be reliable (Morin and Hutt 2006). The values in this proposed framework are chosen only as examples and are intended to be reviewed and agreed upon by the stakeholders. The specific parameters and values selected might vary considerably depending on site-specific factors, which might include environmental risk. It is up to the stakeholders to modify and use this framework to develop objectives that meet the specific requirements at their site and use their modifications to reach a consensus on test duration.

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